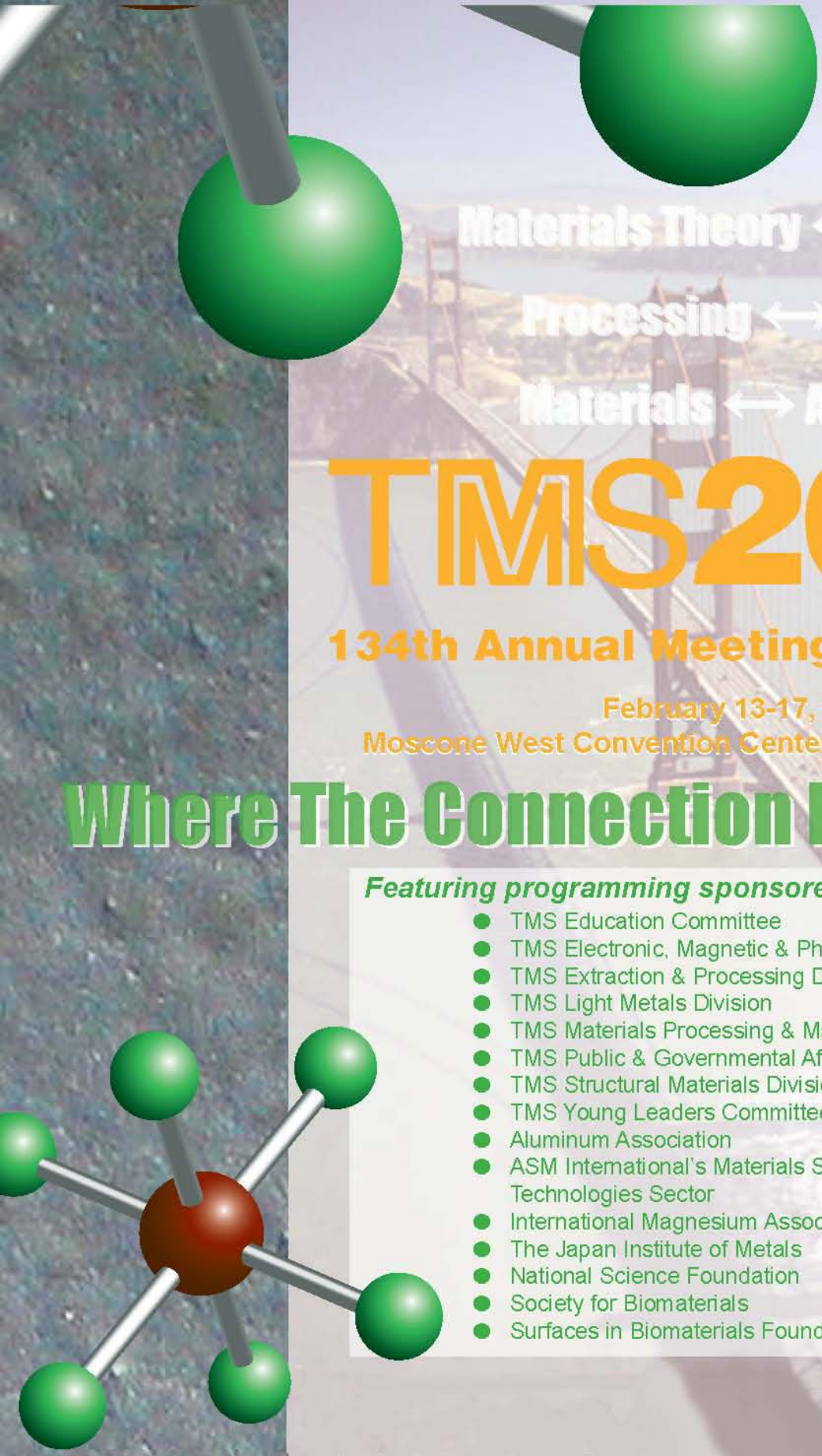

Materials Theory ⟷ Experiment

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6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Rapid Prototyping

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Tuesday AM — Room: 2005
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: James W. Sears, South Dakota School of Mines & Technology, Rapid City, SD 57701 USA; Jianxin Liu, Extrude Hone Corporation, ProMetal, Irwin, PA 15642 USA

8:30 AM
Supersolidus Liquid Phase Sintering of Tool Steel for Rapid Tooling: *Jianxin Liu*[1]; Howard Kuhn[1]; [1]Extrude Hone Corporation, ProMetal, 1 Industry Blvd., PO Box 1000, Irwin, PA 15642 USA

Three dimensional printing (3DP) is a solid-free form fabrication technique using metal powder as the building material. Green performs made by 3DP usually consist of less than 60 volume percent of metal powder, about 10 volume percent binder to hold metal powder together, and the remainder being pore space. Because of high porosity level a liquid phase must present in the thermal process to attain high density. In this work, supersolidus liquid phase sintering of prealloyed tool steel powder, M4-Co, was investigated. Promising experimental results and successful manufacture practice proved the M4-Co can be used successfully for rapid tooling.

8:50 AM
Development of Laser Ultasonics for Defect Detection During Laser Powder Deposition: Jason Nemeth[1]; Marvin Klien[2]; *James W. Sears*[1]; [1]South Dakota School of Mines & Technology, Additive Mfg. Lab., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]Lasson Technologies, 6059 Bristol Pkwy., 1st Fl., Culver City, CA 90230 USA

Laser ultrasonics shows great promise for on-line monitoring of product integrity in laser powder deposition (LPD). In the LPD process, we wish to detect defects on each layer as it is being deposited. The defect information can be then used to control critical weld parameters or alert the operator of a problem requiring maintenance. In this way, defect scan then be corrected before many defective parts are produced. Laser ultrasonics uses a pulsed laser to generate an ultrasonic wave and a continuous-wave laser interferometer to detect the small surface displacement when this wave arrives at the point of detection. Laser ultrasonics is ideal for in-line measurements because there is no sensor in contact or near-contact with the work piece. In this study, we have used laser-generated surface waves to interrogate LPD parts in both stainless steel and Ti. The processing approach was optimized using simulated pores produced using blind holes.

9:10 AM
Micron Scale Deposition for Rapid Prototyping of Electronic Components: John Preheim[2]; Jacob Colvin[1]; Keith Whites[2]; *James W. Sears*[1]; [1]South Dakota School of Mines & Technolgy, Additive Mfg. Lab., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines & Technology, Electl. & Computer Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA

Maskless Meso-Scale Material (M3D) is a Direct Write technology that uses nano-particles inks to build micron-scale electronic prototypes. The process can deposit most any nano-particle that can be suspended in a liquid with a viscosity of 1 to 1000 cp. Particle loading can be has high as 60%. A variety of materials have been deposited including; silver, gold, platinum, copper, rhodium, PMA, graphite epoxy, kapton, and barium titanate to name a few. The response of several electronic devices (e.g., capacitors, filters, resistors, inductors) have been characterized and compared to simulations.

9:30 AM
Structure-Property-Process Parameters Correlation in Rapid Prototyped 316L by Direct Laser Deposition: *Jyotsna Dutta Majumdar*[1]; Indranil Manna[1]; Lin Li[2]; [1]Indian Institute of Technology, Metallurgl. & Matls. Engrg., Kharagpur, W. Bengal 721302 India; [2]University of Manchester Institute of Science and Technology, Mech., Mfg. & Aeros. Engrg., Manchester M60 1QD UK

Laser assisted fabrication is a technique that utilizes high-power lasers to melt materials in the form of a powder or wire and controlled deposition of the molten metal in a layer-by layer fashion to induce a pre-determined shape. In the present study, 316L stainless steel has been fabricated by laser assisted direct metal deposition technique with a high power continuous wave Diode laser using Ar as shrouding environment. The main process variables were, applied power, and scan speed. Powder feed rate was maintained constant to 20 mg/s. After fabrication, the surface and cross section of the fabricated components were characterized using optical and scanning electron microscopy. X-ray diffraction study and energy dispersive spectroscopic analysis. The mechanical and electrochemical properties of the fabricated component were evaluated. Finally, a detailed study of the influence of process parameters on the characteristics and mechanical properties of the fabricated component has been undertaken to optimize the processing zone.

9:50 AM
Multi-Material Extrusion for Fabrication of Artificial Teeth: Jiwen Wang[1]; *Leon L. Shaw*[1]; Thomas B. Cameron[2]; [1]University of Connecticut, Dept. of Metall. & Matls. Engrg., Inst. of Matls. Sci., 97 N. Eagleville Rd., U3136, Storrs, CT 06269 USA; [2]Dentsply Ceramco, Burlington, NJ 08016 USA

A solid freeform fabrication (SFF) based technique, called multi-material slurry extrusion (M2SE), has been developed to fabricate artificial teeth made of dental porcelains and silver metals. The rheological and extrusion behavior of dental porcelain and silver slurries are controlled by the solids loading, pH value, and drying speed of the slurries after delivery. Dental crowns and solid teeth made of dental porcelains alone and dental porcelains plus silver metals are demonstrated using the M2SE method. Issues related to fabrication of dental units, such as slumping, overhang angles, bridging capability, material overfilling, generation of porosity, resistance of the green body to cracking under the weight of the top layers and dimensional accuracy, are discussed. The effects of the rheological behavior and extrusion parameters on these issues are investigated and the solutions are proposed.

10:10 AM Break

10:25 AM
Microstructure and Mechanical Properties of Spray-Formed H13 Tooling: *Yaojun Lin*[1]; Kevin M. McHugh[2]; Young-Soo Park[1]; Yizhang Zhou[1]; Enrique J. Lavernia[1]; [1]University of California, Cheml. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Idaho National Engineering and Environmental Laboratory, Industl. & Matl. Tech. Dept., Idaho Falls, ID 83415 USA

Spray forming is a recently developed technology that has been demonstrated to successfully fabricate molds and dies. During this approach, atomized droplets are directed towards a pre-designed pattern where they accurately capture features of the pattern during solidification to form the desired mold or die. Compared with the conventional techniques for mold/die production, a series of delicate machining, grinding, and polishing steps can be eliminated in spray-formed tooling. Furthermore, rapid solidification associated with spray deposition can produce metastable phases in the deposited tooling material. As a result, die properties such as hardness and toughness can be tailored via subsequent aging (tempering) heat treatment. In contrast, conventional austenitization/quench/temper heat treatment is required in conventional mold/die making techniques. Therefore, spray-formed tooling will save energy, production cost and production time and avoid mold/die distortion during heat treatment. This paper reports the microstructures and mechanical properties of H13 alloy samples processed via spray-formed tooling.

10:45 AM
Fabrication of PEM Fuel Cell Bipolar Plates by SLS: *Ssuwei Chen*[1]; David Bourell[1]; [1]University of Texas, Lab. for Freeform Fabri-

cation, TX Matls. Inst., 1 Univ. Sta., MC C2200, Austin, TX 78712-0292 USA

One of the barriers to commercialization of Polymer Exchange Membrane (PEM) fuel cells is economic fabrication of the bipolar plate. Manufacturing of pure graphite bipolar plates with machined flow fields is both time consuming and costly. The advantage of applying Selective Laser Sintering (SLS) for bipolar plate fabrication is that integrated cooling channel can be simultaneously formed on the central surfaces of the plate rather than by gluing two half plates back-to-back as is used in many other techniques. Requirements such as electrical conductivity, gas impermeability, etc. for bipolar plates are reported as affected by material selection and fabrication process.

11:05 AM
Application of In-Situ Melt Pool Sensor and Z-Height Control System to Laser Engineered Net Shaping (LENS™) Process: *John E. Smugeresky*[1]; Baolong Zheng[2]; Leonardo Ajdelsztajn[2]; Yizhang Zhou[2]; Julie M. Schoenung[2]; Enrique J. Lavernia[2]; [1]Sandia National Laboratories, Dept. 8724, Livermore, CA USA; [2]University of California, Cheml. Engrg. & Matls. Sci., 1 Shields Ave., Davis, CA 95616 USA

Direct fabrication of metallic parts with LENS™ is achieved by feeding powders to the melt pool created by a high-powered laser beam on a substrate. During LENS™ deposition, both a constant melt pool size and a constant deposit height are of importance, in particular for microstructural homogeneity and dimension accuracy of the parts. Using a melt pool sensor (MPS) to maintain the same melt pool size, and a Z-height control system to retain the same deposit height are seen as positive measures to insure the most uniform features. The objective of this work is to establish the correlation of process parameters to the geometrical features of the deposit microstructure and dimensional accuracy of the deposited parts, with and without MPS and Z-height control system. The microstructure characteristics and micro-hardness of the laser deposited parts, as a function of type of control environment, are reported. Work by Sandia is supported by the U. S. Department of Energy under contract DE-AC04-94AL85000. Work at UC Davis is supported by the National Science Foundation under grant number DMI-0423695.

11:25 AM
Characterization of Interfaces Between LENS® Deposited and Cast or Wrought 304L Stainless Steel: *J. E. Smugeresky*[1]; C. V. Robino[2]; D. D. Gill[2]; M. F. Harris[2]; M. L. Griffith[2]; [1]Sandia National Laboratories, Livermore, CA 94551 USA; [2]Sandia National Laboratories, Albuquerque, NM 87185 USA

The repair and/or modification of expensive metal hardware using LENS® (Laser Engineered Net Shaping™) instead of complete refabrication has the potential for huge savings in cost and delivery time for transportation industries. As such, the interface between LENS deposited material and the underlying base material must be metallurgically sound and possess mechanical properties equal to/or better than the base material. A metallurgical evaluation was conducted on thirteen different such interface configurations of deposited material and the underlying base material, representing a spectrum of deposition conditions from complete part build, to hybrid substrate-LENS builds, to repair/ modification builds. Tensile bars, with the interface at the center of the gauge length were pulled to failure to determine mechanical properties and characterize the fracture behavior. Good mechanical properties and full density were observed for all configurations. When tested to failure, fracture occurred by ductile micro-void coalescence. Based on microstructure charactereization, the repair and hybrid interfaces showed the same metallurgical integrity and properties as monolithic LENS deposits. Work by Sandia is supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Sheet Metal Forming

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Tuesday AM — Room: 2009
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: M. R. Stoudt, National Institute of Standards and Technology, Matls. Performance Grp., Gaithersburg, MD 20899-8553 USA; Glenn S. Daehn, Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH 43210 USA

8:30 AM Invited
Strategies for Sheet Metal Forming Using Mechanical Impulse: *Glenn S. Daehn*[1]; Manish Kamal[1]; Mala Seth[1]; Jianhui Shang[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210 USA

Presently sheet metal forming is almost exclusively thought of, performed and analyzed in a manner where "inertial forces are negligible". The main emphases of this presentation are: 1) There are methods that can reliably, predictably and robustly impart dramatic velocities and acceleration levels in sheet metal forming, 2) High velocity and acceleration can dramatically improve the stability of metal flow improving formability and resistance to wrinkling, 3) Acceleration due to impact can be used emboss textures on the surfaces of metals and strongly modify springback, and 4) Elegant methods exist for combining these high velocity forming technologies with more traditional metal forming methods. When used in a strategic manner, impulsive metal forming can treat many persistent problems in sheet metal forming. This overview will also highlight two focus areas. First, is the design and operation of an electromagnetic coil configuration that is very efficient, robust and gives a very uniform electromagnetic pre ssure distribution. This coil is especially useful for forming nominally flat components with fine surface features. Second, we will highlight how formability can be controlled and improved through impulsive means. Improved formability is a compelling reason to consider high velocity forming in sheet metal working.

8:55 AM
Electrohydraulic Forming of Automotive Panels: *Sergey F. Golovashchenko*[1]; Viacheslav S. Mamutov[2]; [1]Ford Motor Company, Mfg. & Processes, 2101 Village Rd., Ford Rsch. & Advd. Engrg., Dearborn, MI 48124 USA; [2]St. Petersburg Polytechnical University, 29 Politekhnitcheskaya st, St. Petersburg 195251 Russia

In this presentation, results on sheet metal forming using pulsed electrohydraulic technology will be discussed. Pulsed electrohydraulic forming is an electrodynamic process, based upon high-voltage discharge of capacitors between two electrodes positioned in a fluid-filled chamber. This technology combines the advantages of conventional hydroforming and high-rate forming processes. Compared to traditional forming in steel dies, electrohydraulic forming allows more uniform distribution of strains, makes wider the formability window, and reduces springback of parts. Parameters of electrohydraulic discharge and experimental results on formability of outer body skin panel material will be reported.

9:15 AM
Pulsed Electromagnetic Forming of Aluminum Body Panels: *Sergey F. Golovashchenko*[1]; Nickolas M. Bessonov[2]; Richard W. Davies[3]; [1]Ford Motor Company, Mfg. & Processes Dept., 2101 Village Rd., Dearborn, MI 48124 USA; [2]Institute of Problems of Mechanical Engineering, 61 Bolshoy prospekt V.O., St. Petersburg 199178 Russia;

[3]Pacific Northwestern National Laboratory, Energy Matls., 902 Batelle Blvd. K2-03, Richland, WA 99352 USA

Pulsed electromagnetic forming is based on high-voltage discharge of capacitors through a coil. This pulse generates an intense transient magnetic field in the coil that applies pressure to the metal work-piece to do the work. In theoretical analysis, the propagation of electromagnetic field is defined by quasi-stationary Maxwell equations transformed in Lagrangian form. Dynamics of elastic-plastic deformation is modeled using solid mechanics equation of motion and modified theory of elastic-plastic flow. Energy conservation law is employed for simulation of heat transfer, which is important to define the appropriate stamping rate without overheating the coil. Results of numerical simulation and experimental data will be presented. Methodology of testing durability of coils for pulsed electromagnetic forming will be also discussed.

9:35 AM

Product and Process Development with Local Thermal Manipulation of Aluminum: *Børge Iver Bjørneklett*[1]; [1]Hydro Aluminium, Auto. Struct., Fabrikkvei 1, PB 15, Raufoss N-2831 Norway

The present paper describes a concept that introduces a new dimension in design and fabrication of components and structures in aluminum for the transportation sector. By intensified heating in short pulses it is possible to manipulate the mechanical properties of aluminum alloys at accurate positions. The basic principle of local thermal manipulation is recently applied in different areas e.g. crash absorbing members, in new forming techniques and assembly methods. Innovations in this field have spurred new product designs and exiting possibilities for through process simulation. Practical examples and conceptual studies are shown for LTM in fabrication of various automotive aluminum components.

9:55 AM

Material Characterisation for Laser-Assisted Sheet Metal Hydroforming: Heinz Haferkamp[1]; Jens Bunte[1]; *Lars Engelbrecht*[1]; [1]Laser Zentrum Hannover, Matls. & Processes, Hollerithallee 8, 30419 Hannover Germany

Localised laser heating used during sheet metal hydroforming processes should reduce the work pressures necessary for hydroforming presses. By reducing the yield strength and the strain hardening using local heating, small form elements like creases can be formed at very low pressures of 2 MPa, whereas cold forming requires pressures which are 20-50 times higher. Besides the forming temperature and work pressure, temperature distribution is very important and can be modified using a special beam forming optics. This leads to improvements in the plastic deformation distribution. A significant improvement of the material's formability is shown by forming limit curves (FLC) which were generated using the bulge-test. Moreover, the mechanical properties and the grain structure of the form elements generated were determined. All investigations were carried out for a deep drawing steel, a 5182 aluminium alloy and an AZ31 magnesium alloy.

10:15 AM Break

10:30 AM

Evaluation of Deformation-Induced Surface Morphologies Generated in an Aluminum Alloy Sheet: *M. R. Stoudt*[1]; J. B. Hubbard[1]; J. Liu[1]; [1]National Institute of Standards and Technology, Matls. Performance Grp., 100 Bureau Dr., Stop 8553, Gaithersburg, MD 20899-8553 USA

Numeric predictions of mechanical behavior and friction during forming are central to the automotive design process; yet presently, significant discrepancies exist between what is predicted by numeric methods and what is measured on real surfaces. These discrepancies indicate that a better understanding of the fundamental relationships between deformation and the ensuing roughness at the free surface is required for better predictive accuracy. Most of the available surface roughness data are derived from assessments that compress the complex surface information into quantities that are too coarse with respect to the length scale of the relevant features. Furthermore, these assessments frequently contain tacit assumptions about the distributions of the surface features within the roughness data that may further degrade the precision. The results of a rigorous statistical protocol characterizing the roughening of a 6xxx series aluminum sheet will be discussed with emphasis on development of a better approach that should produce a robust generic model of roughening behavior.

10:50 AM

Microstructure Based Modelling of Al-Mg-Si Alloys in Development of Local Heating Processes for Automotive Structures: *Hallvard G. Fjaer*[1]; Børge I. Bjørneklett[2]; Ole R. Myhr[2]; [1]Institute for Energy Technology, Dept. of Process & Fluid Flow Tech., PO Box 40, NO-2027 Kjeller Norway; [2]Hydro Aluminium Structures Raufoss AS, Product & Process Dvlp., PO Box 15, NO-2831 Raufoss Norway

This work addresses the exploitation of local heating on automotive parts of age hardening aluminium alloys. Local heating can be applied for many purposes, e.g. correction of geometrical shapes, reduction of the flow stress in forming operations, and manipulation of the yield strength and the ultimate strain (i.e. local material design). One particularly promising application of local material design is in controlling the buckling behaviour during a crash situation by deliberately imposing local soft zones (i.e. thermally induced triggers). The use of an appropriate modelling tool is essential for a successful industrial implementation of such local heating processes. A FE-model with an integrated numerical model for presipitates has been developed, where constitutive equations incorporate the effect of the evolving microstructure on flow stress and work hardening. The model is applied in simulations of induction heating for creating thermally induced triggers and finally to simulate the effect of these triggers on the initial buckling performance of a crash box.

11:10 AM

The Potential of Bake Hardening in Hot Rolled Multiphase Steels: *Heinz Palkowski*[1]; *Thorsten Anke*[1]; [1]Institut for Metallurgie, Collaborative Rsch. Ctr. 362, Robert-Koch-Str. 42, Clausthal-Zellerfeld 38678 Germany

Hot rolled multiphase steel qualities, e.g. Complex Phase steel or Martensite steel, show a clear Bake-Hardening-Effect rising with the degree of prestrain. Using a complex shaped construction unit (e.g. B-pillar) a locally different strength behaviour results after thermal treatment. Fundamental questions have to be answered: - Maximum for the increase of yield and tensile strength at a given prestrain? - Influence given by the kind of prestrain (one and/or biaxial)? - Rise of yield strength as a function of temperature and holding time to a maximum and when is the maximum reached? - Potential of the Bake-Hardening-Effect concerning light weight optimised body structures? The main aspect of the investigation is to determine the influence of the Bake-Hardening-Effect on mechanical properties of a formed construction unit made of multiphase steels manufactured as hot rolled strip. The mechanical-technological properties are analyzed using the Design of Experiments DoE.

11:30 AM

The Role of Non-Metallic Inclusions on the High Temperature Fracture Behavior of Low C, Al-Si Steels: *Edgar Omar Garcia-Sanchez*[1]; Armando Salinas-Rodriguez[1]; Luis Leduc-Lezama[2]; [1]Centro de Investigacion y de Estudios Avanzados del IPN, Metalurgia, Carretera Saltillo-Monterrey km 13, Ramos Arizpe, Coahuila 25900 Mexico; [2]HYLSA-DAP, Ave. Los Angeles 325 Ote, San Nicolas de los Garza, Nuevo León 66452 Mexico

The effect of temperature on the ductility of hot rolled, low C steel plates alloyed with 0.2%wt Al and 0.5%wt Si was investigated by isothermal tensile tests from 850 to 1150°C at a strain rate 5x10-4s-1. It was found that the temperature dependence of the percent reduction of area at fracture exhibits a minimum at 1000°C. Extensive characterization by SEM of fracture surfaces and deformed microstructures near the fracture surface of test samples showed that at temperatures below and above the ductility minimum, fracture takes place by nucleation, growth and coalescence of microvoids at interfaces between ferrite or austenite and (Al, Si)-rich inclusions. Energy dispersive X-ray microanalysis and electron backscattering diffraction were used to characterize the chemistry and structure of these inclusions. The ductility minimum was attributed to a synergistic effect of microvoid formation and strain-induced formation of ferrite films at austenite grain boundaries. The results obtained are discussed in terms of the hot rolling conditions used to manufacture these steels in a Continuous Strip Processing Plant.

11:50 AM

Study on the Cold Formability of Drawn Dual-Phase Steels: *Kyung Soo Park*[1]; Seong Il Hong[1]; Duk Lak Lee[2]; Chong Soo Lee[1]; [1]Pohang University of Science and Technology, Dept. of Matls. Sci. & Engrg., San 31, Hyoja-dong, Nam-gu, Pohang, Gyeongbuk 790-784 S. Korea; [2]Pohang Steel Company, Techl. Rsch. Labs., Koedong-dong, Nam-gu, Pohang, Gyeongbuk 790-600 S. Korea

Non-heat treated steels are attractive in the steel-wire industry since the spheroidization and quenching-tempering treatment are not involved during the processing, reducing the production costs. Dual-phase steel receives a great interest nowadays as a candidate for the non-heat treated steels due to its high hardening capability. Therefore, in this study, the cold formability of dual-phase steels were investigated with the variation of microstructure through different heat-treatment; intercritical quenching (IcQ), intermediate quenching (ImQ)

and step quenching (SQ). The cold formability was studied by estimating the deformation resistance and the forming limit. The deformation resistance, an important factor in determining die life, was estimated by calculating the deformation energy. Also, the forming limit was estimated by measuring the critical strain revealing crack initiation at the notch tip of the specimens. The IcQ-DP steel was found to represent the superior cold formability than other microstructures.

Applications and Fundamentals of High Aspect Ratio Nanomaterials: Inorganic Nanostructures

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Nanomaterials Committee
Program Organizers: Jud Ready, Georgia Tech Research Institute - EOEML, Atlanta, GA 30332-0826 USA; Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; Lourdes G. Salamanca-Riba, University of Maryland, Materials Science and Engineering Department, College Park, MD 20742-2115 USA; Nagarajan Valanoor, Forschungzentrum Juelich, IFF and Institute for Electronic Materials, Juelich, Germany D52425

Tuesday AM Room: 3018
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Jud Ready, Georgia Tech, GTRI-EOEML, Atlanta, GA 30332-0826 USA; Lourdes G. Salamanca-Riba, University of Maryland, College Park, MD 20742-2115 USA; Nagarajan Valanoor, Forschungzentrum Juelich, Juelich D52425 Germany

8:30 AM Opening Remarks

8:35 AM Invited
Semiconducting and Piezoelectric Nanobelts, Nanosprings and Nanorings: *Zhong Lin Wang*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA

Nanowire and nanotube based materials have been demonstrated as building blocks for nanocircuits, nanosystems and nano-optoelectronics. Quasi-one-dimensional nanostructures (so called nanobelts or nanoribbons) have been successfully synthesized for semiconducting oxides of zinc, tin, indium, cadmium and gallium, by simply evaporating the desired commercial metal oxide powders at high temperatures. The belt-like morphology appears to be a unique and common structural characteristic for the family of semiconducting oxides with cations of different valence states and materials of distinct crystallographic structures. Using the technique demonstrated for measuring the mechanical properties of carbon nanotubes based on in-situ transmission electron microscopy, the bending modulus of the oxide nanobelts has been measured, and the nanobelt is shown to be a dual mode nanoresonator for NEMS technology. Field effect transistors and ultra-sensitive nano-size gas sensors, nanoresonators and nanocantilevers have also been fabricated based on individual nanobelts. Thermal transport along the nanobelt has also been measured. Very recently, nanobelts, nanorings and nanosprings that exhibit piezoelectric properties have been synthesized, which are potential candidates for nano-scale traducers, actuators and sensors. The discovery of single-crystal perfect nanorings and its "slinky" growth model will be presented.

9:05 AM Invited
Semiconductor Nanowires as Building Block for Nanoelectronics and Nanophotonics: *Deli Wang*[1]; [1]University of California, ECE Dept., 9500 Gilman Dr., EBU1 3201, La Jolla, CA 92093 USA

Semiconductor nanowires are attractive and versatile building blocks to assemble and electrically interconnect active devices, which enable potential integration of multi-functional components on a single chip. In this presentation, I will first describe LEDs and FET based decoders assembled from bottom-up approach using semiconductor nanowires. The FET based decoders show substantial signal gain enabling signal restoration without external amplification. Second, bistable nanoscale switches based upon core-shell nanowire heterostructures organized in a crossbar architecture have been demonstrated to exhibit well-defined bistability, large ON/OFF ratios and long retention time, and from which the crossed nanowire nonvolatile RAM arrays have been assembled. Furthermore, we have integrated the new decoder with crossed-NW NVRAM arrays. Lastly, epitaxially branched single crystal nanowires were synthesized by a multiple step VLS synthesis with well controlled dimension, density and properties of nanobranches, and demonstrated active nanoscale devices. The implication of these results for nanoelectronics and nanophotonics will be discussed.

9:35 AM Break

10:00 AM Invited
Semiconductor Nanowires and Their Optical Properties: *Peidong Yang*[1]; [1]University of California, Dept. of Chmst., Berkeley, CA 94720 USA

Nanowires are of both fundamental and technological interest. They represent the critical components in the potential nanoscale electronic and photonic device applications. In this talk, we will introduce the vapor-liquid-solid crystal growth mechanism for the general synthesis of nanowires of different compositions, sizes, orientation and doping profile. Particularly, synthesis and organization of different types of heterostructured nanowires will be discussed. Wide band gap semiconductor nanostructures with near-cylindrical geometry and large dielectric constants exhibit two-dimensional ultraviolet and visible photonic confinement (i.e. waveguiding). Combined with optical gain, the waveguiding behavior facilitates highly directional lasing at room temperature in controlled-growth nanowires with suitable resonant feedback. The nanowire optical emission has been studied in detail using high-resolution optical microscopy. The waveguiding behavior of individual zinc oxide (ZnO, GaN) nanowires depends on the wavelength of the emitted light and the directional coupling of the photoluminescence (PL) to the emission dipoles of the nanowire. Pumping at high pulse intensity leads to the transition from spontaneous to stimulated emission, and analysis of the polarization, linewidth, and spacing of the spectral features facilitates identification of the transverse and longitudinal cavity modes and their gain properties. The observation of lasing action in arrayed and isolated ZnO/GaN nanowires without requiring fabrication of mirrors suggests the single-crystalline, well-facetted nanowires can indeed function as effective resonance cavities. This concept of using well-cleaved nanowires as natural optical cavities may be extendable to many other different semiconductor systems.

10:30 AM Invited
Nanowire Formation: Interfacial Morphology and Growth Kinetics: T. Savin[2]; A. Golovin[2]; S. H. Davis[2]; F. Ross[3]; J. B. Hannon[3]; J. Tersoff[3]; C. T. Black[3]; M. C. Reuter[3]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Engrg., 2220 Campus Dr., Cook Hall, Evanston, IL 60208 USA; [2]Northwestern University, Engrg. Sci. & Appl. Math, Evanston, IL 60208 USA; [3]IBM Watson Research Center, Yorktown Heights, NY USA

We examine the vapor-liquid-solid (VLS) process that is used to growth nanowires. Motivated by insitu electron microscopy images of the later stages of Si nanowire growth, we have examined the effects of capillarity on the growth rate of a wire, the composition of the liquid droplet, and the evaporation rate of the liquid into the surrounding atmosphere. Using this information we examine the time rate of change of the volume of a liquid droplet during growth and the growth rate of the wire as a function of wire diameter. The planarity of the solid-liquid interface is extremely important to insure that the composition of the resulting solid is uniform. We examine the morphological stability of the growing solid-liquid interface during VLS growth. We find that the diameter of most wires are less than the critical wavelength for the onset of morphological instability, implying that the solid-liquid interface can remain planar without the presence of faceting.

11:00 AM
In Situ Formed Beta-Ti(Ta) Nanorod Arrays Embedded in Continuous Matrix in a Multicomponent Metallic System: *Guo He*[1]; Masuo Hagiwara[1]; [1]National Institute for Materials Science, Light Matl. Grp., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

When the size of a solid is reduced into nanoscale in one, two or three dimensions, its properties usually deviate from those of "large" crystals and/or glasses with the same average chemical composition. The novel properties of nanometer scale structure have shown their advantages in many applications, such as nanoelectronics, probe microscopy, photonic crystals, molecular sieves, semiconductors and other functional nanostructured materials. So far, many different physical or chemical methods have been successfully used to synthesize or fabricate one-dimensional nanometer scale structure, e.g., nanowires, nanotubes, and nanorods. For example, using sputter deposition to from tungsten nanorods, using arc-discharge to synthesize GaN nanorods, using chemical vapor deposition to synthesize SiC nanorods, etc. The successful synthesis and/or fabrication of one-dimensional nanometer scale structures mainly include C, Si, Ta, ZnO, GaN, SiC, SiN, etc. In this presentation, we describe an in situ formed beta-Ti(Ta) nanorod arrays embedded in continuous matrix in a multicomponent metallic system.

Arsenic Metallurgy: Fundamentals & Applications: Thermodynamics and Pyrometallurgy

Sponsored by: Extraction & Processing Division, EPD-Copper, Nickel, Cobalt Committee, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, LMD/EPD-Recycling Committee
Program Organizers: Ramana G. Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; V. Ram Ramachandran, Scottsdale, AZ 85262-1352 USA

Tuesday AM Room: 2014
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Florian Kongoli, FLOGEN Technologies Inc., Metals Dept., Montreal, QC H3S 2C3 Canada; Trevor M. Bergfeldt, Teck Cominco Metals Ltd., Trail Operations, Trail, BC V1R 4L8 Canada

8:30 AM
Arsenic Removal from a Nickel Concentrate: *Frank Richard Jorgensen*[1]; Terry P. Hall[1]; Steve Sanetsis[1]; [1]CSIRO, Minerals, Bayview Ave., Clayton, Melbourne, Victoria 3168 Australia

The bodies of the future will contain increasing amounts of toxic elements such as arsenic which will pose quality control, environmental and safety issues during downstrem processing. A number of roasting pretreatments were assessed and applied to the treatment of a nickel concentrate containing approximately 0.5% As. The laboratory scale investigation was carried out in a small vibrated bed reactor with additions of sulphur(added either as sulphur vapour or as additional pyrite) or chlorine (added either as elemental chlorine or as chlorine produced by the in situ decomposition of added chlorides such as calcium chloride) to facilitate the decomposition and volatilisation of the arsenic impurity. The pretreatments, which were successful in removing more than 90% of the arsenic from the concentrate, are discussed in terms of the mineralogy.

8:55 AM
Fractional Distribution of Arsenic in the Teniente Continuous Converting Process: *Jonkion Font*[1]; Gerardo Alvear[1]; Alex Moyano[2]; Carlos Caballero[2]; [1]Codelco-Chile, Inst. for Innovation in Mining & Metall., Avenida del Valle 738, Santiago Chile; [2]Codelco-Chile, Div. Codelco Norte, Avenida Tocopilla s/N, Fundición Codelco Norte, Chuquicamata Chile

A thermodynamic model for the distribution of arsenic has been developed for the Teniente Continuos Converting Process, TCCP. The model is based on the quasi equilibrium fractional distribution of As among the copper, white metal, slag and gas phases. The model was validated with the data obtained in pilot and industrial tests performed at the Codelco Norte Smelter of Codelco-Chile. The model demonstrated that can be a very useful tool to predict a-priori the arsenic distribution in different operational configuration of the TCCP.

9:20 AM
Industrial Technologies Related to Arsenic Extraction: *Florian Kongoli*[1]; Robert Budd[2]; Ian McBow[1]; S. Llubani[1]; [1]FLOGEN Technologies Inc., Metals Dept., 5757 Decelles Ave., Ste. 511, Montreal, QC H3S 2C3 Canada; [2]FLOGEN Technologies Inc., 3422 Old Capitol Trail, #791, Wilmington, DE 19808 USA

Arsenic is a particular element whose properties make it equally desirable and undesirable depending on the field of application or the process involved or related to. Arsenic trioxide, one of the common compounds of arsenic is classified as a suspected human carcinogen a cause for certain types of cancers. It has also beneficial effects in other fields such as in wood preservation, agricultural chemicals etc. Similarly arsenic metal is slightly poisonous but it is beneficial since it is primarily used in the manufacture of lead alloys for use in lead-acid batteries. Arsenic is rightly a cause for concern in non-ferrous smelting metallurgy however its extraction as a by products might be beneficial. As a result industrial and feasible technologies for arsenic extraction play an important role. In this paper, in order to have a clear picture of the state-of-the art of the past and present arsenic metallurgy the authors review several industrial technologies used for extraction of arsenic as a by-product or in other ways. Some bench scale technologies or other recently proposed extraction technologies are also reviewed.

9:45 AM
An Approach for Scaling Arsenic Removal from Smelter Gases Using Iron Oxides: *Eduardo V. Balladares*[1]; Roberto F. Parra[1]; Mario M. Sánchez[1]; José G. Palacios[2]; [1]Universidad de Concepción, Metallurgl. Engrg., Edmundo Larenas 285, Concepcion Chile; [2]Universidad de Atacama, Metallurgl. Engrg.

Most of the arsenic contained in copper concentrates is removed in the gas phase during the smelting and converting processes and, according to recent environmental regulations, it is required to fix it in a solid stable form. In this regards, ferric arsenate has been reported as thermal and aqueous stable compound. Therefore, various alternatives for treating gas containing arsenic were considered in this work. From data obtained at laboratory scale on fixation of arsenic using iron oxide, a new process to treat copper concentrates containing arsenic was evaluated. The effect of temperature, oxygen potential and the sample porosity on the fixation of arsenic contained in a gaseous stream was studied, and the iron oxide converted into ferric arsenate was up to 10%. The obtained results suggest a new alternative for gas treatment with an interesting and attractive technical development. The arsenic was retained as ferric arsenate with thermal and aqueous stability, and it allows a safe disposal keeping the environmental restrictions. The kinetics parameters are used to scale up laboratory results to higher processing rates.

Beta Titanium Alloys of the 00's: Phase Equilibria

Sponsored by: Structural Materials Division, SMD-Titanium Committee
Program Organizers: Rod R. Boyer, Boeing Company, Metall./6-20J1, Seattle, WA 98124-2207 USA; Robert F. Denkenberger, Ladish Co., Inc., Cudahy, WI 53110-8902 USA; John C. Fanning, TIMET, Henderson, NV 89009 USA; Henry J. Rack, Clemson University, School of Materials Science & Engineering, Clemson, SC 29634-0921 USA

Tuesday AM Room: Salon 10/11
February 15, 2005 Location: San Francisco Marriott

Session Chairs: Lothar Wagner, TU Clausthal, Inst. of Matls. Sci. & Engrg., Clausthal-Zellerfeld Germany; Paul Bania, TiPro LLC, Boulder City, NV 89005 USA

8:30 AM
Continuous Cooling Transformation Curve for Beta-Processed Ti-6Al-2Sn-4Zr-6Mo: *John E. Matz*[1]; [1]Pratt & Whitney Aircraft Engines, Matls. & Processes Engrg., 100 Aircraft Rd., M/S 403-35, Middletown, CT 06457 USA

The near-beta alloy Ti-6Al-2Sn-4Zr-6Mo is used for aeroengine applications due to its high-strength and thick-section hardening capability. However, this alloy suffers from poor weld ductility and fatigue crack growth properties when welded using conventional techniques. Orthorhombic martensite forms during rapid cooling behind the weld pool. While this phase is soft and ductile, fine interfacial beta precipitates form during the post-weld stress relief treatment, causing embrittlement. Weld development efforts at P&W are focused on preventing orthorhombic martensite formation through optimization of weld thermal history. To provide a starting point for these efforts, the CCT curve for beta-processed Ti-6246 was determined from dilatometric measurements carried out in a Gleeble test machine. This material has a large prior-beta grain size relative to alpha-beta-processed material and can therefore be expected to more closely simulate the phase transformation kinetics of weld metal.

8:55 AM
Predictions of Titanium Alloy Properties Using Thermodynamic Modeling Tools: *Fan Zhang*[1]; Fanyou Xie[1]; Shuanglin Chen[1]; Y. Austin Chang[2]; Dave Furrer[3]; Vasisht Venkatesh[4]; [1]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA; [2]University of Wisconsin, Dept. of Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA; [3]Ladish Co., Inc., PO Box 8902, Cudahy, WI 53110 USA; [4]TIMET, R&D, PO Box 2128, Henderson, NV 89015 USA

Thermodynamic modeling has become essential tools in understanding the effect of alloy chemistry on the final microstructure of a material. Implementation of such tools to improve titanium processing via parameter optimization has resulted in significant cost savings through the elimination of shop/laboratory trials and tests. In this study, a thermodynamic modeling tool developed at CompuTherm, LLC, is being utilized to predict beta transus, phase proportions, phase chemistries, partitioning coefficients and phase boundaries of multi-component titanium alloys. This tool includes Pandat, software for multi-component phase equilibrium calculations, and PanTitanium, a

thermodynamic database for titanium alloys. Model predictions are compared to experimental results for number of beta and near-beta alpha-beta alloys. The effect of the alloying elements, especially the light elements: O, N, H, and C, on beta transus will be discussed.

9:20 AM
A Combinatorial Approach to the Elemental Optimization of a Beta Titanium Alloy Using Directed Laser Deposition: *Peter C. Collins*[1]; Daniel Huber[1]; Rajarshi Banerjee[1]; Daniel J. Evans[2]; Patrick Martin[2]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Dayton, OH USA

The chemistry of the alloy Timetal 21S has been selected as a baseline chemistry for the development of a new high temperature beta titanium alloy. A combinatorial approach has been used to rapidly develop the new alloy. This combinatorial approach consists of employing directed laser deposition of blends of elemental powders in order to rapidly process test alloys of varying compositions. Subsequently, mechanical testing and microstructural characterization of these alloys has been carried out to populate databases that will be used for training and testing fuzzy logic based models for predicting the mechanical properties. These models will also be used to optimize the alloy composition for specific property requirements. In addition to varying the amounts of the base elements (Ti, Mo, Nb, Al, and Si), a variety of additional elements are also being tested as potential alloying additions. These include neutral elements (Zr and Sn), b stabilizers (W), and dispersoid formers (C and B). Based on the results of the coupled mechanical tests and computer models, a new group of alloys for application in high temperature thermal protection systems are being developed.

9:45 AM
Characterisation of the α Phase Nucleation in a Two Phase β-Metastable Titanium Alloy: *Astrid Lenain*[1]; Pascal J. Jacques[1]; [1]Université catholique de Louvain, IMAP, Place Sainte Barbe 2, Louvain-la-Neuve B-1348 Belgium

α / β titanium alloys are best choice materials for the automotive and aerospace industries due to their high performance to density ratio. Among these alloys, the TIMET Ti LCB is more and more used since it presents excellent mechanical properties and a lower cost compared to other Ti alloys. The present study deals with the nucleation and growth of the α phase during several thermomechanical processes. Indeed, distribution and size of the α phase strongly influence the mechanical properties of the resulting microstructures. Several heat treatments were conducted after either cold rolling or annealing. The resulting microstructures were characterised by SEM, EBSD, XRD and TEM, showing a strong influence of the β grain size or deformation level on the conditions of α nucleation.

10:10 AM Break

10:25 AM
Properties of TIMETAL 555 (Ti-5.5Al-5Mo-5V-3Cr): *John C. Fanning*[1]; [1]TIMET, PO Box 2128, Henderson, NV 89009 USA

TIMETAL 555 is a high strength near-beta titanium alloy designed for improved producibility and excellent mechanical property combinations, including deep hardenability. The nominal chemical composition of TIMETAL 555 is Ti-5Al-5Mo-5V-3Cr (weight percent). This paper provides a summary of available data for this relatively new alloy.

10:55 AM
Influence of Thermo-Mechanical Treatments on Microstructure and Mechanical Properties of Near Beta Titanium Alloy VST55531: *J. Panter*[1]; A. Dalloz[1]; Karl-Heinz Rendigs[2]; *Nathalie Hellard*[3]; Wegmann Gerhard[3]; [1]EADS CCR, Ctr. Commun de Recherches Louis Bleriot, 12, Rue Pasteur, BP 76, 95152 Suresnes, Cedex France; [2]Airbus Deutschland, Abt. ESWG, Hünefeldstraße 1-5, 28199 Bremen Germany; [3]Airbus-France, M0122/4 ESW-T, 316, Rte. de Bayonne, 31060 Toulouse cedex 03 France

Ti-5Al-5Mo-5V-3Cr-1Zr (VST55531) is a beta Titanium alloy recently developed in the frame of a collaboration between AIRBUS and VSMPO. For the introduction of this new alloy on an airplane, a static sized part on the new AIRBUS A380 Engine Pylon has been chosen. In order to increase its use at AIRBUS, and especially to produce in the future damage tolerance sized forged parts with a excellent compromise between strength and damage tolerance resistance, a study has been carried out to increase our knowledge on the relationship between thermo-mechanical treatments, microstructure and mechanical properties of this new alloy. To do so, as received rolled and forged materials, forged in the beta phase field material and forged in the alpha + beta phase field material have been used. On all the different combinations of raw material + heat treatment, microstructure and mechanical properties have been assessed. Microstructures have been characterized using OM and FEG-SEM while mechanical properties have been characterized by tensile tests and fracture toughness tests. The results of this extensive characterization will be discussed in this paper.

11:20 AM
Modeling Property-Microstructure Relationships in Ti-5Al-5Mo-5V-3Cr-1Fe (TIMETAL-5553): *Megan Harper*[1]; Rajarshi Banerjee[1]; Daniel J. Evans[2]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Dayton, OH USA

One of the more recently developed beta titanium alloys which is promising for a number of different applications is the Ti-5Al-5Mo-5V-3Cr-1Fe alloy, commercially referred to as TIMETAL-555. This alloy exhibits wide variations in microstructure resulting from different heat-treatments. The microstructural evolution and resultant mechanical properties of this alloy have not yet been explored in great detail. As-received, alpha+beta processed Ti-555, was subjected to a number of heat treatments with the objective of studying the microstructure and phase evolution in this alloy and obtain widely varying microstructures. Heat-treatments have been carried out in both the beta as well as the alpha+beta phase fields. The typical heat-treatment consisted of solutionization in the beta phase field or the alpha+beta phase field, cooling at different rates, and a second ageing treatment in the alpha+beta phase field. The microstructure at each stage of the heat-treatment cycle has been characterized in detail using SEM and TEM based studies. These microstructures will be quantified using rigorous stereological procedures and the corresponding room temperature tensile properties will be measured. The resulting database will be used to train and test a fuzzy-logic neural-network model that will allow for the prediction of the mechanical properties from the microstructure. The model will also be used to develop functional dependencies of the mechanical properties on the microstructural features.

Biological Materials Science and Engineering: Biological Materials/Bio-Medical Applications I

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Society for Biomaterials, Surfaces in Biomaterials Foundation, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), EMPMD/SMD-Biomaterials Committee
Program Organizers: Marc Andre Meyers, University of California, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Tuesday AM Room: 3009
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sungho Jin, University of California, Matls. Sci. & Engrg., La Jolla, CA 92093-0411 USA; Subra Suresh, Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139-4307 USA

8:30 AM Invited
Molecular Biomimetics: Materials Science and Engineering Using Genetically Engineered Proteins: *Mehmet Sarikaya*[1]; [1]University of Washington, Matls. Sci. & Engrg., Roberts Hall, Box 352120, Seattle, WA 98195 USA

Physical and chemical functions of organisms are carried out by a very large number (billions) of proteins through predictable and self-sustaining interactions. Using biology as a guide, we design, synthesize, genetically tailor and utilize short polypeptides as molecular linkers and erectors in self-assembly, ordered organization, and fabrication of hybrid systems. Our objectives are accomplished in four focused areas: Selection of inorganic-binding short (7-15 amino acids) polypeptides using combinatorial biology protocols using metals, oxides and semiconductors; Quantitative assessment of the nature of binding and assembly of polypeptides on inorganics using experimental (SPR, QCM, AFM, NMR, TOF-SIMS) and modeling (molecular dynamics) approaches; Nanoassembly using functionalized designer biomolecules (chaperonins, phages, and S-layers, DNA), and electrochemically nanopatterned substrates; Creation of synthetic/biological molecular hybrids and inorganics using polypeptides in functional materials and

systems for potential utility in a wide variety of applications in nano- and nanobio-technology. Research supported by ARO-DURINT.

9:00 AM Keynote
Single-Cell Nanomechanics and Human Disease States: *Subra Suresh*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg. & Div. of Biologl. Engrg., Rm. 8-309, Cambridge, MA 02139-4307 USA

The mechanical response of living cells and subcellular cytoskeleton can undergo dramatic alterations due to biochemical changes introduced by the progression of human diseases. In this presentation, we provide experimental results on systematic alterations to the elastic properties of human red blood cells parasitized in vitro by Plasmodium falciparum malaria. By recourse to optical tweezers experiments, we extract direct force versus displacement relationships for the cell and examine contributions to cell elasticity from specific proteins transported to the membrane from the parasite. Continuum and molecular-level computational simulations of the deformation of red blood cell are also performed to quantify the nanomechanics of cell response. The mechanical properties of changes to cell deformability from P. falciparum infestation are also compared and contrasted with similar results for the P. vivax parasite. Finally, the similarities and differences in cell elasticity and disease states between malaria and human pancreatic cancer are also examined.

9:45 AM Invited
Protein Self-Assembly Creates a Nanoscale Device for Biomineralization: *Malcolm l. Snead*[1]; Michael L. Paine[1]; Wen Luo[1]; [1]University of Southern Californa, Ctr. for Craniofacial Molecular Bio., 2250 Alcazar St., CSA Rm. 109, Los Angeles, CA 90033 USA

The outer-most covering of teeth, enamel, rarely undergoes catastrophic failure despite a lifetime of repeated loading, in a wet-, acidic-, bacteria-laden-environment. Enamel forms when specialized cells, ameloblasts, synthesize proteins that self-assemble to form an enamel extracellular organic matrix that is competent to control the initiation, rate of growth and habit of the inorganic crystallites. The physical properties of mature enamel stem from the regulation imposed by a self-assembly process at the nanoscale level of organic matrix components. The physical properties of the enamel composite ceramic depend entirely on the number of genes expressed, the timing of their expression, their ability to self-assemble and the stoichiometry among the various types of enamel matrix proteins. Evolutionary change in the organization of the enamel is a reflection of the addition, diminution or change in timing for the expression of a gene and the protein it encodes being recorded during tooth development.

10:15 AM
Using DNA for Formation of Photonic Crystals and Introducing Controlled Defects: *Harris Marcus*[1]; [1]University of Connecticut, Dept. of Matls. Sci. & Engrg., Chmst. Dept, Storrs, CT 06269-3136 USA

Photonic crystals provide an innovative way for controlling light propagation. Light in photonic crystals can be trapped by a point defect in the crystal or can be channeled by a series of voids, a one dimensional waveguide. To create a two-dimensional photonic crystal, polystyrene particles were assembled in close-packed array on a glass substrate by DNA hybridization. The approaches used to create these crystals will be described with the critical impact of the DNA on the formation and subsequent creation of controlled defects. Using a continuous wave single beam laser, polystyrene particles were released to create the defects. For pattern particle release, holographic techniques will be described.

10:35 AM Break

10:50 AM Invited
Titanium Oxide Nanotubes with Controlled Morphology for Enhanced Bone Growth: Brian Oh[1]; *Sungho Jin*[1]; [1]University of California, Dept. of Mechl. & Aeros. Engrg., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA

Orthopedic and dental implants frequently utilize titanium and its alloys because of their bio-compatibility and desirable mechanical properties. We have studied nanoscale surface structures of titanium oxide with various crystal structures (including anatase) and morphologies. An array of vertically aligned titanium oxide nanotubes with ~80 nm diameter has been synthesized, which is firmly attached onto the solid Ti implant surface as a three-dimensional nanoscale structure. It is found that such a nanotube structure with a very large surface area significantly accelerates the formation of hydroxypaptite phase which is also in the nano regime. Additional nano-inspired nanostructures were also investigated. SEM and TEM microstructural analysis and possible formation mechanisms of titanium nanotubes and calcium phosphate nanostructure will also be discussed.

11:20 AM
Functionalized Magnetic Nanoparticles for Early Breast Cancer Detection: *Jikou Zhou*[1]; Challa S.S. R. Kumar[2]; Carola Leuschner[3]; Josef Hormesa[2]; *Wole O. Soboyejo*[1]; [1]Princeton University, Dept. of MAE & PRISM, E-Quad, Olden St., Princeton, NJ 08540 USA; [2]Louisiana State University, Ctr. for Advd. Microstructures & Devices, Baton Rouge, LA 70806 USA; [3]Pennington Biomedical Research Center, 6400 Perkins Rd., Baton Rouge, LA 70808 USA

Breast cancer is the second leading cause of cancer death in women. At the time of diagnosis using current methods and techniques, 34-40% of breast cancer patients already have occult metastases. These suggest that it is extremely critical and important to develop new techniques for early detection. However, the sensitivity of magnetic resonance imaging (MRI) is not efficient for early cancer detection using current method. We developed a novel approach to send MNPs into cancer tissues, so that MRI contrast is significantly enhanced. Human breast cancer cells express receptors for luteinizing hormone releasing hormone (LHRH) and can be specifically targeted by compounds linked to LHRH. By conjugating these ligands to MNPs, they can be targeted to the cancer cells, and incorporated into the cancer cells through receptor mediated endocytosis. Using this method, early detection of breast cancer would be improved, thus treatment efficiency could be significantly increased.

11:40 AM
Biological Effects of Nanoparticulate Materials: *K. F. Soto*[1]; A. Carrasco[2]; T. G. Powell[2]; L. E. Murr[1]; K. M. Garza[2]; [1]University of Texas, Dept. of Metallurgl. & Matls. Engrg., El Paso, TX 79968 USA; [2]University of Texas, Dept. of Biologl. Scis., El Paso, TX 79968 USA

The use of chrysotile asbestos nanotubes and fibril bundles in a variety of composite blends, including cement and plastics continues to be somewhat pervasive world-wide. For example, at the end of the 20th Century more than 2 million tons of asbestos were processed annually. By contrast, plastic composite development using multi-walled carbon nanotubes, which started around 1995, began to approach 1000 tons of carbon nanotubes processed annually at the same time; and the use of nanoparticulate materials is currently escalating dramatically. Both chrysotile asbestos and multi-wall carbon nanotubes have been treated as toxic materials commercially. Epidemiologic and animal studies have indicated that inhalation of asbestos can result in pulmonary fibrosis, lung cancer, and mesothelioma. In work to be reported herein cytotoxicity assays using a murine lung macrophage cell line have shown that a variety of commercial single wall and multi-walled carbon nanotubes and aggregate morphologies exhibit the same relative toxicity as chrysotile asbestos. In fact, a host of nanoparticulate materials exhibit the same levels of toxicity while characterization of these nanoparticulates by transmission electron microscopy exhibit a variety of nanoaggregate morphologies ranging from fibrils and fibril bundles to complex, branched, fractal-like aggregates of spherules. These include commercial carbon black, TiO2, Si3N4, Al2O3, Fe2O3, ZrO2, and nanoparticulate Ag. Silver is also demonstrated to be more cytotoxic than asbestos, but Ag is also a well-known bactericide; and current ointments utilizing Ag or Ag compound nanoparticulates have remarkable properties in treating wounds and certain skin diseases.The implications for nanotechnology developments and especially nanoparticulate materials processing will be discussed in terms of their parallel with the historical and world-wide asbestos trade developments and health concerns. The atmospheric occurrence of anthropogenic carbon nanotubes will also be briefly discussed in this context. Finally, it will be emphasized that in spite of the fact that nanoparticulate materials may be toxic, this is certainly not a reason to impose a moratorium on their production or use. The important issue is to recognize the necessity for precautions so as not to repeat the materials mistakes of the past. Research supported in part by a University of Texas System Louis Stokes Alliance for Minority Participation (LSAMP) Bridges to Doctorate Fellowship (KFS), RCMI Grant G12RR008124 (AC,TGP,KMG) and Mr. and Mrs. MacIntosh Murchison Endowed Chair (LEM).

12:00 PM
Synthesis and Characterization of Functionalized Magnetic Biomaterials: *Raju V. Ramanujan*[1]; L. Wong[1]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Block N4.1, Nanyang Ave., Singapore 639798 Singapore

Magnetic materials can be used for a variety of bioengineering applications, including cell separation, immunoassay, magnetic resonance imaging (MRI), drug and gene delivery, minimally invasive surgery, radionuclide therapy, hyperthermia etc. It is often essential to

coat the particles with suitable materials, such coatings can provide increased functionality compared to the uncoated particles. The synthesis and characterization of magnetic powders, followed by coating with dextran and gold will be described. SEM, TEM, XRD, EDX and VSM techniques were used in this investigation. Iron oxide particles were synthesized by the reverse micelle technique and coated with dextran. Gold-coated iron nanoparticles were also synthesized, the gold coating was found to be effective in protecting the particles from oxidation. The optimum coating parameters were determined and the magnetic properties of the coated and uncoated powder were determined. The investigation demonstrated the versatility of the coating techniques in the production of functionalized magnetic biomaterials.

Bulk Metallic Glasses: Fatigue and Fracture

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA

Tuesday AM Room: 3006
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Reinhold H. Dauskardt, Stanford University, Matls. Sci. & Engrg., Stanford, CA 94305 USA; Gary Harlow, Lehigh University, Mechl. Engrg. & Mech., Bethlehem, PA 18015 USA

8:30 AM
High Cycle Fatigue in Zr-Based Bulk Metallic Glass: Damage Initiation and Growth: *Brian Christian Menzel*[1]; Reinhold H. Dauskardt[1]; [1]Stanford University, Matl. Sci. & Engrg., 416 Escondido Mall, Bldg. 550, Rm. 551A, Stanford, CA 94305 USA

A high-cycle stress-life fatigue study was conducted on a Zr-based bulk metallic glass to elucidate the anomalously low endurance limits that have been reported. Distributed damage was observed to initiate very early in the fatigue process as either shear bands or mixed mode surface cracks. Damage initially grew under mixed mode loading conditions in the maximum shear stress direction. On reaching a characteristic size, they abruptly changed orientation and continued to grow as mode I cracks. A focused ion beam was used to introduce a well-defined distributions of initial defects to systematically elucidate damage initiation and growth processes. High-resolution techniques were used to characterize the effect of defect size, shape and orientation on damage initiation and the early stages of damage growth. The effect of stress state was investigated using cylindrical specimens under uniaxial compression and uniaxial tension-compression loading. Surface modification techniques including coating/anodization and e-beam re-melting were investigated as ways to improve surface quality, remove damage initiation sites from the material and improve fatigue life.

8:50 AM
Free Volume and Mechanical Properties of Zr-Cu-Al: *Yoshihiko Yokoyama*[1]; Peter K. Liaw[2]; R. A. Buchanan[2]; A. Inoue[3]; [1]University of Hyogo, Matl. Sci. & Engrg., Shosha 2167, Himeji 671-2201 Japan; [2]University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [3]IMR Tohoku University, Katahira, 2-1-1, Sendai 980-8875 Japan

Relative difference of free volume can be estimated by density measurement using same rod shape bulk glassy alloys (BGAs). In order to decide the standard (as crystalline state), we use ideal solution with close packed structure as mixed faced centered cubic and hexagonal close packed structures. We define the free volume to be a volume expansion ratio from the ideal solution to glassy state. Therefore, the free volume as cast state can be divided into two factors, one is the minimum required free volume for amorphization, and the other is the excess free volume. The origins of the strength and toughness in Zr-Cu-Al ternary BGAs are considered as Zr-Al networks and excess free volume, respectively. A Wöhler curve of Zr50Cu40Al10 BGAs, which composition is close to ternary eutectic point, indicates two drawbacks, i.e., low fatigue limit and significant decrease in fatigue strength in the cycle range of 10^3 to 10^4. However, these drawbacks can be improved by addition of Pd element, which can promote excess free volume and nano-network structure formation. The Zr50Cu37Al10Pd3 BGA indicates a significant increase of fatigue limit over 1 GPa.

9:10 AM
Testing-Volume Effects on the Fatigue Lifetimes of a Zr-Based Bulk Metallic Glass: *William Hutchison Peter*[1]; Peter K. Liaw[1]; C. T. Liu[2]; Raymond A. Buchanan[1]; Mark L. Morrison[1]; Gong Yao Wang[1]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA

After a decade of fabricating "bulk metallic glasses" (BMGs), some fundamental concepts and observations regarding the fracture mechanisms of these glasses have been made. However, a full understanding of the degradation process of BMGs during fatigue has not been realized. Early fatigue studies of Zr-Based BMGs have revealed large variabilities between fatigue-endurance-limits of various testing procedures. While four-point-bend studies of BMGs have resulted in low fatigue-endurance-limits, tensile-tensile fatigue testing of radial notched specimens has resulted in fatigue-endurance-limits comparable or higher than conventional high-strength crystalline materials. Changes in the volume of BMG material that experiences the maximum stress state may cause the variation in lifetime results. The present research will address this issue by studying the lifetimes of tensile fatigue specimens with various testing volumes, and comparing the results. The location and mechanism of crack initiation will also be addressed. This research effort was made possible by the funding of the National Science Foundation Integrative Graduate Education and Research Training (IGERT) Program on "Materials Lifetime Science and Engineering" (DGE 9987548) with Drs. L. Clesceri, W. Jennings, and L. Goldberg as Program Directors; and by the Division of Materials Science and Engineering, Department of Energy under contract DE-AC-00OR22725 with Oak Ridge National Laboratory (ORNL) operated by UT-Battelle, LLC.

9:30 AM
Cyclic Fatigue Damage Zones and Anomalously High Fatigue Crack Growth Rates in Zr-Based Bulk Metallic Glass: *Peter A. Hess*[1]; Reinhold H. Dauskardt[1]; [1]Stanford University, Matls. Sci. & Engrg., 416 Escondido Mall, Bldg. 550, Rm. 551A, Stanford, CA 94305 USA

Zr-based bulk metallic glass (BMG) is susceptible to fatigue processes, however, the fatigue crack growth mechanisms remain largely unknown. The relaxation effect of time-dependent overloading and block loading on subsequent fatigue crack growth was measured. Results indicate a damaged and/or relaxed zone of material ahead of the fatigue crack tip. Anomalously high growth rates and low fatigue thresholds were measured following overloading and at low testing temperatures. This rapid growth was associated with fracture surface features that initiate from a point and widen in a V-shaped manner as the crack propagates. The features were reminiscent of those found in some amorphous polymers, and are associated with simultaneous cracking on multiple planes. It is analogously proposed that the features in BMG are a result of multiple shear band formation ahead of the fatigue crack tip. The role of multiple crack planes on BMG fatigue mechanisms was examined.

9:50 AM
Molecular Dynamics Investigation of Deformation and Fatigue of an Amorphous Metallic Alloy: *Kimberly K. Cameron*[1]; Reinhold H. Dauskardt[1]; [1]Stanford University, Matls. Sci. & Engrg., 416 Escondido Mall, Bldg. 550, Rm. 555F, Stanford, CA 94305 USA

A molecular dynamics study was conducted on a four component, Lennard-Jones amorphous solid in order to understand the atomistic mechanisms of deformation and fatigue. The deformation behavior observed was typical of that seen experimentally involving both elastic and plastic strains, load history dependence and strain rate sensitivity. Changes in free volume and excess strain were monitored during deformation to demonstrate how the stress state affects the distribution of free volume and how regions with excess free volume will preferentially deform. Under monotonic shear loading, the system developed a shear band in the region with the highest free volume. The same system developed a shear band under much lower stress under cyclic fatigue loading. This fatigue behavior can be related to the notion of directional shear transformation zones. Once a particular cluster of atoms undergoes a shear transformation, it does not return to its original state on unloading. Furthermore, in subsequent cycles although this cluster will not transform again, the surrounding region is more likely to have further shear transformations. This localization of the deformation process accelerates the shear banding process and is used to rationalize the poor fatigue properties commonly reported for metallic glasses.

10:10 AM Break

10:30 AM

Influence of Composition and Structure on Fatigue Behavior of Zr-Based Bulk Metallic Glasses: *Gongyao Wang*[1]; P. K. Liaw[1]; Y. Yokoyama[2]; M. Freels[1]; D. L. Weinberg[1]; B. Yang[1]; W. H. Peter[1]; R. A. Buchanan[1]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996 USA; [2]University of Hyogo, Matls. Sci. & Engrg., Shosha 2167, Himeji City Japan

High-cycle fatigue (HCF) experiments were conducted on zirconium (Zr)-based bulk- metallic glasses (BMGs): $Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Ni_{10}Al_{10}$, and $Zr_{50}Cu_{37}Al_{10}Pd_3$, in atomic percent. The X-ray diffraction results show that $Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Ni_{10}Al_{10}$, and $Zr_{50}Cu_{37}Al_{10}Pd_3$ have an amorphous structure. However, $Zr_{50}Cu_{37}Al_{10}Pd_3$ contains much more free volumes and some nano structures. The HCF experiments were performed using an electrohydraulic machine at a frequency of 10 Hz with an R ratio of 0.1 and under tension-tension loading, where $R = \sigma_{min.} / \sigma_{max.}$, $\sigma_{min.}$ and $\sigma_{max.}$ are the applied minimum and maximum stresses, respectively. The test environments were air and vacuum. A high-speed and high-sensitivity thermographic-infrared (IR) imaging system has been used for the nondestructive investigation of the temperature evolution during fatigue experiments of BMGs. Limited temperature evolution was observed during fatigue. A sparking phenomenon was observed at the final fracture moment of $Zr_{50}Cu_{30}Ni_{10}Al_{10}$. However, no sparking phenomenon was found at the final fracture moment of $Zr_{50}Cu_{40}Al_{10}$ and $Zr_{50}Cu_{37}Al_{10}Pd_3$. The vein pattern and droplets with a melted appearance were observed in the apparent melting region. The fatigue lives in vacuum are longer than those in air. The fatigue-endurance limit of $Zr_{50}Cu_{37}Al_{10}Pd_3$ was found to be significantly greater than those of $Zr_{50}Cu_{40}Al_{10}$ and $Zr_{50}Cu_{30}Ni_{10}Al_{10}$, which indicates that the inclusions of Pd that increased the free volume and nano structures improve the fatigue resistances of the Zr-based BMGs. The fracture morphology indicates that fatigue cracks initiate from the outer surface of the sample. A mechanistic understanding of the fatigue behavior of the Zr-based BMGs is suggested.

10:50 AM

An Approach to Modeling the S-N Behavior of Bulk-Metallic Glasses: *D. Gary Harlow*[1]; Peter K. Liaw[2]; William H. Peter[2]; Gongyao Wang[2]; Raymond A. Buchanan[2]; [1]Lehigh University, Mechl. Engrg. & Mech., 19 Memorial Dr. W., Bethlehem, PA 18015 USA; [2]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996-2200 USA

Current approaches to modeling the S-N behavior of materials are primarily empirical based on statistical analyses of experimental data. Consequently, they do not adequately reflect long-term performance. Furthermore, they do not identify the key sources and extent of their contributions to randomness arising from microstructure, environment, or loading. An approach for modeling the S-N response using a standard fatigue crack growth model is proposed. The model captures the variability in fatigue lives by relating it to key material variables, both deterministic and random, that are readily identified in the proposed model. The identification and significance of these variables are paramount for predicting fatigue crack growth and the subsequent damage evolution. The effectiveness of this approach is demonstrated with an amalgamated set of S-N data for bulk-metallic glasses. Variability associated with manufacturing and material variables are considered. Adoption of this approach is recommended for sound scientific and probabilistic life prediction.

11:10 AM

Fatigue Behavior of an Amorphous Steel: *Gongyao Wang*[1]; P. K. Liaw[1]; J. Poon[2]; M. W. Freels[1]; D. L. Weinberg[1]; D. Qiao[1]; R. A. Buchanan[1]; C. R. Brooks[1]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996 USA; [2]University of Virginia, Dept. of Physics, Charlottesville, VA 22904 USA

A non-magnetic amorphous steel, $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ (atomic percent), generally has a three-times greater strength than the conventional steel. Four-point-bend fatigue experiments were conducted on the amorphous steel. Specimens were cycled under a load control using an electrohydraulic machine at a frequency of 10 Hz (sinusoidal waveform) with an R ratio of 0.1, where $R = \sigma_{min.} / \sigma_{max.}$, $\sigma_{min.}$ and $\sigma_{max.}$ are the applied minimum and maximum stresses, respectively. The test environment was in a laboratory air. $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ exhibited a high fatigue-endurance limit (694 MPa). However, the stress versus number of fatigue cycles curve of $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ revealed that the lifetime decreased abruptly with increasing the tensile stress at higher stress levels. The fatigue behavior of Fe-Based and Zr-Based BMGs and compared. The fatigue-endurance limit of $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ was about 5 times greater than that of the Zr-based bulk-metallic glass (BMG). However, at high stress levels, the amorphous steel demonstrated a shorter fatigue life than the Zr-based BMG. The fracture morphology indicated that $Fe_{48}Cr_{15}Mo_{14}Er_2C_{15}B_6$ has a brittle fracture. Fatigue cracks initiated from the outer tensile surface of the specimen, and the fatigue-crack-propagation region was very small. The mechanisms of fatigue-crack initiation and growth are suggested.

11:30 AM

Fatigue Behavior of a Cu-Based Bulk Metallic Glass: *Matthew W. Freels*[1]; Peter K. Liaw[1]; Gongyao Y. Wang[1]; Devin L. Weinberg[1]; Q. S. Zhang[2]; Z. Q. Hu[2]; Raymond A. Buchanan[1]; [1]University of Tennessee, Matls. Sci. & Engrg. Dept., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA; [2]Chinese Academy of Science, Shenyang Natl. Lab. for Matls. Sci., Inst. of Metal Rsch., 72 Wenhua Rd., Shenyang 110016 China

If bulk metallic glasses (BMGs) are to find uses in structural applications, it is important to understand their fatigue behavior. Initial fatigue studies of BMGs showed lower fatigue strengths than were anticipated. However, more recent fatigue studies of Zr-based BMGs have shown much higher fatigue strengths than the initial results. To our knowledge, no fatigue studies have been performed on Cu-based BMGs. In this study, four-point-bend fatigue experiments were conducted on (Cu60Zr30Ti10)99Sn1 (atomic percent) BMGs in air. The fatigue experiments were performed using an electrohydraulic machine under a load control mode at frequencies of 1 and 10 Hz (sinusoidal waveform) with an R ratio of 0.1, where $R = \sigma_{min.}/\sigma_{max.}$, and $\sigma_{min.}$, and $\sigma_{max.}$, are the applied minimum and maximum stresses, respectively. The results are compared to similar conventional crystalline alloys. The present work is supported by the National Science Foundation (NSF), the Combined Research-Curriculum Development (CRCD) Program, under EEC-9527527 and EEC-0203415, the Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, and the International Materials Institutes (IMI) Program, under DMR-0231320, with Ms. M. Poats, and Drs. P. W. Jennings, L. S. Goldberg, L. Clesceri, and C. Huber as contract monitors.

11:50 AM

A Comprehensive Synopsis of Fatigue Behavior in Zr-Based Bulk-Metallic Glasses: *Devin Lance Weinberg*[1]; Matthew Webster Freels[1]; Gongyao Y. Wang[1]; Dongchun Qiao[1]; William H. Peter[1]; Peter K. Liaw[1]; Raymond Allen Buchanan[1]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA

A great deal of research has been performed throughout the materials science field in recent years to quantify and understand the high-cycle fatigue (HCF) behavior of zirconium (Zr)-based bulk-metallic glasses (BMGs). However, the published results remain scattered in various academic journals ensuring that comparisons and trends are difficult to analyze. Data from a variety of HCF experiments on Zr-based BMGs as well as common crystalline alloys was combined and organized by testing method and graphed as stress range and stress amplitude/tensile strength versus cycles to failure. Although it is difficult to make true comparisons, given variations in material composition, sample geometry, and testing procedure, several interesting trends were observed, which present a clear opportunity for future fatigue studies. One such trend appears to be decreasing endurance limits as the frequency of the HCF experimental loading is reduced. Furthermore, the comparison implies that four-point bend HCF experiments yield lower endurance limits than axial fatigue tests, which could be contrary to commonly accepted results in the well-studied crystalline alloys. The present work is supported by the National Science Foundation (NSF), the Combined Research-Curriculum Development (CRCD) Program, under EEC-9527527 and EEC-0203415, the Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, and the International Materials Institutes (IMI) Program, under DMR-0231320, with Ms. M. Poats, and Drs. P. W. Jennings, L. S. Goldberg, L. Clesceri, and C. Huber as contract monitors.

Cast Shop Technology: Aluminum Melting: Furnace Design and Refractories

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday AM Room: 2001
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Paul King, Albany Research Center, Albany, OR 97321 USA; Subodh K. Das, Secat Inc., Ctr. for Al Tech., Lexington, KY 40511 USA

8:30 AM
An Analytical Furnace Model for Optimizing Aluminum Melting Furnaces: *Tianxiang Li*[1]; Paul King[2]; Mohamed Hassan[1]; Kazunori Kuwana[1]; Kozo Saito[1]; [1]University of Kentucky, Dept. of Mechl. Engrg., 151 RGAN Bldg., Lexington, KY 40506-0108 USA; [2]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave., SW, Albany, OR 97321-2198 USA

An analytical furnace model, originally established by Essenhigh and Tsai, is developed in this paper. The practical application of this model is to predict optimum furnace operation and was verified by experiments conducted in the Experimental Research Furnace (ERF) at Albany Research Center of the U.S. Department of Energy. The development of the Essenhigh/Tsai model is based on a few melting and holding tests with two main assumptions; the thermal conduction loss in aluminum melting is the same for both melting and holding processes, and the heat loss through the flue gases is lineally proportional to the melting rate. The former is reasonable because the thermal conduction loss is small as compared with the firing rate, while the later is quite accurate as shown in the test results. Tests of aluminum melting were conducted on the ERF furnace in the cases of high, normal and low roof heights. From the tests, the relations between firing rate, heat absorption rate, melting rate, and energy efficiency were developed, and the optimum operation conditions under which the maximum energy efficiency can be achieved were predicted. In addition, the effect of roof height on the energy efficiency was determined. This model could be a valuable tool in diagnostic analysis of day-to-day operations in aluminum melting.

8:55 AM
Experimental Investigation on Stress-Corrosion of Refractories Exposed to Liquid Aluminum: Roger Pelletier[1]; *Claude Allaire*[1]; Vincent Ébacher[1]; [1]École Polytechnique de Montréal, Engrg. Physics, CIREP, Campus CRIQ, 8475, Christophe-Colomb, Montréal, Québec H2M 2N9 Canada

The interaction between stress and corrosion is a well known phenomenon observed in many environment/material engineering systems. This paper presents the results of a study whose objective is to verify if stress-corrosion interactions are possible between liquid aluminum and refractories. To observe the possible interaction, specimens are submitted to a creep flexion test while being partially submerged into liquid aluminum. Pure corrosion and pure creep tests are used as references to put forward the interactions. The results obtained so far suggest that tensile stresses enhance corrosion of refractories.

9:20 AM
Scale Modeling of Aluminum Melting Furnace: Sita Rama Raju S. Penmetsa[1]; *Tianxiang Li*[1]; Paul King[2]; Kozo Saito[1]; [1]University of Kentucky, Dept. of Mechl. Engrg., 151 RGAN Bldg., Lexington, KY 40506-0108 USA; [2]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave., SW, Albany, OR 97321-2198 USA

Secondary (recycled) aluminum constitutes around 35% of the total aluminum used in the United States. Secondary aluminum melting is accomplished in large reverberatory furnaces, and improving its energy efficiency has been of a major interest to the aluminum industry. To assist the aluminum industry in improving the melting efficiency, an experimental research furnace (ERF as a prototype) of about 907 kg (2000 lbs) has been built at Albany Research Center of the U.S. Department of Energy as part of multi-partner research program. This paper describes the scaling laws of aluminum melting furnace, the design of a small scale model furnace (14 kg capacity), and the scale model experiments. A partial scale modeling technique was applied to derive achievable scaling laws on temperature distributions in and at the furnace walls including the roof. Temperature distributions obtained for the small scale model furnace was favorably compared with prototype. Well agreement of the results from model experiments with these from tests on ERF validates that we can use the scale modeling technique to infer the physics on the melting processes of industrial furnaces.

9:45 AM
Combustion Space Modeling of an Aluminum Furnace: *Brian Mark Golchert*[1]; Chenn Q. Zhou[2]; Antoine Quenette[1]; Quinyou Han[3]; Paul E. King[4]; [1]Argonne National Laboratory, Energy Systems, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Purdue University Calumet, Mechl. Engrg., 2200 169th St., Hammond, IN 46323-2094 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., One Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6083 USA; [4]Albany Research Center, 1450 Queen Ave., SW, Albany, OR 97321 USA

Secondary aluminum production (melting from aluminum ingots, scraps, etc.) offers significant energy savings and environmental benefits over primary aluminum production since the former consumes only five percent of the energy used in the latter process. The industry, however, faces technical challenges of further improving furnace melting efficiency and has been lacking tools that can help understand combustion process in detail and that will facilitate furnace design. Computational Fluid Dynamics (CFD) modeling has played increasingly important roles in evaluating industrial processes. As part of a larger program run by SECAT, a CFD model has been developed at Argonne National Laboratory to simulate fuel combustion, heat transfer (including thermal radiation), gaseous product flow (mainly CO2 and H2O), and production/transport of pollutant species/greenhouse gases in an aluminum furnace. Using this code, the surface heat fluxes are calculated and then transferred to a melt code. In order to have a high level of confidence in the computed results, the output from the code will be compared and validated against in-furnace measurements made in the Albany furnace. Once validated, the combustion code may be used to perform inexpensive parametric studies to investigate methods to optimize furnace performance. This paper will present results from the combustion modeling of an aluminum furnace as well as results from several parametric studies.

10:10 AM Break

10:20 AM
Potentials for Increasing Efficiency of Aluminium Melting Furnaces: *Jan Migchielsen*[1]; Jan D. de Groot[1]; [1]Thermcon Ovens B.V., Process Dept., PO Box 97, Geldermalsen 4190 CB The Netherlands

The introduction of heat recovery on aluminium melting furnaces has lead to a considerable reduction of the specific fuel requirement for the melting of aluminium. Since already a considerable time now, melting furnaces equipped with regenerative burners are generally the most economic solution for melting solid metal. With the Kyoto treaty and the increased cost for energy, new incentives are created to further reduce the specific energy cost for melting aluminium. Operating a furnace in a "smart" way by optimizing charging procedures and fine-tuning existing burner systems, does achieve additional energy saving. A smarter operation can also be incorporated into the furnace control system. Dynamically adjusting the air-fuel ratio and optimizing the regenerative burner setting will reduce energy cost and increase productivity. The introduction of a hybrid type furnace with both regenerative burners and high velocity burners to optimize heat transition will lead to a further reduction of the fuel cost for specific applications. This paper addresses the latest developments on the field of aluminium melting furnaces to achieve both by design and smart operation of the furnaces lower specific fuel consumption, minimized maintenance and balanced productivity.

10:45 AM
Design and Operation of an Experimental Reverberatory Aluminum Furnace: *Paul E. King*[1]; Michael C. Hayes[1]; Tianxiang Li[2]; Qingyou Han[3]; Mohamed Hassan[2]; Brian M. Golchert[4]; [1]U. S. Dept. of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA; [2]University of Kentucky, Dept. of Mechl. Engrg., 151 RGAN Bldg., Lexington, KY 40506 USA; [3]Oak Ridge National Laboratory, One Bethel Valley, Oak Ridge, TN 37831-6083 USA; [4]Argonne National Laboratory, Process Simulation & Modlg., 9700 S. Cass Ave, ES/362, Argonne, IL 60439 USA

The U. S. Dept. of Energy, Albany Research Center, in cooperation with industrial support through Secat, Inc. has designed, built and is operating a test-bed reverberatory furnace. Studies in the Albany Research Center (ARC) experimental reverberatory furnace (ERF) include melt efficiency as a function of combustion space volume, power input and charge alloy. This paper details the furnace design, experimental equipment, conditions, procedure, and measurements and in-

cludes results and discussions of melt efficiency research. Specific results reported include an analysis of the overall efficiency of the furnace as a function of power input and the effect of changing the combustion space volume has on the melting efficiency. An analytic analysis of the theoretical efficiency of the furnace is carried out to determine overall characteristics of the furnace. Experimental data is utilized to validate numerical (computational fluid dynamics) predictions.

Characterization of Minerals, Metals and Materials: Characterization of Structural Engineering Materials - II

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee
Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

Tuesday AM Room: 2010
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Jian Li, Natural Resources Canada, CANMET-MTL, Ottawa, Ontario K1A 0G1 Canada; Louis Evrard, Universite Catholique de Louvain, Physl. Chmst. & Matls. Engrg., Louvain-la-Neuve Belgium

8:30 AM
A Comparative Evaluation of Precipitation Behavior in Equivalent Strength V-Nb-Ti and V-Containing Microalloyed Steels During Processing of Structural Beams: *Sankaran Shanmugam*[1]; Devesh K. Misra[1]; Dhiren Panda[2]; Fulvio Siciliano[3]; [1]University of Louisiana, Cheml. Engrg. Dept., Matls. Sci. & Engrg. Grp., PO Box 44130, Lafayette, LA 70504-4130 USA; [2]Nucor-Yamato Steel, PO Box 1228, 5929 E. State Hwy. 18, Blytheville, AR 72316 USA; [3]Reference Metals Company, 1000 Old Pond Rd., Bridgeville, PA 15017 USA

The paper describes a comparative evaluation of the precipitation behavior of V-Nb-Ti and only V-containing steels to investigate the effective cumulative role of the addition of Nb-Ti to V-steel. Transmission electron microscopy was employed to characterize the precipitates in equivalent strength V-Nb-Ti and vanadium-containing steels. While the mechanical properties are similar, there are significant differences in the precipitation behavior of the two steels. The microstructure of V-Nb-Ti steels consists of predominantly polygonal ferrite, fine pearlite, and a small % of bainite. Titanium forms coarse cuboidal carbonitrides, and niobium and vanadium form very fine precipitates of carbonitrides. The former tends to preferentially precipitate along the grain boundaries, whereas the latter are dispersed in the matrix. The niobium and vanadium carbonitrides cause ordering of precipitates in the ferrite lattice, which is displayed by the diffraction pattern as chemically sensitive or superlattice reflection. Additionally, niobium carbonitrides exhibit a ring SAD pattern. The different precipitates exhibit an orientation relationship with the matrix and their partitioning is not observed in the pearlitic or bainitic ferrite. The bainite region is characterized by a complete precipitation of cementite. In general, the microstructure is free of dislocations except at the interface of the ferrite/pearlite.

8:55 AM
The Precipitation Behaviors of Inconel 740 and Super 304H for Ultra Supercritical Application: *Quanyan Wu*[1]; Vijay K. Vasudevan[1]; John Shingledecker[2]; Robert Swindeman[2]; [1]University of Cincinnati, Cheml. & Matls. Engrg., 2624 Clifton Ave., Cincinnati, OH 45221 USA; [2]Oak Ridge National Laboratory, Ceram. & Metals, Bldg. 4500S, MS6155, PO Box 2008, Oak Ridge, TN 37831 USA

New alloys have been developed to meet higher temperature demands for higher thermal efficiency and lower emission in coal power plants. Inconel 740 is a new nickel-base superalloy which has demonstrated good thermal stability and strength up to 750°C. The major precipitates were MC, $M_{23}C_6$, and gamma prime when solutionized at 1050°C. Eta precipitates formed a Widmanstatten pattern when creep tested, in addition to gamma prime rafting. A modified version of Inconel740 showed reduction of eta phase and better creep strength. The advanced austenite stainless steel Super 304H also showed excellent long-term strength, and microstructural study revealed massive precipitation of fine-scale copper-rich precipitates. These precipitation behaviors, after ageing and creep-test under a series of temperatures will be characterized using TEM, EDS, SEM and comparison between these two alloys will be presented.

9:20 AM
Characterization of the Inhibition Layer on Hot Dip Galvanized and Galvannealed Steels: S. Dionne[1]; *J. Li*[1]; V. Guertsman[1]; G. A. Botton[2]; F. E. Goodwin[3]; [1]CANMET-Materials Technology Laboratory, 568 Booth St., Ottawa, Ontario K1A 0G1 Canada; [2]McMaster University, Dept. of Matls. Sci. & Engrg., 1280 Main St. W., Hamilton, Ontario L8S 4M1 Canada; [3]ILZRO, 2525 Meridian Pkwy., PO Box 12036, Rsch. Triangle Park, NC 27709-2036 USA

An iron aluminide inhibition layer is formed on the steel surface during continuous hot dip galvanizing. On a galvanized product, this inhibition layer must be thick enough to prevent the formation of iron-zinc intermetallics and to obtain a ductile coating. In the case of galvannealed products, the galvanizing parameters are adjusted to obtain a thinner inhibition layer which is broken down during the galvannealing treatment to allow interdiffusion of iron and zinc. An understanding of the effects of process parameters and substrate characteristics on the coating microstructure is critical to optimize the coating performance. In the present study, the microstructure of galvanized coatings produced on a variety of steel types under conditions typical of industrial continuous galvanizing was examined using a combination of advanced characterization techniques.

9:45 AM
Characterization of Deterioration in a Shunting Locomotive for Lifetime Assessment: *Jeongguk Kim*[1]; Jong-Duk Chung[1]; Jang-Sik Pyun[1]; Yong-Ki Hong[1]; Dae-Sung Bae[2]; [1]Korea Railroad Research Institute, Railroad Safety Rsch. & Testing Ctr., 360-1 Woulam, Uiwang, Kyunggi 437-757 S. Korea; [2]Hanyang University, Dept. of Mechl. Engrg., Seoul 426-791 S. Korea

The deterioration of a shunting locomotive was characterized for the lifetime assessment. The locomotive was using for shunting works in steel industry, and in this paper, various types of technical evaluation methods for the locomotive parts were introduced. Unlike other rolling stocks in railway applications, the diesel shunting locomotive is composed of major components such as diesel engine, transmission, gear box, brake system, etc., which cover more than 70 percent of the total price of the locomotive. Therefore, in this investigation, each part of major components in the diesel locomotive was analyzed in terms of the degree of deterioration. The performance evaluation tests were performed on the diesel engine and gear box, and precision inspection was conducted on each part of transmission to provide the current wear information. Also, corrosion test and nondestructive evaluation techniques were employed to demonstrate the wear status of coachwork and bogie parts in locomotive. In this investigation, several engineering characterization techniques for the lifetime analysis of a shunting locomotive were applied and presented.

10:10 AM Break

10:20 AM
Development of Heat Resistant Cast Alloy for Engine Exhaust Manifold: *Sung Hwan Park*[1]; *Jong Moon Kim*[1]; *Hak Jin Kim*[1]; *Se Jin Ko*[1]; Hyoun Soo Park[1]; Jong Dae Lim[1]; [1]Hyundai-Motor Company, R&D Ctr./Matl. Rsch. Team, Jangduk 772-1, Whasung, Kyunggi 445-706 S. Korea

The heat resistant cast alloy has been developed for exhaust manifold of new passenger diesel engines. Operating demands on exhaust manifold have increased significantly over the past decade because of higher exhaust gas temperature by tighter emission requirement, improved fuel efficiency and design toward higher specific engine power. This has led to much higher elevated temperature strength and oxidation resistant demands on exhaust manifold alloy. Besides, thermal fatigue occured directly as a result of thermal expansion and mechanical constraint, becomes an important issue. Our reserach work focuses on alloy design to improve durability of engine exhaust manifold. Low cycle fatigue and high temperature oxidation properties are evaluated. Casting design and condition are optimized by numerical simulation for better manufacturing.

10:45 AM
AISI 304 K Steel Subjected to Small Charge Explosions: Microstructural Changes with Limited or No Macro-Deformation: *Donato Firrao*[1]; Paolo Matteis[1]; Giorgio Scavino[1]; Graziano Ubertalli[1]; Maria G. Ienco[2]; Paolo Piccardo[2]; Maria R. Pinasco[2]; Enrica Stagno[2]; Roberto Montanari[3]; Maria Elisa Tata[3]; Giovanni Brandimarte[4]; S. Petralia[4]; [1]Politecnico di Torino, Dip. di Scienza dei Materiali e

Ingegneria Chimica, Corso Duca degli Abruzzi, 24, Torino 10129 Italy; [2]Università di Genova, Dip. di Chimica e Chimica Industriale, Via Dodecanneso, 30, Genova 16146 Italy; [3]Università di Roma "Tor Vergata", Dip. di Ingegneria Meccanica, Via del Politecnico, 1, Roma 00133 Italy; [4]Marina Militare, Istituto di Chimica Esplosivi, Viale S. Bartolomeo n. 400, La Spezia 19100 Italy

Microstructural variations induced on AISI 304 K steel disks by explosions which do not cause gross macro-deformation are illustrated. 50 and 100 g NSP explosive spherical charges and explosive-target distances in the range from 0.05 to 0.8 m were used to achieve pick pressures in the 960 to 3 bars range. Two grain sizes (60 and 30 μm) were tested. Microstructural features were studied by XRD, OM, SEM and STM microscopy. Surface OM and SEM evidenced dark spots, due to oxidation and deposition of explosive components, zones with recrystallization phenomena, grain boundary melting, and twins. Phenomena in the interior of the samples restricted to twins. They can be seen up to some distance from the explosion impinged surface and again for a smaller distance around the reflecting surface. The maximum charge-to-target distance at which the phenomena disappear has been singled out for each charge and grain size and related to the critical resolved shear stress for twinning.

11:10 AM

Textural Inhomogeneities in Drawn and Annealed OFHC Copper Wire: *D. R. Waryoba*[1]; P. N. Kalu[1]; [1]FAMU-FSU College of Engineering, Dept. Mechl. Engrg., 2525 Pottsdamer St., Tallahassee, FL 32310-6046 USA

Textural inhomogeneities have been investigated in oxygen free high conducting (OFHC) copper wire drawn at room temperature to a true strain of 2.31, and isothermally annealed at 250°C and 750°C for annealing times ranging from 10 s to 1 hr. Local orientations were mapped by means of orientation imaging microscopy (OIM). The microtexture of the drawn wires showed a strong <111> + weak <100> duplex fiber texture at the inner core, whereas the mid and surface regions had a comparatively weak texture. Annealing at 250°C resulted into a recrystallization which originated from the mid section, proceed towards the surface regions and ends in the inner core. Recrystallization resulted into a strong<100>+weak<111> duplex fiber texture. A similar textural inhomogeneity was observed during short annealing at 750°C. However, prolonged annealing gave rise to abnormal grain-growth that proceed from the inner core to the outer surfaces with a dominant <111> fiber component at the inner region and mixed components of <111>, <100>, and <112> at the outer surfaces.

11:35 AM

HRTEM Charaterization of Ni-Ni_3Ti Eutectic Alloy: *Y. Sikaddour*[1]; K. Taibi[1]; [1]Université des Sciences et de la Technologie Houari Boumedienne (USHB), Faculté de Génie Mécanique & Génie des Procédés, Dépt. des Scis. des Matériaux, BP 32 EL Alia Bab, Ezzouar 16000 Alger

Ni-Ni_3Ti eutectic alloy as cast exhibit a widmanstätten structure in the nickel matrix, it has been found that depending on cooling rate and chemical composition. The precipitates are thin plates of the hexagonal phase $Ni_3Ti(Do_{24})$. Ni-Ni_3Ti eutectic alloy has been prepared by unidirectional solidification in order to investigate a widmanstätten structure in FCC Nickel phase. Analysis of HRTEM images has demonstrate in Do_{24} structure, the presence of misfit dislocation with cores associated with very small ledges with a height of four close-packed plans spacing and its burgers vectors is $(-1/6)[112]_{Ni}$ parallel to the interface facets.

Characterization of Minerals, Metals and Materials: Characterization of Light Weight Materials - I

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee

Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

Tuesday AM — Room: 2012
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: Roderick I.L. Guthrie, McGill University, McGill Metals Procg. Ctr., Montreal, Quebec H3A 2B2 Canada; Michael J. McKelvy, Arizona State University, Ctr. for Solid State Sci., Tempe, AZ 85287-1704 USA

8:30 AM

Characterization of the S-Phase with a Modified Orientation Relationship: *Libor Kovarik*[1]; Stephen A. Court[2]; Michael J. Mills[1]; [1]Ohio State University, 2041 College Rd., Watts Hall #477, Columbus, OH 43210 USA; [2]Alcan Technology & Management, Badische Bahnhofstrasse 16, Neuhausen CH 8212 Switzerland

Two possible orientation relationships (OR) are known for the S-phase in Al-Cu-Mg alloys. It has been previously shown that one of the two OR is metastable and found only for S-phase particles that do not exceed certain dimensions. In our work on alloys with low Cu/Mg ratio and small additions of Si, we employed HRTEM and Z-contrast imaging to study the origin of the metastable OR. As compared to the previously published explanation based on an invariant line concept, we find that the OR may be governed instead by a particular S-phase/matrix interface that has excellent atom-site matching for the metastable OR considered. Microscopy observations are supported by a model interface that shows this atom-site matching. Based on EDX analysis, we also find that the creation of metastable OR may be facilitated by the local enrichment of Si.

8:55 AM

Study of Deformation of Cube-Textured Aluminum Using Laser-Induced Photoelectron Emission: *Mingdong Cai*[1]; Lyle E. Levine[2]; J. T. Dickinson[1]; [1]Washington State University, Physics Dept., Pullman, WA 99164 USA; [2]National Institute of Standards and Technology, Metall. Div., 100 Bureau Dr., Gaithersburg, MD 20899 USA

Uniaxial deformation of a cube-oriented aluminum sample was monitored by a laser-induced photoelectron emission technique. A retarding-field energy analyzer was used to determine the energy distribution of photoelectrons. Due to anisotropy of the surface work function in aluminum, electrons of 0.5 eV (using 248-nm laser radiation) were found to dominate the photoelectron energy spectra indicating a strong cube texture. After deformation, obvious increases in the intensities of photoelectrons with 0.7 and 1.0 eV kinetic energies were detected, which correspond to increases of {111} and {110} surface area. Quantitative texture analysis by electron backscattered diffraction (EBSD) data showed an increase of {110} surface area after deformation. Laser-induced photoelectron emission is sensitive to slip events and possible grain rotation, while EBSD demonstrates texture evolution as a result of slip and lattice rotation. The higher energies observed in the photoelectron spectra are consistent with the measured evolution in texture during deformation.

9:20 AM

Nucleation and Early Growth of Fatigue Cracks in 2024-T3 Aluminum Alloy: *Jonathan Tsang*[1]; Ali Merati[2]; [1]Carleton University, Dept. of Mechl. & Aeros. Engrg., 1125 Col. By Dr., Ottawa, Ontario K1S 5B6 Canada; [2]National Research Council of Canada, Inst. for Aeros. Rsch., 1200 Montreal Rd., Bldg. M-13, Ottawa, Ontario K1A 0R6 Canada

While the long crack regime has been well established and understood, our understanding of the mechanisms controlling fatigue crack nucleation and short crack growth are still under debate. Crack nucleation and short crack growth consume up to ~95% of the fatigue life. Therefore, early crack growth is of great importance and must be fully understood before an accurate holistic and materials-based life prediction model can be developed. An extensive study has been carried out to evaluate the main factors affecting the fatigue crack nucleation process and to characterize the growth of physically short cracks. In

this work, different crack detection and monitoring techniques such as surface replication, marker bands, and electrical potential drop have been used. It was observed that almost all the fatigue failures originated at constituent particles. Early crack growth patterns were documented and a novel hypothesis for the short crack mechanism was presented.

9:45 AM
Characterization of Al-SiC Particulate Composite by Using Digital Image Analysis for Three-Dimensional Reconstruction of Microstructural Volume: *Harpreet Singh*[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Schl. of Matls. Sci. & Engrg., 771 Ferst Dr., NW, Atlanta, GA 30318 USA

Al-SiC metal matrix composites (MMC's) find applications in various fields ranging from automotive, aerospace to electronics (i.e. structural to functional material). The key factors of this MMC that make it useful for the wide variety of applications are light weight aluminum and SiC particles which provide mechanical stability to the composite. In this paper, we present a technique to reconstruct the three dimensional microstructural volume of the Al-SiC MMC from serial sections and characterize three samples with different processing parameters. A total of hundred sections, approximately 1 mm apart, were used to reconstruct the 3D volume. The 3D reconstruction is utilized to characterize local particle morphologies, particle connectivity, and near neighborhood spatial heterogeneities of microstructure in three-dimensional space.

10:10 AM Break

10:20 AM
Interface Reaction of Boron Carbide in Aluminum Matrix Composites and its Control: *X. Grant Chen*[1]; [1]Alcan International Limited, Arvida R&D Ctr., 1955, Mellon Boul., PO Box 1250, Jonquière, Québec G7S 4K8 Canada

Aluminum matrix composites containing reinforcing particles of B4C have been used in certain high-performance applications such as neutron-shielding components and aircraft and aerospace structures. B4C is a desirable reinforcement because of its special capability to capture neutrons, lightweight and superior stiffness and strength. However, further use of the B4C particle-reinforced composite material is limited due to its complicated manufacturing and cost. One of the major challenges is the interface reaction between B4C and Al that is detrimental to the castability and material properties. The present paper describes work conducted to better understand the interface reaction during the liquid mixing process and the resulting cast structure. The instability of B4C in liquid aluminum and its reaction products were investigated using thermodynamic calculation, optical metallography and scanning electron microscopy. A novel method is presented for controlling the interfacial reaction by forming an in-situ barrier layer using a Ti addition in molten aluminum.

10:45 AM
Characterization of Subsurface Damage Accumulation in Aluminum Alloys Subjected to Multiple Asperity Sliding Contacts: *S. S. Akarca*[1]; W. J. Altenhof[1]; A. T. Alpas[1]; [1]University of Windsor, Dept. of Mechl., Auto., 401 Sunset Ave., Windsor, ON N9B 3P4 Canada

In certain tribological applications of aluminum, sliding wear surfaces involves large plastic deformation and subsurface damage, even under light loads. In the subsurface layers adjacent to the contact surfaces, the competition between the plastic strain, which enhances void growth, and the hydrostatic pressure, which suppresses it, is considered to be responsible for the generation of a damage gradient. On the basis of this concept, debris formation can be attributed to the delamination of the subsurface layers at a certain depth where the damage accumulation rate is highest. Numerical analyses are necessary to determine the magnitude and distribution of the hydrostatic pressure and hence determine the subsurface damage gradient under various loading conditions. Accordingly, the main objective of this study is to build a finite element model to analyze subsurface deformation that occurs in aluminum alloys subjected to sliding contact. An Eulerian model capable of accounting for large strain accumulation under multiple asperity contact situation was employed. The effects of friction, normal load and distance between the asperities on multiple asperity contacts were investigated. Numerical results were tested by normal contact experiments.

11:10 AM
Experimental Study and Modelling of Phase Transformation in 3003 Aluminium Alloys During Homogenisation: *Moukrane Dehmas*[1]; Mickael Serriere[1]; Pierre Archambault[1]; Elisabeth M. Aeby-Gautier[1]; Charles-Andre Gandin[1]; [1]LSG2M, ENSMN, Parc de Saurupt, Nancy Cedex 54042 France

This paper aims to study microstructural evolutions in the 3003 aluminium alloys during the heating from the as-cast state. After solidification, both the solid solution and the primary precipitates are far from equilibrium and during heating, a precipitation of dispersoïds and an eutectoïd transformation of the primary particles occur. Quantitative experimental results, obtained by high energy synchrotron radiation and by image analysis, are compared to in-situ electrical resistivity and to a precipitation model. In a first time, the different quantitative methods are analysed (synchrotron results, and MET quantitative image analysis) and lead to evolutions of the volume fraction of dispersoïds and primary particles as a function of temperature. In a second time, the evolution of the electrical resistivity are compared to the manganese content in the solid solution predicted by a precipitation model. This manganese content evolves by the fine precipitation and then, by the enrichment of the primary particles.

11:35 AM
An Electron Microscope Study of Mechanical Twinning and Fracture in Two-Phase Intermetallic Titanium Aluminides: *Fritz Appel*[1]; [1]GKSS Research Centre, Inst. for Matls. Rsch., Geesthacht D-21502 Germany

Deformation twinning is an important mode of plastic deformation in gamma-TiAl based titanium aluminide alloys. The mechanism apparently compensates for the lack of independent slip systems that can operate at comparable stresses and, thus, supports the plasticity of polycrystalline materials. On the other hand, the octahedral planes of gamma -TiAl serve as slip planes, twin habit planes and cleavage planes. Thus, blocked slip may easily lead to fracture. This complex association of twinning and fracture is investigated in the present paper by conventional and high-resolution electron microscope investigations of twinning arrangements. The major areas of the study are: (i) twin nucleation and propagation, (ii) effects of solutes and precipitates on the kinematics and dynamics of twin propagation, (iii) association of mechanical twinning and fracture. The implications of the results will be discussed with respect to alloy design strategies towards improved mechanical properties.

Computational Aspects of Mechanical Properties of Materials: Nano-Scale and Meso-Scale Modeling

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA

Tuesday AM Room: 3012
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: M. I. Baskes, Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA; K. S. Ravi Chandran, University of Utah, Metallurgl. Engrg., Salt Lake City, UT 84112 USA

8:30 AM Invited
A Finite-Temperature, Dynamic Coupled Atomistic/Discrete-Dislocation Model: *W. A. Curtin*[1]; V. Shastry[1]; M. Dewald[1]; R. Miller[2]; [1]Brown University, Div. of Engrg., Providence, RI USA; [2]Carleton University, Dept. of Mechl. Engrg., Ottawa Canada

To handle plastic deformation at multiple scales, a computational method has been developed wherein atomistic and continuum regions communicate across a coherent boundary while dislocations seamlessly pass through the boundary, changing from an atomistic to continuum description or vice-versa. Here, the zero-temperature quasistatic method is extended to include finite-temperature molecular dynamics and dislocation dynamics. This is achieved through the use of "stadium boundary conditions" in the atomistic region that simultaneously provide a temperature reservoir for the atoms and prevent reflection at the atom/continuum boundary of energetic pulses emanating from the atomic region. The new method is applied to several basic problems, including brittle and ductile crack growth in Al, Ni, and Fe at finite temperatures, and dislocation pile-up/grain-boundary interactions. These examples demonstrate the power of integrated multiscale modeling for capturing complex mechanical phenomena in solids. Finally, progress on related multiscale models that embed quantum mechanics into the atomistic region and that connect discrete dislocation or

impurity models into continuum crystal plasticity or diffusion, respectively, is reported. [1]L. Shilkrot, R. M. Miller, and W. A. Curtin, J. Mech. Phys. Solids 52, 755 (2004).

9:05 AM
Modeling Elastic and Plastic Deformations of Nanocrystalline Materials: Peter Stefanovic[1]; Mikko Haataja[1]; *Nikolas Provatas*[1]; [1]McMaster University, Dept. of Matls. Sci. & Engrg., 1280 Main St. W., Hamilton, Ontario L8S4L7 Canada

A continuum field theory approach is presented for modeling elastic and plastic deformation, free surfaces and multiple crystal orientations in systems with both hexagonal and cubic symmetry. The model is based on a free energy, and its dynamics incorporates both diffusive and elastic phenomena. By introducing a variable elastic time scale, we are able to maintain mechanical equilibrium while simulating microstructural evolution on time scales well beyond those accessible by conventional atomistic simulation methods (e.g. molecular dynamics). We apply this model to elucidate the role of dislocations and to study texture evolution during deformation of nanocrystalline materials.

9:25 AM Invited
Modeling Dislocation Behavior in Multi-Layer Nanostructures: *Nasr M. Ghoniem*[1]; [1]University of California, Mechl. & Aeros. Engrg., 420 Westwood Blvd., Los Angeles, CA 90095-1597 USA

Understanding dislocation behavior in multi-layer nanostructures is critical to the design of ultra-strong nano-structured materials, which exhibit significant ductility. We present the results of recent research on dislocation properties in anisotropic multilayer thin films utilizing several computational techniques. First, newly developed methods of dislocation dynamics in these systems will be introduced. Such methods are applied to delineate the various modes of material deformation as a function of the nanolayer size and the applied external stress. Second, Molecular Dynamics simulations will be presented, where we focus on the determination of core properties as dislocations approach interfaces, and on the critical Koehler barrier, which determines the resistance of the interface to dislocation transmission. Finally, an extension of the Peierls-Nabbarro dislocation core model is used together with Ab Initio calculations of the gamma surface to describe the structure of dislocation loop cores under a wide variety of conditions.

10:00 AM
Precipitate Shape and Coherency Loss Mechanisms in Ni-Ag Alloys: *Peihua Jing*[1]; Jae-Hyeok Shim[1]; Brian D. Wirth[1]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA

In precipitate hardened materials, the strength and creep properties are controlled by dislocation interactions, which depend on the precipitate interfacial structure. We present the results of atomistic simulations specifically designed to investigate the precipitate shape, coherency loss and dislocation bypass mechanisms in a Ni-Ag alloy with a large lattice misfit. Embedded atom method interatomic potentials have been re-derived to accurately describe the elastic properties of Ni and Ag, and the Ni-Ag mixing enthalpy. The results provide insight into the precipitate coherency loss mechanisms and the effect of precipitate coherency on dislocation interaction and bypass.

10:20 AM Break

10:30 AM
Crack-Tip Fields from Discrete Dislocation Simulations: A Comparative Study with Continuum Mechanics: *Silvester J. Noronha*[1]; Nasr M. Ghoniem[1]; [1]University of California, Mechl. & Aeros. Engrg. Dept., 420 Westwood Plaza, #48-121, Los Angeles, CA 90095-1597 USA

Crack-tip plastic behavior is simulated using two dimensional discrete dislocation dynamics simulations. 3D mechanisms are incorporated in 2D dislocation dynamics simulations of crack-tip plasticity to include the effect of dislocation reactions like dislocation annihilation, dislocation junction formation, dynamic source generation, etc. We find that the size and shape of the equilibrated plastic zone compares well with the continuum mechanics predictions. The stress fields ahead of the crack along the crack plane are in excellent agreement with the small scale yielding models of crack tip plasticity. In contrast to the continuum models in which strain hardening exponent are an input parameter, in our simulations the hardening behavior evolves naturally. The variation of the hardening exponent with temperature is also recovered.

10:50 AM
Microstructural Effects in Modeling the Flow Behavior of Single Crystal Superalloys: *Y. S. Choi*[1]; T. A. Parthasarathy[1]; D. M. Dimiduk[2]; M. D. Uchic[2]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA

The [001] orientation deformation of a single-crystal superalloy (CMSX-4) having a high volume fraction of regularly-arrayed cuboidal γ' precipitates was simulated using a non-local gradient-dependent plasticity for the γ matrix and the anisotropic-elasticity for the γ' precipitate. Unit-cell meshes were adopted for representation of the γ-γ' microstructure. Numerical studies focused on clarification of the effects of the γ-γ' geometry, the deformation constraint induced by the γ-γ' geometry, the applied BCs, and the flow properties of the γ and γ' phases on the macroscopic flow behavior. The simulated flow curves showed a softening behavior, which was accompanied by the organized massive plastic flow in the γ matrix and influenced by the thickness of γ-matrix channels, the flow property of the γ matrix, and the geometry of the γ'-precipitate edge. The simulation results also showed a tension-compression (T-C) asymmetry, which was related to the geometric constraint of the γ-γ' unit cell.

11:10 AM
Modeling the Tensile Properties of Alpha/Beta Titanium Alloys: *Sujoy Kar*[1]; Thomas Searles[1]; Eunha Lee[1]; Jaimie Tiley[2]; Gopal Babu Viswanathan[1]; Rajarshi Banerjee[1]; Hamish L Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Materials Laboratory, Matls. & Mfg. Direct., Dayton, OH USA

The development of a set of computational tools that permit microstructurally-based predictions for the tensile properties of commercially important titanium alloys, such as Ti-6Al-4V, is a valuable step towards the accelerated maturation of materials. This paper will discuss the development of Fuzzy Logic and Neural Network Models based on Bayesian statistics to predict the yield strength, ultimate tensile strength and elongation of Ti-6Al-4V at room temperature. The development of such rules-based models requires the building up of extensive databases, which in the present case are microstructurally-based. The steps involved in database development include controlled variations of the microstructure using novel combinatorial approaches to heat-treatments, the use of standardized stereology protocols to rapidly characterize and quantify microstructural features, and mechanical testing of the heat-treated specimens. These databases have been used to train and test Neural Network models to predict the tensile properties. In addition, these models have been successfully used to identify the influence of individual microstructural features on the mechanical properties, consequently guiding the efforts towards development of more robust phenomenological models.

11:30 AM
Prediction of the Microstructure Parameters of Heavily Deformed OFHC Copper Using Artificial Neural Network: *Jamaa Bouhattate*[1]; Daudi R. Waryoba[1]; Peter N. Kalu[1]; [1]FAMU-FSU College of Engineering, Natl. High Magnetic Field Lab., Dept. of Mechl. Engrg., 2525 Pottsdamer St., Tallahassee, FL 32310 USA

An artificial neural network (ANN) model has been developed for predicting key microstructural parameters in heavily deformed OFHC copper. The initial phase of the model centered on the correlation between the processing parameters and the microstructure of the deformed and annealed OFHC copper. The material was heavily drawn to different strains at room temperature, and annealed for one hour at various temperatures. The ANN model was based on multilayer backpropagation neural network, and was trained by using experimental and numerical data. The input parameters were annealing temperatures and the processing strains, while the output parameters were orientation texture, microhardness, grain size and recrystallization temperatures. A good correlation was found between the predicted results and the original training data. Also, the model was used to predict properties of OFHC copper at different temperatures for different strains outside the training data set.

11:50 AM
Modeling the Mechanical Response of Open-Cellular 6101-T6 Foams at Ambient and at Intermediate Temperatures Through Finite Element and Density Compensation Methods: *Ian Ivan Nieves*[1]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., 916 Engrg. Tower, Irvine, CA 92697-2575 USA

The tensile elastic response exhibited by open cellular 6101-T6 Al foams (Duocell) at 293K, 423K and 523K was simulated through finite element (FE) analysis. These results were compared with previously obtained experimental results and with values obtained from a density compensation (DC) model. FE analysis was performed with MSC Patran preprocessing software using Nastran post-processing. These simulations used a staggered cubic cell geometry that was comprised of hourglass-shaped struts with morphologies similar to those observed through optical and SEM metallography. The simulated cells approximated the relative density and dimensions possessed by the actual foam cells.

Longitudinal and transverse geometries were produced by scaling the appropriate cell dimensions by the experimentally determined aspect ratio. Tetrahedral meshes and numerical values for Al properties were then applied to the cell geometries. Boundary conditions appropriate to plane straining and loads equivalent to those at various stages of tensile testing were then applied to the meshes. Values of the resulting cellular elastic modulus (E*), were then compared with those obtained from the experimental data and with those derived from the DC model. The longitudinal results displayed significantly greater stiffness than the corresponding transverse results as expected. FE, DC and experimental results for 293K were all observed to correspond closely to each other. However, both the FE and experimental values displayed significantly greater stiffness than predicted by the DC values at 423K and 523K. The 523K FE and experimental results also differed greatly from the corresponding values obtained at other temperatures. Comparisons of FE results at all testing temperatures and inspection of the metallographic results indicated this discrepancy is due in part to the greater contribution of localized shear failure at elevated temperatures.

12:10 PM
Dynamic Behavior of IF Steel in the Two-Phase Conditions: *Janusz Majta*[1]; Anna K. Zurek[2]; [1]AGH University of Science and Technology, Metall. & Matl. Sci., Mickiewicza 30, Krakow 30-059 Poland; [2]Los Alamos National Laboratory, MST-8, MS: G755, Los Alamos, NM 87544 USA

There is increasing interest to study dynamic behavior of materials at high temperatures and non stable multi-phase conditions. In order to examine the failure behavior of materials tested in the two-phase conditions, the IF steel heated to austenite-ferrite region was employed. Present study is a continuation of previous analysis of C-Mn steel. Very unexpected results obtained in spall test of A36 steel forced us to continue investigations for the case of IF steel where role of carbon on mechanical response is minimalized. A series of high rate forming experiments are also performed on microalloyed and low carbon steels using SHPB. We evaluated the effects of variations in austenite/ferrite phase ratio during loading in the material flow stress and microstructure, followed by the optical, scanning and TEM microscopy analysis. The impact experiments resulted in obtaining various microstructures and localization of the deformation. However, in each test the absence of failure is observed after shock loading of material in austenite-ferrite two-phase condition. The importance of obtaining such results is very important for computer simulations of dynamic response of non stable two-phase materials. The results and conclusions of the study could be directly employed in estimating the different contribution to the shock-induced plastic deformation without failure in other two- or multi-phase materials.

Computational Thermodynamics and Phase Transformations: Grain Boundaries and Interfaces II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Corbett C. Battaile, Sandia National Laboratories, Materials and Process Modeling Department, Albuquerque, NM 87185-1411 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Tuesday AM Room: 3005
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Mark Andrew Miodownik, King's College London, Mechl. Engrg., Strand, London WC2R 2LS UK

8:30 AM Invited
Growth of Special Texture Components During Grain Growth: Simulation & Theory: *Anthony D. Rollett*[1]; [1]Carnegie Mellon University, MSE, 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The growth of minority texture components during the annealing of deformed metals is a phenomenon that has yet to be fully understood. Many theories have been advanced to explain the origins, for example, the cube component, {001}<100>, in fcc metals. One such theory concerns "microselection" whereby the cube component is favored during the early stages of recrystallization where coarsening takes place in the subgrain structure formed during recovery. To address this possibility, Monte Carlo simulation of grain growth is used to study the behavior of the cube component during grain growth in a polycrystal for which the dominant texture is representative of rolling textures in fcc metals. The cube component was treated as a special texture component, inserted into the microstructure by assigning near-cube orientations to particular grains. A sensitivity analysis varied such parameters as initial cube volume fraction, the grain boundary energy and mobility functions, spatial correlation of the special component, and rotations of the special component away from the exact cube position. The results indicate a strong tendency for the cube component to grow provided that a moderate level of anisotropy exists in the grain boundary energy and mobility. However, long-range correlations in orientation also play an important role in constraining growth in majority texture components, which is an effect known as orientation pinning.

9:00 AM Invited
An Inherently Discrete Approach for Modeling Microstructural Evolution with Elastic Effects: Cubic Anisotropy and Grain Coarsening Influenced by Elastic Stress: *Mark T. Lusk*[1]; T. Schacht[1]; E. Buddy Damm[2]; J. Wei[2]; Elizabeth A. Holm[3]; [1]Colorado School of Mines, Mechl. Engrg. Prog., Golden, CO 80401 USA; [2]The Timken Company, 1835 Dueber Ave. SW, PO Box 6932, Canton, OH 44706-0932 USA; [3]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185-1411 USA

Monte Carlo simulators like the classic Q-state Potts are now used routinely to consider grain coarsening, but coupling these simulations to an evolving elastic stress state has only received passing attention. Such a coupling is established in the present work. Bond stiffnesses in a harmonic lattice model are expressed in terms of the elastic stiffness elements of a tetragonal elastic solid with general tilt orientation. The harmonic model is then used to calculate the mechanical driving force associated with a change in orientation of one lattice site. This additional free energy is included in a Q-state Potts model. The resulting Hybrid Monte Carlo (HMC)model was used to investigate the development of grain texture in response to mechanical loading. The variation in grain orientation reduces with an applied uniaxial tension, and a relationship was established between appliedtraction and the rate at which this variation reduces.

9:30 AM
Grain Size Distribution in 3D Ideal Grain Growth: *Won Tae Kim*[1]; Seong Gyoon Kim[2]; [1]Cheongju University, Applied Sci. Div., 36 Naedok Dong, Cheongju 360-764 Korea; [2]Kunsan National University, Dept. of Matls. Sci. & Engrg., 68, Miryong Dong, Kunsan 573-701 Korea

Details on grain size distribution during the ideal 3D grain growth have been studied by using a phase field modeling. We developed a phase field model of grain growth, allowing unlimited number of grains with greatly improved computational efficiency. For accuracy test of the model showed that von Neumann's law on ideal 2D grain growth was completely satisfied irrespective of number of edges, indicating the model operates in a correct way. Then 3D computations on grain growth with several different initial grain size distributions have been performed on a 420x420x420 grid system. Irrespective of the initial grain size distribution, all computational results converges to a universal size distribution, which is exactly same as the prediction of grain growth model proposed by Hillert about 40 years ago. This study is the first numerical confirmation of Hillert's grain growth model.

9:50 AM
Effect of Magnetic Field on Fe-Si Phase Diagram and Grain Growth Behavior in Fe-1wt%Si: *Michael C. Gao*[1]; Tricia A. Bennett[2]; David E. Laughlin[2]; Anthony D. Rollett[2]; [1]Northeastern University, Dept. of Physics, Boston, MA 02115 USA; [2]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

In parallel to the study of grain growth behavior of Fe-1wt%Si alloy under high applied magnetic fields, the effect of the magnetic field on the Fe-Si phase diagram in the Fe-rich corner is investigated. The magnetic Gibbs free energy of fcc austenite, bcc ferrite and ordered bcc phases are calculated using Weiss molecular field theory. The focus of the investigation is on the influence of magnetization and thermodynamics on grain boundary behavior as well as estimation of driving forces between different orientations.

10:10 AM Break

10:45 AM Invited
Energetics of Ge/Si(100) Island Formation: Role of Strain-Dependent Surface Energies: Oleg Shklyaev[2]; Matthew J. Beck[1]; *Mark D. Asta*[1]; Michael J. Miksis[2]; Peter W. Voorhees[1]; [1]Northwestern Uni-

versity, Dept. of Matls. Sci. & Engrg., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [2]Northwestern University, Dept. of Engrg. Sci. & Applied Math., 2145 Sheridan Rd., Evanston, IL 60208 USA

The energetics underlying formation of nanometer-scale, pyramid-shaped Ge islands in vapor-phase, thin-film growth on Si(100) have been calculated using a computational approach combining first-principles results for strain-dependent surface and interface energies with an analytical continuum analysis of the epitaxial elastic strain energy. The calculations point to pronounced effects associated with the strain dependence of surface energies. Island stability is found to be governed in large part by the reduction in the wetting-layer excess energy arising from the compressive strain in the vicinity of the coherent islands. This strain leads to a substantial reduction in Ge(100) surface energy. The origin of the pronounced strain dependence of the surface energy will be discussed in terms of the nature of the rebonded surface reconstructions.

11:15 AM Invited

Atomistic Simulations of Reactive Wetting and Brazing: *J. J. Hoyt*[1]; E. B. Webb[1]; G. S. Grest[1]; D. R. Heine[1]; [1]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185 USA

Molecular dynamics (MD) simulations utilizing interatomic potentials of the embedded atom type have been used to study reactive wetting in the eutectic Ag-Cu system. For pure liquid Ag on Cu substrates, the radius of the drop, R(t), is found to spread with time according to a power law of the form R(t)~t^1/2. This dissolution controlled regime is compared with the case of Ag-Cu binary droplets on Cu where a lower power law exponent emerges at late times. The slower kinetics suggests a possible crossover to a diffusion controlled reactive wetting mechanism. In addition, brazing has been studied for the case of liquid Cu infiltrating a small channel of solid Ni base metal. The rate of infiltration, the development of the equilibrium contact angle and the dissolution of Ni are monitored as a function of time. For both reactive wetting and brazing the dissolution rate of the solid plays a key role in the kinetics. A model for the dissolution rate is developed based on previous models for the crystallization of pure metals as a function of undercooling.

11:45 AM

Continuum Simulation of Faceted Thin Film Growth: *Danxu Du*[1]; David J. Srolovitz[1]; Michael E. Coltrin[2]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., D214 E-Quad, Olden St., Princeton, NJ 08540 USA; [2]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185 USA

Many semiconductor, ionic and oxide thin films grow in a facetted manner. In some cases, the facetted nature of the growth can be used to manipulate defect structures and morphology. We describe a level set formalism for modeling faceted growth based upon knowledge of the growth rate as a function of surface orientation. Simple rules emerge for the growth of both convex and concave shapes. Interestingly, for convex growth, the morphology is dominated by the fastest growing facets, while in concave growth the slower facets are more important. While corners/edges are sharp in convex growth, rounded corners/edges are more common in concave growth. We apply the new level set method for facetted film growth to the case of Epitaxial-Lateral-Overgrowth (ELO) of GaN.

Converter and Fire Refining Practices: Process Improvements and Anode Casting

Sponsored by: Extraction & Processing Division, EPD-Pyrometallurgy Committee

Program Organizer: Alistair G. Ross, INCO, Ltd., Canadian Smelting & Copper Business, Copper Cliff, P0M 1N0 ON Canada

Tuesday AM Room: 2016
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Rob L. Stephens, Teck Cominco Metals Ltd, Lead Products, Trail, BC V1R 4L8 Canada; Robert Matusewicz, Ausmelt Limited, Dandenong, Victoria 3175 Australia

8:30 AM

Anode Casting Operation, Process Improvements and Anode Quality: *Victor Raul Alarcon*[1]; [1]Southern Peru Copper Corporation, Ilo Refinery, Caminos del Inca 171, Pampa Caliche Km.9, Surco-Lima, Ilo-Moquegua Peru

The Ilo Refinery was commissioned in 1975, with a cathode production capacity of 150,000 tpy. In 1994, Southern Perú Copper Corporation (SPCC) purchased the Refinery from Minero Perú SA. Subsequently, in November 1999, Grupo Mexico acquired control of Southern Perú. The anode production facility is located at the Ilo Refinery. A number of effective operating and quality control practices have been developed that have improved anode quality, as well as increasing anode production capacity to 350,000 tpy. These, and other optimization actions have allowed for a current density increase from 213 A/m2 to 275 A/m2, while maintaining current efficiency at 98% and sustaining the high quality of the cathode. The paper will outline the scope of operational practices in the Anode Plant and provide some discussion on the Ilo Smelter modernization project, and the resulting impact on the existing anode plant.

9:00 AM

The Application of 'Six Sigma' Methodologies to Improve the Quality of Converter Slag at Atlantic Copper: Jesús Contreras[1]; *Rafael Fernández-Gil*[1]; *Miguel Palacios*[1]; [1]Atlantic Copper, S.A., Smelter Ops., Av Francisco Montenegro, s/n., 21001 Huelva Espana

Within its program for continuous improvement and cultural change, Atlantic Copper has installed the Six Sigma methodology as a basic tool for the improvement of management techniques in all its production centres. Following an implementation period, the methodology is widely used for analysis and improvement, not only in the fields of production and maintenance, but also in other areas of the business such as supplies or energy management. The present paper describes, phase-by-phase, the project that was developed to improve the quality of converter slag through the application of Six Sigma techniques, from the initial set up to the final results obtained.

9:30 AM

New Casting Moulds for Anode Copper: *Torben Edens*[1]; *Dirk Hannemann*[1]; [1]Norddeutsche Affinerie, Copper Production & Recycling, Hoverstrasse 50, Hamburg D-20539 Germany

Norddeutsche Affinerie's (NA's) primary smelter continually increased production in the past decade. The smelter's concentrate capacity was expanded from 500,000 in 1993 to 1,000,000 t/a dry concentrate in 2000 and the goal of the present upgrade is 1,150,000 t/a. These goals were reached without erecting new equipment in the anode area, but instead by refitting the existing plant. The life-span extension of the casting moulds was absolutely necessary to increase the casting performance from 260,000 to 450,000 t anodes per year on a single casting wheel. Different mould manufacturing processes and alloys were tested in order to find the optimal mould. In the past the moulds were cast directly, now the anode mould is milled into NA copper cakes. Accompanying measures taken were the shortening of maintenance and repair times as well as improvements in the refining furnace equipment.

10:00 AM Break

10:15 AM

Hoboken Converter Performance Improvements at the Phelps Dodge Miami Smelter: *Ovidiu Pasca*[1]; Vlad Ushakov[1]; Eugene Welker[1]; [1]Phelps Dodge, Miami Smelter, PO Box 4444, Claypool, AZ 85532 USA

The Phelps Dodge Miami Smelter utilizes four Hoboken converters to process molten matte from the Isasmelt vessel. This paper presents past and present improvements in operation, maintenance and process optimization, based on data collection, which has led to increased converter campaign life and copper throughput.

10:45 AM

Productivity Increase in a Peirce-Smith Converter Using the 'COP KIN' and 'OPC' System: *Thomas Prietl*[1]; Andreas Filzwieser[2]; Stefan Wallner[3]; [1]RHI Non-Ferrous Metals Engineering GmbH and Christian Doppler Laboratory for Sekundary Metallurgy of the Non-Ferrous Metals, Non-Ferrous, Magnesitstrasse 2/RHI, Franz Josef Strasse 15/CD-Lab., Leoben 8700 Austria; [3]RHI Non-Ferrous Metals Engineering GmbH, Non-Ferrous, Wienerbergstrasse 11, Vienna 1100 Austria

The use of gas stirring systems through the bottom of a furnace in the copper industry is common for anode and holding furnaces. The first implementation of a gas stirring COP KIN® system in a Peirce-Smith converter was in Sweden at the New Boliden smelter in Rönnskar. A decrease in process time and a decrease of the oxygen content in the blister copper were observed. To determine the effects of the gas stirring system and the process endpoint, an optical production control 'Semtech OPC system' was used. The light emission of the converter flame as an optical process parameter provides qualitative on-line process information. This information is also used for endpoint determination of the slag making process, on-line control of iron content in white metal, quality control of slag etc. The results, ben-

efits and risks of using the COP KIN® and OPC system for a Peirce-Smith converter are reported.

11:15 AM
Pyrometallurgical Refining of Copper in an Anode Furnace: H. Antrekowitsch[1]; Chr. Wenzl[1]; *I. Filzwieser*[2]; D. Offenthaler[2]; [1]University of Leoben, Christian-Doppler Lab. for Secondary Metall. of Non-Ferrous Metals, Franz-Josef-Strasse 15, A-8700 Leoben Austria; [2]University of Leoben, Dept. of Nonferrous Metall., Franz-Josef-Strasse 15, A-8700 Leoben Austria

The decreasing quality of the input materials in copper recycling leads to a higher content of impurities in the anode copper. An improvement of the pyrometallurgical refining process is therefore necessary to produce high quality anodes for the copper refining electrolysis. The behaviour of the most important accompanying elements (e.g. nickel, tin, lead, zinc etc.) at different reaction conditions has to be investigated in order to improve the metal/slag reactions as well as the vaporisation by selective oxidation in the anode furnace. Therefore the thermodynamic conditions like reaction order and the activity coefficients at the copper refining process have to be known for the optimisation of this technique. Additionally the interaction between different elements has been investigated as a function of the temperature, the content of the elements and the slag composition especially for nickel. These investigations were done at the Christian Doppler Laboratory for Secondary Metallurgy of Nonferrous Metals.

Extractive Metallurgy: Recycling and Waste Minimization

Sponsored by: Extraction & Processing Division, EPD-Aqueous Processing Committee, EPD-Pyrometallurgy Committee, EPD-Waste Treatment & Minimization Committee
Program Organizers: Thomas P. Battle, DuPont Titanium Technologies, Wilmington, DE 19880-0352 USA; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA; Courtney A. Young, Montana Tech of the University of Montana, Metallurgical Engineering, Butte, MT 59701 USA

Tuesday AM Room: 2018
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Junji Shibata, Kansai University, Dept. of Cheml. Engrg., Osaka 564-8680 Japan; Boyd R. Davis, Kingston Process Metallurgy Inc., Kingston, Ontario K7P 1S6 Canada

8:30 AM
Mercury Recovery in Autoclave Effluents from Refractory Ore Treatment: *Rainer Bauder*[1]; Steven F. McGrath[1]; Katherine Milidakis[1]; [1]MR3 Systems, Inc., 435 Brannan St., Ste. 200, San Francisco, CA 94107 USA

Refractory Gold deposits are often associated with mercury as contamination. When those ore types undergo oxidation in an autoclave system, a significant mercury level appears in the wash waters and effluents. The paper describes a hydrometallurgical approach to remove mercury directly in the autoclave effluent. Because of the typical very strongly acidic conditions caused by the production of sulfuric acid during the oxidation of the sulfides, a silica based chelating absorbent is utilized to extract the mercury efficiently, providing a way to remove it effectively for the further treatment necessary. This novel approach allows selective isolation of mercury in the high metal environment, and therefore addresses the environmental issues associated with it.

8:55 AM
Metallic Secondary Raw Materials Recycling Strategy in Serbia: *Sasha D. Djokic*[1]; [1]MAG, Zeleni bulevar bb, BOR 19210 Serbia

In developed countries, the difference between demanded and produced quantities of non-ferrous and rare metals are compensated by secondary raw materials recycling and reprocessing of by-products from both ferrous and non-ferrous extractive metallurgy. Utilization of non-ferrous and rare metals from the metallic secondary raw materials is very significant, both for recovery of the metal value and contribution to the environment protection. Similar to all non-developed countries, the extractive metallurgy in Serbia was based on available natural resources and not so strict environment protection regulations, which was either insufficient or completely absent. Today, Serbia is at the beginning of development of efficient and economical technical and technological solutions in order to "ecologize" technogenic systems by waste materials, secondary raw materials and degraded soils regeneration (by recycling and revitalization). Several metallurgical technogenic systems in Serbia, together with secondary raw materials classification according to their potentiality for metal extraction by using the existing technologies, determination of their structure, composition, and expected economical and ecological effects of recycling, are discussed in this paper.

9:20 AM
Valorization of Solid Wastes as Sorbents for Heavy Metals: Luciano R.G. Santos[1]; Rafael Falco Rodrigues[1]; *Versiane Albis Leao*[1]; Eucler B. Paniago[2]; Vagner L. Botaro[2]; [1]Universidade Federal de Ouro Preto, Metallurgl. Matl. Eng., Praça Tiradentes, 20, Centro, Ouro Preto, MG 35400-000 Brazil; [2]Universidade Federal de Ouro Preto, Dept. of Chmst., Inst. de Ciências Exatas e Biológicas, Campus Morro do Cruzeiro, s.n., Ouro Preto, MG 35400-000 Brazil

The majority of the industrial activities produce some kind of solid wastes. In this work two industrial residues (spent zeolite and wood sawdust) were chemically modified to increase their heavy metals sorption capacities. Manilkara longifolia (a Brazilian plant known as parajú) sawdust was first treated with NaOH and modified with different citric acid concentrations (0.6-1.2mol/L) at 120°C, for 90 min. Infrared spectroscopy confirmed the presence of carboxylate groups in the wood structure as result of their mixing. Similarly, spent zeolites were also submitted to a chemical treatment (NaOH) to remove carbon deposited during oil cracking and to recover its sorption capacity. Batch experiments carried out at ambient temperature showed that both materials present sorption capacity for copper and cadmium comparable to weak acid ion exchange resins. A maximum of 78mg-Cd/g and 59.7mg-Cu/g as well as 29.9mg-Cd/g and 14.6mg-Cu/g were observed for the sawdust and the zeolite, respectively. The results were modeled according to the Freundlich and Langmuir equilibrium isotherms and compared with results of other studies.

9:45 AM
Aqueous Oxidative Precipitation of Manganese by SO2/O2: *Vincent Menard*[1]; George P. Demopoulos[1]; [1]McGill University, Mining, Metals & Matls. Dept., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

In many hydrometallurgical processes, manganese is an impurity that needs to be removed from solution prior to metal recovery. The simplest method of removal is precipitation as hydroxide, i.e. Mn(OH)2, by pH adjustment. However, this method is not selective as most of the other elements present in the solution precipitate as well. An alternative method of manganese removal is by oxidative precipitation. This method consists in oxidizing Mn2+ to Mn3+/Mn4+ which precipitate readily as hydrous manganese oxide compounds. The purpose of the present work is to remove selectively manganese from a zinc-rich solution using a gas mixture of SO2/O2 as oxidizing agent. Several semi-batch experiments were performed where the effects of redox potential, SO2/O2 ratio, mixing intensity, etc. were investigated. These tests were followed by setting-up and running a multi-reactor continuous circuit employing pH/ORP control and product recycling in an effort to improve purity and solid-liquid separation of the precipitates.

10:10 AM Break

10:25 AM
Aqueous Processing of Lithium Cobalt Ion Battery Scrap for Cobalt Recovery: *Raj P. Singh*[1]; Thomas A. Wolfe[1]; [1]Chemicals & Powders R&D, Precision Matl. & Components Div., Osram Sylvania, Towanda, PA 18810 USA

Cobalt is a strategic metal, which is used in many diverse industrial and military applications such as super alloys, magnets, hard metals (carbide), catalysts, colors, sulfate, batteries, tires and paint driers. Cobalt is mainly produced from byproducts of other more abundant metals. For example, more than one-half of the world's supply of cobalt is produced from byproduct of copper mining and refining in African countries Zaire and Zambia. Cobalt production in most other countries is carried out from byproducts of nickel mining and / or refining. The United States is the world's largest consumer of cobalt, and is 85% dependent on imports for its supply of primary cobalt. About 15% of U.S. cobalt consumption comes from recycled scrap. Since their commercialization in 1991, lithium-cobalt batteries have been widely used in electronic applications. These batteries have certain advantages over nickel-cadmium and nickel-metal hydride batteries and due to this reason, in near future; their market share is expected to grow large. Current research on these batteries is concerned on the development of large sized batteries for their use in electric vehicles. This is a fast developing area and will result into appreciable growth of such batteries. The production and use of large sized lithium-cobalt batteries for electric vehicles applications would generate large amount

of primary and secondary scrap, which is expected to be a potential feed source for cobalt. In this paper we will present characterization of lithium cobalt oxide battery scrape from industrial sources and aqueous processing of these scraps for cobalt via a cobalt hexammine chloride process. Chemistry, efficiency and accountability of various steps of the method for cobalt processing will be discussed.

10:50 AM
The Behavior of Heavy Metal, Alkali and Chloride Derived from Waste Sludge on the Cement Industrial Process: *Kwang-Suk You*[1]; Ji-Whan Ahn[1]; [1]Korea Institute of Geoscience & Mineral Resources, Taejeon 305-350 Korea

The purpose of this study is to use the waste sludges generated at Pohang Iron & Steel Co. Ltd.(POSCO) by the raw materials of ordinary portland cement(OPC). To evaluate the possibility of its application, in this study, it was investigated that the behavior of heavy metal, alkali and chloride generated from waste sludge of steel making industry when it is used as raw mixture for cement. The waste sludge was mainly composed of CaO, SiO2, Al2O3, and Fe2O3, similar as the main component of OPC. It was reported that alkali and chloride occur various troubles in the Kiln, and heavy metals affected the properties of cement clinkers. This study discussed the hazardous effects by them and the characteristic of cement manufactured from waste by POSCO sludge. From the result of study, it was concluded that POSCO sludge can be used as the source materials of iron and calcium oxide.

11:15 AM
Tin and Zinc Recovery and Pellet Preparation from Iron Ores Containing Tin and Zinc: *Yuanbo Zhang*[1]; Yufeng Guo[1]; Guanghui Li[1]; Yongbin Yang[1]; Zhucheng Huang[1]; Xiaohui Fan[1]; Tao Jiang[1]; [1]Central South University, Sch. of Resources Procg. & Bioengrg., Changsha, Hunan 410083 China

The complex iron ores containing tin, zinc are typical refractory ores. Great reserves of the ores are found in China and have not been utilized efficiently. A new process of tin and zinc recovery and pellet preparation from iron ores containing tin and zinc by controlled roasting is developed in this investigation. The results of compression strength of product pellets of 2573 N/P, the volatilization of tin and zinc of 91.49% and 82.22% respectively, are achieved for a concentrate containing 0.39% tin and 0.28% zinc. The compression strength and chemical compositions of the product pellets meet the requirement of blast furnaces. Volatilization mechanism of tin and zinc are investigated. Thermodynamic study shows that the reduction balance curves of SnO2 and SnO are proximate under the condition of the existence of carbon, and the standard free enthalpy of FeO and SnO2 is also approachable. Therefore, it is difficult to realize the effective volatilization of SnO by selective reduction. But SnO2 and SnO will form SnCl4 or SnCl2 and can be easily volatilized from the ores if they are transferred into their chlorides.

Friction Stir Welding and Processing III: Friction Stir Processing

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Lienert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Tuesday AM Room: Nob Hill C/D
February 15, 2005 Location: San Francisco Marriott

Session Chair: Murray W. Mahoney, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA

8:30 AM Keynote
Friction Stir Processing for Microstructure Modification of NiAl Bronze: *Terry R. McNelley*[1]; Keiichiro Oh-ishi[1]; Alex P. Zhilyaev[1]; Robert A. Williams[1]; [1]Naval Postgraduate School, Mechl. & Astronautical Engrg., 700 Dyer Rd., Monterey, CA 93943-5146 USA

Friction stir processing (FSP) has been applied to as-cast NiAl bronze materials, which are widely used for marine components, in order to selectively convert the microstructures in the near-surface layers from a cast to a wrought condition. The physical metallurgy of NiAl bronze is complex and interpretation of the effects of FSP on the homogenization and refinement of microstructure has required detailed analysis by optical and electron microscopy methods. Peak temperatures in the stir zone due to thermomechanical cycle of FSP have been estimated from microstructures and compared to model predictions. Isothermal hot rolling and hot compression testing has been employed to confirm microstructure-based estimates of stir-zone peak temperatures. The variation of mechanical properties was assessed by use of miniature tensile samples and correlated with microstructure for samples from stir zones as well as hot rolled material. Extension of these results to multi-pass processing will be presented.

9:00 AM
Friction Stir Processing of Ferrous Alloys for Increased Wear Resistance: *Uma Ramasubramanian*[1]; Glenn J. Grant[2]; Glen A. Stone[1]; William J. Arbegast[1]; [1]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD USA; [2]Pacific Northwest National Laboratories, Matls. Procg. & Performance, 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

The Advanced Materials Processing Center (AMP) at the South Dakota School of Mines and Technology in conjunction with the Pacific Northwest National Laboratories is developing methods to stir micron size TiB2 particles into the surface of gray cast iron. Surface enrichment with ceramic particles may results in improved brake materials for heavy-duty brake systems. The objective is less high temperature fading and increase in wear resistance. The present work investigates the distribution of particles as a function of the pin tool design, processing parameters, powder size, slit geometry and position.

9:20 AM
Incorporating Titanium Powder to Create In-Situ Composites on the Surface of Cast Iron via Friction Stir Reaction Processing (FSRP): *Raja L. Veluchamy*[1]; Glenn J. Grant[2]; Glen A. Stone[1]; William J. Arbegast[1]; [1]South Dakota School of Mines and Technology, AMP, 501 E. St. Joesph St., Rapid City, SD 57701-3995 USA; [2]Pacific Northwest National Laboratories, Matls. Procg. & Performance, 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

The Advanced Materials Processing Center (AMP) at the South Dakota School of Mines and Technology in conjunction with the Pacific Northwest National Laboratories is exploring in-situ chemical reactions during FSP. The titanium/graphite system is thermodynamically favorable so as to produce TiC during FSP. It is expected that excess carbon (graphite) contained within gray cast iron and Ti powders stirred into the material by FSP, will form hard, wear resistant, TiC compounds dispersed within matrix. This research is designed to test the feasibility of initiating and controlling this chemical reaction during FSP. The end point goal of the program is to increase the friction on the surface of cast iron brake disks and to improve the wear characteristic of brake rotors, drums and pads. Brake cast iron typically has type A graphite, with a pearlitic matrix and low ferrite and carbide content.

9:40 AM
Friction Stir Technology for Superplastic Forming: Sky5083 Alloy: *Alok Vats*[1]; Stanley M. Howard[1]; William J. Arbegast[2]; Darrell R. Herling[3]; Glenn J. Grant[3]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [3]Pacific Northwest National Laboratory, Matls. Sci. Div., K2-03, 902 Battelle Blvd., Richland, WA 99352 USA

The scientific understanding of superplastic forming (SPF) has evolved immensely over the last decade driven primarily by the need for economic competitiveness compared to metal stamping methods. Much research has focused on aluminum's thermomechanical processing schedules to produce microstructures that exhibit superplastic behavior. It is agreed that for aluminum-magnesium alloys an equiax fine-grain microstructure is the most desirable. Typical methods of processing aluminum sheet for SPF is by a series of hot and extensive cold rolling schedules. Although a robust method sheet materials, it requires either starting with large billet cross sections or ending up with a limited gage thickness to achieve the required strain energy. Friction Stir Processing (FSP) is an emerging processing method that has been proved can produce a fine-grain structure in most aluminum alloys. The objective was to develop FSP parameters for both linear and spot welding to produce optimum microstructures and behavior.

10:00 AM
High Strain Rate Superplastic Properties of 7075 Aluminum via Multiple Pass Friction Stir Processing: *L. B. Johannes*[1]; I. Charit[1]; R. S. Mishra[1]; [1]University of Missouri, Dept. of Matls. Sci. & Engrg., Ctr. for Friction Stir Procg., Rolla, MO 65409 USA

Friction stir processing (FSP) leads to fine grained microstructure in commercial 7075 aluminum plates. In this study, the effects of performing multiple, overlapping passes of FSP on 7075 Al were examined. Samples ranging from one to four passes and a nine pass sample were used with a 6.4 mm separation between passes (~50% overlap). In the temperature range of 400°C to 490°C, it was shown that the area of overlapping passes exhibits elongations >200% at the commercially important strain rate of $1x10^{-2}$ s^{-1}. In the nine pass sample, elongations averaging greater than 550% were found at 490°C at this strain rate. The unprocessed 7075 exhibited elongations <200% over the same range of temperatures and hence, no superplastic properties. Strain rate sensitivities of regions in the various passes were also found to be around 0.5, indicating grain boundary sliding dominant mechanism. These results show the potential for using FSP to obtain superplastic properties in commercial aluminum alloys.

10:20 AM Break

10:40 AM Invited
Thick Plate Bending of Friction Stir Processed Aluminum Alloys: *Murray W. Mahoney*[1]; Christian Fuller[1]; Mike Miles[2]; William Bingel[1]; [1]Rockwell Scientific, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA; [2]Brigham Young University; Coll. of Engrg. & Tech. 265 CTB, Provo, UT 84602 USA

Friction stir processing (FSP) was used to modify the surface microstructure in thick plate 7050-T7451 aluminum to enhance room temperature bending. Plate, 254 x 216 x 50mm thick, was friction stir processed on the pre-tensile surface to shallow and different depths (3.1 and 6.3 mm) using different approaches, i.e., the FSP raster included both linear and spiral patterns. Following FSP, plates were bent at room temperature to different degrees including a compound curvature. Results illustrate the ability to significantly enhance room temperature formability via FSP. To illustrate the extreme capability of this technique, similar processing was applied to15cm thick 6061 aluminum. In this case, FSP was applied to a depth of 6mm and this very thick plate bent to 30º at room temperature without failure. Thick plate bending can be used to substitute for welding of corner structures or to shape plate for machining of a monolithic structure where a plate thickness is not attainable.

11:00 AM
Friction Surface Reaction Processing on Aluminum Substrates: Stanley M. Howard[1]; *Bharat K. Jasthi*[1]; William J. Arbegast[2]; Glenn J. Grant[3]; Darrell R. Herling[3]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., Rapid City, SD 57701 USA; [3]Pacific Northwest National Laboratory, Matls. Sci. Div., K2-03, 902 Battelle Blvd., Richland, WA 99352 USA

Friction Stir Processing (FSP) is a variation of Friction Stir Welding that uses a spinning tool to modify the material through severe plastic deformation. Recent work has shown that surface modified regions in some aluminum materials can contain increased quantities of fine-grained silicide phases stabilized during nugget formation. Plastic work energy combined with the "new" surface created during shearing may promote some solid-state reactions. The work outlines results of several metallurgical systems to determine if energetic solid-state reactions during FSP can be sustained to produce novel surface materials and microstructures. The potential exists to form regions enriched in nitrides, borides or carbides in-situ by stirring elemental or oxide powders into the surface of monolithic materials. A goal of this work was to determine: 1) the efficacy of FSP mechanical energy in initiating and maintaining these energetic reactions, 2) product distribution and morphology, and 3) reaction control.

11:20 AM
The Effect of Friction Stir Processing and Subsequent Rolling on the Superplastic Behaviour of Aluminium Alloys: *Stavros Katsas*[1]; Graham Todd[1]; Martin Jackson[1]; Roger Grimes[1]; Richard J. Dashwood[1]; [1]Imperial College London, Dept. of Matls., S. Kensington Campus, London SW7 2AZ UK

Friction stir processing (FSP) can dramatically reduce grain size conferring excellent superplastic behaviour in certain aluminium alloys. FSP of thick plate followed by rolling to sheet could potentially be used as a method to improve performance of established superplastic alloys or to induce superplastic behaviour in alloys not normally associated with this phenomenon. An extruded Al-Mg-Zr alloy was FSP?d prior to rolling to sheet. The development of microstructure and superplastic behaviour was characterised using a combination of light, scanning, orientation imaging, and transmission microscopy, coupled with hot uniaxial tensile testing. FSP transformed the coarse, highly textured extruded, structure into a very fine (approximately 600nm) randomly orientated, equiaxed material. The structure of the material after rolling and thermal treatment was complex and in certain cases significant grain coarsening resulted. A detailed investigation of factors responsible for this will be described.

11:40 AM
Influence of Prior Microstructure on Friction Stir Processing of Cast A356 Plates: *S. R. Sharma*[1]; R. S. Mishra[1]; [1]University of Missouri, Dept. of Matls. Sci. & Engrg., Ctr. for Friction Stir Procg., Rolla, MO 65409 USA

Friction stir processing (FSP) leads to microstructural refinement and, in case of a cast microstructure, leads to significant improvement in static and dynamic mechanical properties. The present work investigates the influence of prior heat treatment on the microstructure and mechanical properties of cast A356 after FSP. Cast plates were friction stir processed in as cast, solution treated and T6 conditions. Influence of prior heat treatment on forces during FSP and mechanical properties after FSP was evaluated.

12:00 PM
Superplastic Forming of Friction Sir Welded Ti-6-4 Sheet: *Glenn J. Grant*[1]; D. Sanders[2]; A. P. Reynolds[3]; Wei Tang[3]; [1]Pacific Northwest National Laboratory, Richland, WA 99352 USA; [2]Boeing, Seattle, WA 98124 USA; [3]University of South Carolina, Columbia, SC 29208 USA

Titanium 6-4 alloy sheets, 2 mm thick, were friction stir welded in a square butt configuration. The friction stir welded sheets were subsequently characterized via optical and scanning electron microscopy. Elevated temperature, transverse (loading perpendicular to the welding direction), tensile tests were performed to assess the superplastic performance of the sheet and the weld regions. After establishing parameters for superplastic forming of the welds, pans were formed from the welded sheets. Superplastic behavior of the sheet was retained or enhanced in the welds. In this paper, weld and base metal microstructure and superplastic performance of the welded structure will be presented.

Frontiers in Solidification Science: Nucleation

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Solidification Committee
Program Organizers: Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; James R. Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Tuesday AM Room: 2020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: John Perepezko, University of Wisconsin, WI USA; Mark D. Asta, Northwestern University, Evanston, IL 60208 USA

8:30 AM Invited
Coupled Processes in Nucleation: *Kenneth F. Kelton*[1]; [1]Washington University, Dept. of Physics, Campus Box 1105, One Brookings Dr., St. Louis, MO 63130 USA

It is becoming increasingly clear that coupling plays an important role in many nucleation processes. Generally, this cannot be explained quantitatively within the commonly used classical theory of nucleation. For example, often the initial and final phases have different chemical compositions, requiring a new model that couples the fluxes of long-range diffusion and interfacial attachment. Recently, we have identified a case of order parameter coupling for nucleation from an undercooled metallic liquid. From high-energy x-ray and undercooling studies of an electrostatically-levitated Ti-Zr-Ni liquid, we demonstrated that fluctuations in the short-range order of the liquid determined the primary crystallizing phase, linking the local structural order parameter of the initial phase with the nucleation barrier. These and other examples of coupled processes in nucleation are discussed. The implications of coupling on nucleation theory are discussed. Supported by NASA under contract NAG 8-1682, and by the NSF under grant DMR 03-07410.

9:05 AM Invited
Short-Range Order in Undercooled Metallic Melts and its Influence on Solidification: *Dirk Holland-Moritz*[1]; [1]DLR, Inst. fuer Raumsimulation, Linder Hoehe, Koeln D-51147 Germany

This work presents investigations on the short-range order of deeply undercooled liquids of the pure elements (Ni, Co, Zr, Ti and Fe) and of

alloys forming quasicrystalline or polytetrahedral phases ($Al_{60}Cu_{34}Fe_6$, $Al_{65}Cu_{25}Co_{10}$ and $Al_{13}(Fe,Co)_4$). The liquids were containerlessly processed and undercooled by application of the electromagnetic levitation technique, which was combined with elastic neutron scattering at the Institute-Laue-Langevin (ILL) in Grenoble and with energy dispersive diffraction of synchrotron radiation at the European Synchrotron Radiation Facility (ESRF) in Grenoble in order to determine the structure factors of the liquids as a function of the temperature. For all of the investigated metallic liquids the experimental data are well described if an icosahedral short-range order is assumed to prevail in the melt. This icosahedral short-range order is observed already at temperatures above the melting temperature and becomes more pronounced if the melt is undercooled. The impact of the icosahedral short-range order in the undercooled melt on the energy of the interface between the melt and nuclei of solid phases of different structures is discussed. This structure-dependence of the solid-liquid interfacial energy decisively influences the solidification behavior of the liquids.

9:40 AM
Do Icosahedral Phases Melt Into Icosahedral Liquids? A Combined Experimental and Theoretical Study of Liquid CdYb: *M. J. Kramer*[1]; D. J. Sordelet[1]; Y. Ye[1]; T. A. Lograsso[1]; Ulf Dahlborg[2]; J. R. Morris[3]; [1]Iowa State University, Ames Lab., 37 Wilhelm, Ames, IA 50011 USA; [2]Ecole des Mines, LSG2M, UMR 7584, Parc de Saurupt, Nancy 54042 France; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA

There has been a significant amount of recent research arguing that some simple metals, i.e., Ni, show icosahedral structure in the undercooled liquid. Other indirect evidence for icosahedral order in liquids is based on fast nucleation rates of certain icosahedral structures and their approximants. Local icosahedral order is also indicated by the formation of metastable quasicrystalline phases in some devitrified metallic glasses. We present results from diffraction experiments and ab initio calculations, that demonstrate that the converse does not always hold true: Cd5.7Yb forms a binary, congruently melting quasicrystal which melts into a non-icosahedral liquid. This provides an explanation for the observed slow nucleation rate of this stable quasicrystal. These results are still in agreement with Frank's hypothesis; the difference in structure from the liquid and solid phases indicates a large crystal-melt interfacial free energy, resulting in a large nucleation barrier. This research has been sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract W-7405-ENG-82 with Iowa State University of Science and Technology and contract DE-AC05-00OR-22725 with UT-Battelle, LLC.

10:00 AM Invited
Heterogeneous Grain Initiation in Solidification: *A. L. Greer*[1]; T. E. Quested[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

Grain refinement in casting of aluminum alloys has recently been analyzed by assuming that the barrier for grain initiation on an inoculant particle is that for free growth from the particle rather than for nucleation of the solid. Adsorption effects can stabilize a thin layer of solid on the particle even above the liquidus temperature. In modeling so far undertaken, the approximation has been made that grain initiation depends only on undercooling, with no stochastic behavior. In this paper the nature of the barrier for grain initiation is examined quantitatively. The relative importance of deterministic and stochastic elements of grain initiation is explored as a function of inoculant particle size and melt undercooling. The effect of dormant solid on the particles is taken into account. The nature of the adsorption and templating on the inoculant surface are also considered.

10:35 AM Break

10:45 AM Invited
Atomistic Simulations of the Nucleation of Nickel: *Michael I. Baskes*[1]; Frank J. Cherne[1]; Ricardo B. Schwarz[1]; S. G. Srinivasan[1]; William Klein[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Boston University, Dept. of Physics, Boston, MA 02215 USA

The dynamics of homogeneous nucleation and growth of crystalline nickel from its super-cooled melt is examined during rapid quenching using molecular dynamics and a modified embedded atom potential. The character of the critical nuclei of the crystallization transition is examined using common neighbor analysis and visualization techniques. We find that the critical nucleus consists of a small number of atoms arranged in a stacking of planar structures. There is two-dimensional triangular order within each plane but the planes are randomly stacked. Because the stacking is not fcc (nor hcp), our results do not support classical nucleation models. They agree, however, with recent non-classical nucleation models (Shen and Oxtoby (1996); Klein and Leyvraz(1986)). Three-dimensional order develops after the nucleus grows and becomes supercritical. W. Klein and F. Leyvraz, Phys. Rev. Lett. 57, 2845 (1986). Y. C. Shen and D. W. Oxtoby, Phys. Rev. Lett. 77, 3585 (1996). This work was supported at Los Alamos National Laboratory by the U. S. DOE under contract W-7405-ENG-36.

11:20 AM Invited
Phase Field Theory of Nucleation and Polycrystalline Solidification: *László Gránásy*[1]; Tamás Pusztai[1]; James A. Warren[2]; Jack F. Douglas[3]; [1]Research Institute for Solid State Physics and Optics, POB 49, Budapest H-1525 Hungary; [2]National Institute of Standards and Technology, Metall. Div., Gaithersburg, MD 20899 USA; [3]National Institute of Standards and Technology, Polymer Div., Gaithersburg, MD 20899 USA

A phase field theory of crystal nucleation and polycrystalline growth is presented. The model is tested for crystal nucleation in the hard sphere system. Evaluating the model parameters from molecular dynamics simulations, the phase field theory predicts the nucleation barrier accurately. The formation of spherulites is discussed in an extension of the model that incorporates branching with definite orientational mismatch induced by a metastable minimum in the orientational free energy. The mechanism of polycrystalline growth is the quenching of orientational defects (grain boundaries) into the solid due to a reduced ratio of rotational and translational diffusional coefficient expected at high undercoolings. It will be demonstrated that a broad variety of spherulitic patterns can be recovered by changing only a few model parameters (anisotropy, free energy of metastable minimum, branching angle, orientational mobility).

11:55 AM Invited
Growth Front Nucleation: A Mechanism for Polycrystalline Growth: *James A. Warren*[1]; Laszlo Granasy[2]; Tamas Pusztai[2]; Jack F. Douglas[1]; [1]National Institute of Standards and Technology, CTCMS/Metall., 100 Bureau Dr., Stop 8554, Gaithersburg, MD 20852 USA; [2]RISSPO, Budapest Hungary

The formation of a polycrystal is usually considered to occur either by the impingement of nucleating grains in a liquid (equiaxed grains), or via the nucleation of columnar grains on a surface. In this presentation we consider the formation of a polycrystal via a third mechanism: growth front nucleation. This mode of growth manifests when new orientations nucleate on the front of a growing crystal, yielding a densly branched, moprhology, where the classical effects of surface energy anisotropy are disrupted by the nucleation process. We develop a phase field model to describe the phenomenon, and demonstrate that this type of growth can be initiated by either static or dynamic heterogeneities in the solidifying system. We examine several types a growth forms, reexamine some older experiments, and examine prospects for models of this phenomenon in three dimensions.

Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Thin Films, Coatings and Nanostructures

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Orin Wayne Holland, University of North Texas, Department of Physics, Denton, TX 76203 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Stephen J. Pennycook, Oak Ridge National Laboratory, Sold State Division, Oak Ridge, TN 37831 USA; Rajiv K. Singh, University of Texas, Austin, TX 78758-4455 USA

Tuesday AM Room: 3020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Stephen J. Pennycook, Oak Ridge National Laboratory, Condensed Matter Scis. Div., Oak Ridge, TN 37831-6030 USA; Bhushan Sopori, National Renewable Energy Laboratory, Golden, CO 80401 USA

8:30 AM
Control of Bonding and Epitaxy at Metal/Sapphire Interface: Sang Ho Oh[1]; Christina Scheu[1]; Thomas Wagner[1]; *Manfred Rühle*[1]; [1]MPI für Metallforschung, Heisenbergstr. 3, Stuttgart 70569 Germany

The atomic structure and composition of the terminating plane of the sapphire (a-Al2O3) surface is one most crucial parameter control-

ling the bonding mechanism at metal/sapphire interfaces. We report here the experimental methods to control the nature of bonding at copper/sapphire interfaces by utilizing various surface cleaning processes in ultrahigh vacuum (UHV), which can tailor the termination of the sapphire (0001) surface. A clean hydroxylated surface is obtained by room temperature oxygen plasma cleaning, on which Cu atoms adhere principally by polarization and with some characteristics of ionic bonding. The surface can be sufficiently dehydroxylated by using Ar ion sputtering and annealing in UHV, resulting in an Al-rich surface, where Cu grows as a polycrystalline film and CuAl2-type intermetallic Cu-Al bonding forms at the interface. High temperature oxygen plasma treatment can sufficiently oxidize the surface Al ions in a structure similar to •-Al2O3 on which Cu2O-type ionic-covalent Cu-O bonding can be activated to form at an elevated temperature after growth.

9:00 AM Invited
Effects of Grain Size from Millimeters to Nanometers on the Flow Stress of Metals and Compounds: *Hans Conrad*[1]; Kang Jung[1]; [1]NC State University, Matls. Sci. & Engrg. Dept., Raleigh, NC 27695 USA

Data on the effect of grain size d from millimeters to nanometers on the flow stress of metals and compounds are evaluated. Three grain size regimes are identified: Regime I (d > ~1um), Regime II (d = 10nm - 1um) and Regime III (d < ~10nm). Lattice dislocations are active in Regimes I and II, but absent in Regime III. The mechanisms responsible for plastic deformation in each of the three regimes are discussed. More than one mechanism, or even a different mechanism, may become operative in the transitions between the grain size regimes.

9:30 AM Invited
Influence of Dopants on Embrittlement of Metal Grain Boundaries: *Gerd Duscher*[1]; Matthew F. Chisholm[2]; Wolfgang Windl[3]; [1]North Carolina State University, Matls. Sci. & Engrg. Dept., Raleigh, NC 27695 USA; [2]Oak Ridge National Laboratory, Condensed Matter Scis. Div., Oak Ridge, TN USA; [3]Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH USA

The combination of atomic-column resolved Z-contrast imaging, spatially resolved electron energy loss spectroscopy and ab initio density functional theory is used to study the origin of embrittlement of metal grain boundaries. The mechanical properties of these grain boundaries are dramatically influenced by dopant segregation. We will discuss three different materials (Cu, Ni3Al, and Al), in which pristine and doped grain boundaries behave differently. Our findings suggest a new concept for the determination of brittle grain boundaries.

10:00 AM Invited
Oxidation Kinetics of Boron Coating on Ferrous Alloys at High Temperature: *Roumiana S. Petrova*[1]; Naruemon Suwattananont[1]; [1]New Jersey Institute of Technology, Dept. of Physics, 161 Warren St., Univ. Hgts., Newark, NJ 07102 USA

The oxidation behavior of boron coating on steel substrate was investigated at elevated temperature. For oxidation process high temperature range 600-900°C was used. The isothermal oxidation behavior was investigated by means of TGA, Optical Microscopy, SEM, and XRD techniques. The oxidation kinetics of boron coating follows about parabolic rate law. At the oxidation temperature 600°C two fluctuations appear at approximately 12h and 18 h. At temperature 700°C only one distinct step occurs. Forming of oxides on the surface of the coated samples tends to prevent the surface from oxidation.

10:30 AM Break

10:45 AM
Photoluminescence from an Er-Doped Ge-Rich SiO2 Sputtered Films: The Influence of Sputter Gas and Annealing: *Chenglin Heng*[1]; Terje G. Finstad[1]; Anette E. Gunnæs[1]; Preben Storås[2]; *Yanjun Li*[3]; [1]University of Oslo, Dept. Physics, PO Box 1048, Blindern, Oslo N-0316 Norway; [2]SINTEF, ICT, PO Box 124, Blindern, Oslo N-0314 Norway; [3]University of Oslo, Ctr. for Matls. Sci. & Nanotech., PO Box 1126, Blindern, Oslo N-0318 Norway

We report on PL from Er-doped Ge-rich SiO2 films deposited on a Si substrate by magnetron sputtering. The sputtering ambience and annealing temperature has been varied. The structural evolution and the distribution of the elements have been studied with high resolution transmission electron microscopy in combination with analysis of emitted X-rays and energy loss spectroscopy. There is very weak or no luminescence for the as-prepared films. Large PL intensity from the film is reached after annealing at 700°C the films sputtered in Ar. These contain Ge-rich amorphous clusters. After annealing to higher temperatures the PL intensity decreases and there are Ge nanocrystals in the film. For films sputtered in an Ar+O2 gas the PL intensity increases with annealing temperature up to 1100°C while no nanocrystals form. These films are all amorphous while some segregation of Ge-rich and Si-rich oxide takes place.

11:15 AM
Effect of Processing on Microstructure and Electrical Properties of Ta Thin Films: *Vikram M. Bhosle*[1]; Ashutosh Tiwari[1]; P. Kumar[2]; R. Wu[2]; [1]North Carolina State University, MSE, 2153, Burlington, Campus Box-7916, Raleigh, NC 27695 USA; [2]H. C. Starck, Inc., 45 Industrial Place, Newton, MA 02161-1951 USA

We have fabricated thin films of tantalum with grain size ranging from nanosize to single crystal and amorphous tantalum (a-Ta) by nonequilibrium pulsed laser deposition techniques, and compared their electrical properties and diffusion characteristics with properties of Beta tantalum (â-Ta) films produced by magnetron sputtering. Single-crystal Ta films are formed by domain matching epitaxy where integral multiples of lattice planes match across the film-substrate interface. Microstructure and atomic structure of these films were studied by X-ray diffraction and high-resolution electron microscopy, while elemental analysis was performed using electron energy loss spectroscopy and X-ray dispersive analysis. The four-point probe resistivity measurements in the temperature range (10-300K) showed room-temperature values to be 15-30µ Ù- cm for á-Ta, 180-200µ Ù-cm for â-Ta and 250-275µ Ù-cm for a-Ta. The temperature coefficient of resistivity (TCR) for á-Ta and â-Ta were found to be positive with characteristic metallic behavior, while TCR for a-Ta was negative, characteristic of high-resistivity disordered metals. We discuss the mechanism of formation of a-Ta and show that it is stable in the temperature range 650-7000C with a superior Cu diffusion barrier characteristics.

11:30 AM
Microstructural Evolution of Nickel Nanoparticle Catalysts Supported on Gadolinium-Doped Ceria During Autothermal Reforming of Iso-Octane: V. Palaniyandi[1]; *Earl T. Ada*[1]; M. Shamsuzzoha[1]; Giovanni Zangari[2]; R. G. Reddy[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., PO Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]University of Virginia, Dept. of Matls. Sci. & Engrg., Charlottesville, VA 22904 USA

The microstructure and composition of a nanoparticle Ni catalyst supported on gadolinium-doped ceria (Ce1-xGdxO(4-x)/2) were studied using Transmission Electron Microscopy (TEM), X-ray Diffraction (XRD), and X-ray Photoelectron Spectroscopy (XPS). The support of the fresh catalyst exhibits a homogenous aggregation of crystalline grains, with sizes ranging between 20 and 50 nm. The crystalline structure of the fresh catalyst support is of the CeO2 phase, in which gadolinium atoms exist in a solid solution of CeO2. Nickel in the fresh catalyst is highly dispersed and forms granular crystals 5 - 30 nm in size on the surface of the ceria support. The support of the used catalyst exhibits a bimodal distribution of grains in which smaller grains have similar structure and morphology as those in the fresh catalyst, while the larger sized grains appear dull and exhibit non-faceted crystal morphology resulting either from the sintering of a number of CeO2 grains, or by the occupation of highly defective crystals of Ce2O3 and CeO phases. A thin amorphous layer of carbon also covers most of the larger grains in the used catalyst. Ni particles could not be imaged by TEM in the used catalyst, but Energy Dispersive X-ray Spectroscopy (EDX) detected their presence. XPS analysis of the catalyst samples suggests the participation of lattice O atoms from the ceria support in the catalytic reaction. XPS data also show the presence of carbonate species and a higher hydrocarbon concentration in the used catalyst.

11:45 AM Invited
Nitride-Based Thin Films Processed by Pulsed Laser Deposition: *Haiyan Wang*[1]; Xinghang Zhang[2]; Ashutosh Tiwari[3]; A. Gupta[3]; Jagdish Narayan[3]; [1]Los Alamos National Laboratory, MST-STC, MS K763, Los Alamos, NM 87545 USA; [2]Texas A&M University, Dept. of Mechl. Engrg., College Sta., TX 77843-3123 USA; [3]North Carolina State University, Matls. Sci. & Engrg. Dept., Raleigh, NC 27695 USA

Nitride-based materials have attracted broad interest due to their high hardness, superior wear resistance, high temperature thermal stability, and exceptional optical and electrical properties. These types of materials have wide applications as super-hard coatings, diffusion barriers in semiconductor industry, and light-emitting-diodes. Various PVD and CVD techniques have been explored to synthesize nitride-based materials. Among them, pulsed laser deposition (PLD) shows exceptional advantage in producing high quality complex composites. Some successful examples on TiN, TaN, and TaN-TiN binary components will be shown. Microstructures of these thin films were studied extensively by X-ray diffraction, transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM). The concept of domain matching epitaxy will be introduced for understanding the epitaxial growth of high lattice mismatch systems. Elec-

trical, mechanical properties and diffusion barrier characteristics of these nitride-based materials were also studied.

12:15 PM
Growth of High Quality Epitaxial ZnO/Pt Bilayer and ZnO-Pt Nanocomposite Structure on Sapphire (0001) for Transparent Conducting Applications: *Amit Chugh*[1]; [1]North Carolina State University, Matls. Sci. & Engrg., 2141 Burlington Labs, CB 7916, Raleigh, NC 27695-7916 USA

We have grown high quality ZnO/Pt bilayer and ZnO-Pt nanocomposite structure on Sapphire substrate (0001). These films were grown heteroepitaxially on sapphire (0001) substrates by Pulsed Laser Deposition (PLD). Epitaxial relationship between sapphire and platinum, platinum and zinc oxide was studied using X-Ray diffraction, high resolution transmission microscopy (HRTEM) and SAED experiments. Experimental results and epitaxial analysis was compared with theoretical simulations. Electrical property measurements were performed using four-point probe method over the temperature range of 15 K - 300 K. Optical transmission and absorption studies were performed using Hitachi spectrophotometer, quite high transmission of visible light was observed. Room temperature photo luminescence studies also indicate high optical quality of these films. These heterostructures demonstrate the feasibility of integrating them with optoelectronic devices.

TUESDAY AM

General Abstract Session: Nanostructured and Lightweight Materials

Sponsored by: TMS
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Tuesday AM Room: 2007
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Farghalli A. Mohamed, University of California, Dept. of Cheml. Engrg. & Matls. Sci., Irvine, CA 92697-2575 USA

8:30 AM
Grain Growth Behavior in Near Nanostructured Al 5083 Cryomilled Alloy on Isothermal Annealing: *Indranil Roy*[1]; Manish Chauhan[1]; David Edward McDougall[1]; Farghalli A. Mohamed[1]; [1]University of California, Chem. Engrg. & Matls. Sci., 916 Engrg. Tower, Irvine, CA 92696-2575 USA

Grain growth behavior in the near nanostructured Al 5083 alloy of an average grain size of 300 nm, processed by consolidating nanocrystalline cryomilled powder particulates, was investigated in the temperature range of 0.55 to 0.85 Tm, where Tm is the melting temperature, for different annealing times varies from 1 to 50 hours. Appreciable grain growth was observed at 0.85 Tm, whereas there was nominal grain growth at 0.67 Tm. The value of the time exponent 'n' was deduced from the grain growth equation of the general form D1/n - Do1/n = kt was 0.1 at 0.85Tm and 0.08 at 0.73Tm which decreased with decreasing temperature and approached 0.03 as the temperature was reduced down to 0.55Tm.

8:55 AM
Carbon Nanotube/Light-Metal Composites: *Efraín Carreño-Morelli*[1]; Robert Schaller[2]; [1]University of Applied Sciences of Western Switzerland, Design & Matls. Unit, Rte. du Rawyl 47, Sion CH-1950 Switzerland; [2]Swiss Federal Institute of Technology Lausanne, Inst. of Physics of Complex Matter, Lausanne CH-1015 Switzerland

Novel light metal matrix composites have been processed by powder metallurgy. The feasibility of manufacturing magnesium- and aluminium-based composites reinforced with carbon nanotubes has been assessed. Blends of metal powders and CVD processed multi-wall carbon nanotubes were compacted by uniaxial hot pressing followed by hot isostatic pressing. A uniform dispersion of nanotubes in the metal matrix was obtained. Sintering conditions were found, which allowed to obtain bulk specimens with sound mechanical properties.

9:20 AM
Structure-Property Relationships in Cryomilled Al-Mg Alloys: *David B. Witkin*[1]; Bing Q. Han[2]; Enrique J. Lavernia[2]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Irvine, CA 92697-2575 USA; [2]University of California, Cheml. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

Cryomilling, or ball milling within a cryogenic medium, is a mechanical attrition technique that both refines the microstructure and introduces nanometer-scale dispersoids that stabilize the grain size and strengthen the material. Consolidation and extrusion of cryomilled Al 5083 gives a material whose strength is more than twice that of conventional wrought, strain-hardened Al 5083, with comparable ductility. During the course of processing, the average grain size increases from 25 nm in the as-milled powder, to approximately 100 nm in the primary consolidated form (HIPped) and then to between 200 and 250 nm in the final extrusions. At room temperature, the yield stress, tensile strength, and flow stress of the extrusions are roughly 20 percent higher than that of the HIP material despite the larger grain size. This behavior can be understood in light of a bimodal microstructure and the processing history. The microstructural changes that occur during thermomechanical processing were further investigated by uniaxial compression of the HIP material at elevated temperatures bracketing the extrusion temperatures.

9:45 AM
Numerical Simulations of the ECAE Process with Copper, Second Pass: *Andrey Smolyakov*[1]; Petr Nizovtzev[1]; Vyacheslav Solovjev[1]; Alexander Korshunov[1]; [1]RFNC VNIIEF, Theoretical, Mira, 37, Sarov Russia

ECAP second pass simulation has been performed with isotropic model of material behavior. Satisfactory agreement between numerical and experimental data has been obtained on billet final shape and pressing force during ECAP. This approach enables simulations of subsequent ECAP passes for different routes.

10:10 AM Break

10:30 AM
Study on Method of Increasing Viscosity in Fabricating Aluminium Foam: *H. J. Luo*[1]; G. C. Yao[1]; X. M. Zhang[1]; Y. H. Liu[1]; [1]Northeastern University, Sch. of Matls. & Metall. 110004 China

The method of directly foaming in molten Al to prepare closed-cell aluminum foam is descried in this article. Different kinds of ingredients are put into the molten Al alloy to make its viscosity increased. The TiH2 as foaming agent is also added into these molten Al to fabricate aluminum foam. The effectiveness of the method to increase the viscosity of molten Al on foam structure is researched by macroscopic observation and microstructure analysis. The results are showed as follow. After these ingredients, such as calcium, magnesium, Al2O3 and coal ash, etc, had been added into molten Al, the phase component of Al alloy changed. However the phase component and the mechanism of its increasing viscosity is different slightly. Meanwhile, the obtained Al foam also has great difference in its structure. The foams obtained by calcium and coal ash hold thick cell wall and high intensity, in the other hand the foams obtained by magnesium and Al2O3 hold thin cell wall and low intensity.

10:55 AM
Effect of Cu on the Formation Behavior of Intermetallic Compounds in Al-Mn Wrought Alloy: *Young-Ok Yoon*[1]; Dae-Guen Kim[1]; Young-Jig Kim[1]; Hanshin Choi[2]; Hoon Cho[2]; Hyung-Ho Jo[2]; [1]Sungkyunkwan University, Dept. of Advd. Matls. Engrg., 300 Chunchun-Dong, Jangan-Gu, Suwon, Gyounggi-Do 440-746 S. Korea; [2]Korea Institute of Industrial Technology, Advd. Matls. Ctr., 994-32, Dongchun-Dong, Yeonsu-Gu, Incheon 406-130 S. Korea

During the extrusion of Al-Mn wrought alloy to manufacture tube and fin for heat exchangers, sticking between alloy billet and die is regarded as the main drawback for workability. In fact, the sticking makes various deteriorations in die cleanness and surface properties of as-extruded tube or fin. It is well known that the sticking mainly depends on the intermetallic phase formation. The main emphasis of this study is to investigate the effect of Cu content on the formation behavior of intermetallic compounds. The quantification and the phase evolution of intermetallic compounds were intensively conducted. With increasing Cu content, the fraction and size of intermetallic compounds were notably increased and the distribution of them was improved. The tensile strength and hardness of them were also increased.

11:20 AM
A Study on the Casting and Properties of Al Foam: *Bo-Young Hur*[1]; Sang-Youl Kim[1]; Kwang-Ho Song[2]; Yong-Su Um[1]; [1]Gyeongsang National University, Jinju 660-701 Korea; [2]Daelim College, Kyungkido Korea

Al alloy foam used in this study was prepared that Al alloy was molten using a high-frequency induction furnace. The molten Al alloy

was put in a pot furnace for a specific period to make it reach the temperature appropriate for foaming. Thickening and foaming agent were added to the molten Al alloy at a specific temperature, which was stirred. An impeller with 3-stage screw welded was used to stir the molten Al alloy. The impeller was rotated clockwise to generate vortex flow toward the center. For uniform distribution of thickening and foaming agent, the impeller was rotated with high-speed about 400-1000rpm. When left in a furnace for a specific period after uniform dispersion of the foaming agent, gas was generated from the foaming agent and the cell size and distribution inside Al alloy foam could be controlled by the viscosity and surface tension of the molten Al alloy. Al alloy foam has good sound and energy absorption properties.

11:45 AM
Plasma Coating and Magnetic Alignment of Carbon Nano Fibers in Polymer Composites: *Donglu Shi*[1]; Peng He[2]; Jie Lian[3]; Xavier Chaud[4]; Eric Beaugnon[4]; Luming Wang[3]; Rodney Ewing[3]; Robert Tournier[4]; [1]University of Cincinnati, Cheml. & Matl. Engrg., 2624 Clifton Ave., Cincinnati, OH 45221 USA; [2]University of Cincinnati, Mechl. Engrg., 2624 Clifton Ave., Cincinnati, OH 45221 USA; [3]University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109 USA; [4]Consortium de Recherches pour l'Emergence de Technologies Avancées et Laboratoire de Cristallographie, Grenoble Cedex 38042 France

An extremely thin layer of polymer film has been coated onto both outer and inner surfaces of the nanotubes. Due to surface modification, the dispersion of nanotubes in the polymer matrix is significantly enhanced. HRTEM images, SIMS results of coated surface films on nanotubes, and mechanical properties of the composites will be presented. For fundamental study and novel engineering applications, carbon nanotubes also need to be aligned along certain specified directions. In this study, we present a novel method by which these nanotubes can be well aligned in a polymer matrix at moderate magnetic field. Both TEM and SEM results show clear evidence of well aligned nanotubes in the polymer composite. The magnetic alignment mechanism is discussed.

General Abstract Session: Advances in Steels

Sponsored by: TMS
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Tuesday AM Room: 2011
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Philip Nash, IIT, Mechl., Matls. & Aeros. Engrg. Dept., Chicago, IL 60616 USA

8:30 AM
The Role of Impurities and Processing in Damascus Steels: *Khalid Mawani*[1]; [1]Ghulam Ishaq Khan Institute, Matls. & Metallurgl. Engrg., Rm. No. 94, Hostel 5, Topi Swabi, NWFP 23460 Pakistan

Sir Walter Scott, in his book "THE TILSIM" writes about the encounter of Sultan Salahuddin Ayubi and Richard in the thirteenth century. Describing the blades of the swords used by the army of Sultan was not so heavy like the swords used by his European opponent. It was dull blue and marked with millions of meaningless lines. Sultan used to astound people by flying the handkerchief and slicing it into two. Most important thing about these swords were that they never became blunt even after the heavy clashes and were sharp enough to cleave a man in half with only one hand. The secret of these swords lies in the Damascus steel. This steel was not produced in Damascus but since the European army first came across this blade in Damascus that is why it is known as Damascus steel. It is believed that the raw materials for these blades were made in India which is known as the "Wootz Steel", where the process of making Wootz continued till the 19th century. Unfortunately, the technique of producing Wootz Damascus steel blades is a lost art. There are two schools of thought on the processing of these blades. One is that the beautiful patterns present in the swords were because the pattern-welded steels were produced by forge welding alternating sheets of high- and low-carbon steels. This composite was then folded and forge-welded together, and the fold/forge cycle was repeated until a large number of layers were obtained while, other is that the pattern on the sword are because of the hypereutectoid carbon level of these steels. In this review paper we will discus the processing method on the basis of facts of history and the knowledge of metallurgy in the ancient times.

9:00 AM
Influence of Co and Ni on Mechanical Properties in Ultrahigh Strength Secondary Hardening Steels: *Ho Seop Sim*[1]; Kon Bae Lee[1]; Heang Ryeal Yang[2]; Hoon Kwon[1]; [1]Kookmin University, Sch. of Advd. Matls. Engrg., 861-1, Jongnung-dong, Songbuk-ku, Seoul 136-702 Korea; [2]Incheon City College, Dept. of Mechl. Engrg., Incheon 402-750 Korea

Alloys were designed by the variation of Co(5-13 wt%) and Ni(8-14 wt%) based on AerMet 100(0.23C-1.2Mo-3.1Cr-13.4Co-11.1Ni). These alloys were severely rolled by 50%/2p at 700 or 850 C in the low austenite region and then direct quenched after reheating in the range 900-1200 C. In the as-quenched condition, tensile strength to hardness ratio was higher as compared to the aged condition. Influences of reheating and rolling temperatures on hardness/tensile strength(UTS) and impact toughness did not show a clear difference in the aged condition since the aging appeared to screen the effects of those prior treatments. In the aged condition, the hardness(Rc)/UTS(MPa) was arranged as follows; 11Ni-13Co(57/2130) > 14Ni-13Co(57/2115) > 8Ni-13Co(56.5/2065) > 11Ni-9Co(56/2025) > 8Ni-9Co(55/1945) > 11Ni-5Co(54/1905). Co did not promote aging despite the increased hardness/UTS whereas Ni promoted aging accompanied by an increase in hardness.

9:30 AM
Microstructure and Elevated Temperature Stability of 9-12% Cr Steels: *Omer N. Dogan*[1]; Jeffrey A. Hawk[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

Medium Cr steels have been used in fossil fired power plants for many years because of their excellent high temperature stability and mechanical properties. As the desire to increase the efficiency of power plants continues, the operating temperature (>650C) continues to go up. Currently available low and medium Cr containing steels will not withstand the new operating temperature and must be reassessed in terms of their solid-solution and precipitation strengthening schemes. Three medium Cr steels were developed to investigate high temperature alloy strengthening strategies: 0.08C-(9-12)Cr-1.2Ni-0.7Mo-3.0Cu-3.0Co-0.5Ti. The microstructure of the alloy will be described in the as-cast and thermo-mechanically worked states. In addition, the effect on microstructure from long-term high temperature exposure will also be discussed. Finally, the overall stability of these steels will be compared against currently available power plant steels.

10:00 AM
Oxidation Resistance of 9-12% Cr Steels: Effects of Rare Earth Surface Treatments: *Omer N. Dogan*[1]; *Jeffrey A. Hawk*[1]; David E. Alman[1]; Paul D. Jablonski[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

Medium Cr steels have been used in fossil fired power plants for many years because of their excellent high temperature stability and mechanical properties. The environment in a fossil fired power plant is extremely aggressive in terms of corrosion, especially oxidation. This is only accelerated as the operating temperature increases to 650C and beyond. For any new steel to be qualified for power plant use, in addition to adequate strength at the operating temperature, material wastage from all corrosion processes must be kept to a minimum acceptable level. The use of medium Cr steels provides a means to improve overall corrosion resistance. Three medium Cr are under development for use as high temperature power plant steels: 0.08C-(9-12)Cr-1.2Ni-0.7Mo-3.0Cu-3.0Co-0.5Ti. Oxidation tests were performed on the steels for times greater than 1000 hours in order to determine the oxidation kinetics and extent of material wastage. Also, rare earth oxides were incorporated into the outer surface layers of the steels to see if the oxidation resistance could be improved. These results will be compared to current power plant steels.

10:30 AM Break

10:50 AM
Room and Elevated Temperature Mechanical Behavior of 9-12% Cr Steels: Omer N. Dogan[1]; *Jeffrey A. Hawk*[1]; Karol K. Schrems[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

The mechanical properties of medium Cr steels used in fossil fired power plants are very good because of their excellent high temperature microstructural stability. However, as the desire to increase the operating temperature (>650C) of the plant goes up, the need for steels that maintain their strength at these temperatures also increases.

The mechanical properties of three medium Cr steels (0.08C-(9-12)Cr-1.2Ni-0.7Mo-3.0Cu-3.0Co-0.5Ti) were investigated through hardness, hot hardness and tensile measurements. The strength of the 9-12%Cr steels at room temperature after long-term isothermal aging (750C; 1000 hours) compares favorably with that of other power plant steels (e.g., P91). In addition, the elevated temperature strength and hot hardness also behave similarly. The mechanical behavior will be discussed in terms of the strength, elongation and tensile fracture characteristics.

11:20 AM
Using Hardness to Model Yield and Tensile Strength: *Jeffrey A. Hawk*[1]; Omer N. Dogan[1]; Karol K. Schrems[1]; [1]U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

The current direction in hardness research is towards smaller and smaller loads as nano-scale materials are developed. There remains, however, a need to investigate the mechanical behavior of complex alloys for severe environment service. In many instances this entails casting large ingots and making numerous tensile samples as the bounds of the operating environment are explored. It is possible to gain an understanding of the tensile strength of these alloys using room and elevated temperature hardness in conjunction with selected tensile tests. The approach outlined here has its roots in the work done by Tabor for metals and low alloy and carbon steels. This research seeks to extend the work to elevated temperatures for multi-phase, complex alloys. A review of the approach will be given after which the experimental data will be examined. In particular, the yield stress and tensile strength will be compared to their corresponding hardness based values.

11:50 AM
Bainite Transformation in Low Carbon Steel Simulated by Gleeble: *Smati Chupatanakul*[1]; Philip Nash[1]; Robert Binoniemi[2]; [1]IIT, Thermal Process Tech. Ctr., IL 60616 USA; [2]DANA Corporation

Low carbon 43 series alloy steel is a popular carburizing grade steel for automotive gears. Generation of a high carbon bainitic case microstructure and low carbon martensitic core microstructure may provide significant benefit for gear applications where toughness dictates gear performance. In this paper, Gleeble 3500 is used to simulate the real process. In order to get a homogenous starting microstructure in the samples, each specimen is austenitized at 915°C for 5 minutes. After that the samples are cooled down to an austempering temperature above the Ms temperature and held for a period of time to get a bainitic structure. SEM and dilatometry were used to determine the phase fraction in the steel. These data are put into Avrami's equation to get the time exponent n, about 1.5 for transformation from austenite to bainite.

Hume-Rothery Symposium: The Science of Complex Alloys

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Chemistry & Materials Science, Livermore, CA 94551 USA; Thaddeus B. Massalski, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

Tuesday AM Room: 3008
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Chmst. & Matls. Sci. Direct., Livermore, CA 94551 USA; Igor A. Abrikosov, Linköping University, Dept. of Physics & Measurement Tech., Linköping 58183 Sweden

8:30 AM Invited
Pseudo-Gap at the Fermi Level in Al Based Intermetallics: *Esther Belin-Ferre*[1]; [1]CNRS, LCPMR UMR 7614, Université P. & M. Curie, 11 rue Pierre et Marie Curie, Paris 75231 France

Series of alloys are stabilized by a Hume-Rothery mechanism, namely interaction between Bragg planes and electronic waves. This induces a depletion in the electronic densities of states at the Fermi level, the so-called pseudo-gap. Many Al-Cu-based quasicrystals are formed of elements with small differences in atomic radii and electronegativity and their average electron per atom ratio equal same values as in Hume-Rothery phases. Therefore, it was proposed that a Hume-Rothery mechanism should be responsible for their stability. Actually, a pseudo-gap at the Fermi level was predicted from band structure calculations of model phases with local order similar to that of the quasicrystals. It was also observed with different experimental spectroscopic techniques on various quasicrystalline systems. The paper will discuss from the standpoint of experimental spectroscopy data, to which extent the electronic density of states in Al-based quasicrystalline compounds is sensitive to Hume-Rothery stabilization by comparing to Al-Cu and Al-Cu-Fe Hume-Rothery alloys.

9:00 AM Invited
Electronic, Magnetic and Transport Properties of the Pseudogap Fe2VAl System: *Yoichi Nishino*[1]; [1]Nagoya Institute of Technology, Dept. of Matls. Sci. & Engrg., Showa-ku, Nagoya 466-8555 Japan

While the Heusler-type Fe2VAl compound exhibits a semiconductor-like behavior of electrical resistivity, band calculations predict that it is a nonmagnetic semimetal with a sharp pseudogap right at the Fermi level. A substantial mass enhancement deduced from electronic specific-heat measurements suggests that the unusual electron transport is mainly interpreted in terms of the effect of strong spin fluctuations in addition to the possession of a low carrier density. Doping of quaternary elements causes a sharp reduction in the resistivity and a large enhancement in the Seebeck coefficient. Remarkably the Seebeck coefficient plotted against the average electron concentration, instead of the composition of doping elements, is found to fall on a universal curve irrespective of doping elements. Substantial enhancements for the Seebeck coefficient can be explained by using the electronic structure where the Fermi level shifts slightly from the center of the pseudogap.

9:30 AM Invited
Semiconducting Al-Transition-Metal Alloys: *Marian Krajci*[1]; Juergen Hafner[2]; [1]Slovak Academy of Sciences, Inst. of Physics, Dubravska cesta 9, Bratislava SK-84511 Slovak Republic; [2]University of Vienna, Inst. for Matls. Physics, Sensengasse 8/12, Vienna A-1090 Austria

In intermetallic compounds formed by transition-metals (TM) and Al, in some cases a special Al-TM ordering can lead to the formation of a semiconducting band-gap in the electronic spectrum. On the basis of ab-initio electronic structure calculations we studied semiconductivity in some crystalline and quasicrystalline phases. We found that the semiconducting behavior is accompanied with a formation of chemical bonds between aluminum and TM atoms with a high degree of covalency. The bonds form a network with a special Al-TM ordering. Any deviation from this ordering, for instance a existence of substitutional defects, leads to formation of localized states in the gap. We investigated the mechanism of the band-gap formation. The hybridization plays here an important role but for a true band-gap formation the hybridization alone is not sufficient. A breaking of symmetry of the crystal structure via Peierls-like mechanism is also very essential.

10:00 AM Break

10:20 AM Invited
Non-Metallic Properties in Metallic Alloys: An Electron Valence Effect: *Joseph Poon*[1]; [1]University of Virginia, Physics, McCormick Rd., POB 400714, Charlottesville, VA 22904 USA

The electronic properties of quasicrystals and intermetallic compounds will be discussed as examples of Fermi-surface-Jones-Zone interaction effects. When the valence electronic counts are right, it can give rise to a pseudogap or semiconducting gap. Novel properties including electron localization and robust bandgap behaviors unexpected of metallic systems are observed when strong hybridization is coupled with complex crystal structures. Some examples obtained from insulating quasicrystals and semiconducting intermetallic compounds that exhibit promising thermoelectric properties will be described.

10:50 AM Invited
Prediction of Site Preference and Phase Stability of Transition Metal Based Frank-Kasper Phases: *Marcel H.F. Sluiter*[1]; Alain Pasturel[2]; Yoshiyuki Kawazoe[1]; [1]Tohoku University, Inst. of Matls. Rsch., 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan; [2]CNRS, Lab. de Physique et Modélisation des Milieux Condensés, Grenoble France

Site preference of transition metals for crystal sites in Frank-Kasper type tetrahedrally close packed phases has been computed using density functional based electronic structure methods and cluster expansions. Comparisons with results from X-ray and neutron scattering experiments, where available, indicate that theoretical results are accurate. The cluster expansion method is used to analyze whether site preference is driven by onsite effects (such as atomic size) or chemical ordering effects (such as maximization of unlike bonds). Moreover the

role of vibrational degrees of freedom on the sire preference is discussed.

11:20 AM Invited
Phase Stability in Refractory Metal Silicide Phases: Ridwan Sakidja[1]; *John H. Perepezko*[1]; [1]University of Wisconsin, Dept. of Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA

Refractory metal (RM) silicide phases are attractive as high temperature structural materials and at low temperature for microelectronic applications. While silicides can develop oxidation resistant SiO2 coatings, multiphase alloy designs are necessary to achieve satisfactory structural performance. The observed alloying trends in ternary and higher order systems highlight the fundamental factors such as geometric rules that govern phase stability. Often, the drive to achieve a high packing density of metal and metalloid constituents yields a strict range of atomic size ratios of metal to metalloid that favors structural stability. By following the guidance offered by the geometric rule it has been possible to design new multiphase microstructures by systematically modifying the phase stability. Usually, silicides are stoichiometric, but there are cases with a homogeneity range where defect structures are important. The basis for the structural stability analysis is derived from the behavior in RM-Si-B and RM-Si-C systems.

Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Lead-Free Solder Alloy Development

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Mark A. Palmer, Kettering University, IMEB, Flint, MI 48504-4898 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Nikhilesh Chawla, Arizona State University, Department of Chemical and Materials Engineering, Ira A. Fulton School of Engineering, Tempe, AZ 85287-6006 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada

Tuesday AM Room: 3014
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: James Lucas, Michigan State University, Chem. Engrg. & Matl. Sci., E. Lansing, MI 48824-1226 USA; Iver E. Anderson, Iowa State University, Ames Lab., Ames, IA 50011-3020 USA

8:30 AM Invited
Development of Low Melting Temperature Lead-Free Solder Pastes for High Temperature Applications: *Randall M. German*[1]; Louis G. Campbell[1]; [1]Pennsylvania State University, Ctr. for Innovative Sintered Products, 147 Research W., Univ. Park, PA 16802 USA

Gas atomization was used to formulate rapidly solidified tin-based alloy powders for use in a high temperature application. The powders were mixed with reactive alloying additions and formed into pastes useful in automated assembly. For target applications such as outdoor lighting, the creep properties of lead-free tin-based solders is too low. Yet rapid flow during assembly requires a low melting temperature solder. The normally low melting temperature solders required formation of an reactive alloy containing titanium to induce transient liquid phases and precipitation reactions for strengthening. Elevated temperature testing showed that only reactive powders could pass the standard tests. Phase relation rules were formulated with respect to the reaction events needed to form the desired transient phases; the rules considered both solder flow and subsequent reaction with the substrate to form an elevated temperature alloy solder.

9:00 AM
Microstructure and Long Term Reliability of Lead-Free Solder Joints Using Low Temperature Solder Paste: *Wang Ju Lee*[1]; Un-Byoung Kang[1]; Hee Kook Choi[1]; Young-Ho Kim[2]; Se Yong Oh[1]; [1]Samsung Electronics, Memory Div., San #74 Buksu-Ri, Baebang-Myeon, Asan, Chungcheongnam-Do 336-711 Korea; [2]Hanyang University, Div. of Matls. Sci. & Engrg., 17 Haengdang-dong, Seongdong-Ku, Seoul 133-791 Korea

A low temperature packaging is required for some electronic devices containing heat-sensitive components or for low cost Pb-free solution in the near future. In this paper, the ball grid array (BGA) technique using high temperature Pb-free solder ball and eutectic Sn-Bi solder paste is introduced. Sn-3.0Ag-0.5Cu solder balls with a diameter of 450 §- were attached on a Fine-pitch Ball Grid Array(FBGA) package, and eutectic Bi-Sn solder paste was printed on OSP-Cu and Au/Ni pads of PCB substrate. The peak temperature during reflow was fixed at 175¡É which is much lower than that of eutectic Sn-Pb solder, and the reflow time over 139¡É, the Bi-Sn eutectic temperature, was 110 sec. During soldering at 175¡É, the lead-free ball of the package does not melt but the eutectic Sn-58Bi paste does and wets to the lead-free ball. The long term reliability of Pb-free solder joint using Sn-58Bi paste was evaluated, and its microstructure change and intermetallic compound formation was investigated before and after reliability test. After soldering, the Bi content in the bottom of solder joint was confirmed to be decreased down to 12 wt%-20 wt% by energy dispersive spectroscopy (EDS). The melting temperature of solder joint after soldering increased up to 190¡É-200¡É, which was attributed to the interdiffusion of Bi and Sn during soldering process. The intermetallic compound thickness formed between Sn-Bi solder paste and PCB pad was much smaller than that of Sn-3.0Ag-0.5Cu paste in both OSP-Cu and Au/Ni PCB pads. The reliability of Pb-free solder joint using Sn-58Bi paste was as high as that of using Sn-3.0Ag-0.5Cu paste at soldering temperature over 250¡É in thermal cycling test. After thermal 2000 cycles, the intermetallic compound thickness formed between Sn-Bi solder paste and PCB pad was same as that of Sn-3.0Ag-0.5Cu paste in both OSP-Cu and Au/Ni PCB pads. The Cu6Sn5/Cu3Sn double intermetallic layers were formed at the interface between Sn-Bi solder paste and OSP-Cu pad after thermal cycles while the Ni3Sn4 layer was only found at the interface of Au/Ni pad. It is expected that the bonding technique using Sn-Ag-Cu solder ball and eutectic Bi-Sn solder paste is a promising method for the board level packaging. Especially low temperature soldering using Sn-Bi paste is expected to be used widely in application area which has low temperature user environment such as consumer, office, etc. for low cost lead-free solution in the near future.

9:20 AM
Role of Shape-Memory Alloy Reinforcements on the Evolution of Strain Localization in Lead-Free Solder Joints: Deng Pan[1]; Chanman Park[1]; Shuwei Ma[1]; *I. Dutta*[1]; B. S. Majumdar[2]; [1]Naval Postgraduate School, Dept. Mech. Astro. Engr., 700 Dyer Rd., Monterey, CA 93943 USA; [2]New Mexico Tech, Dept. of Matls. Sci. & Engrg., Socorro, NM 87801 USA

Microelectronic solder joints are exposed to aggressive thermo-mechanical cycling (TMC) conditions during service. During TMC, severe inelastic strain localization can occur within joints, eventually causing low-cycle fatigue failure. In order to mitigate the effects of strain localization, a composite solder reinforced with NiTi based shape memory alloy (SMA) particles are being developed. In this scheme, the initially martensitic SMA reinforcement is heavily deformed along with the solder in the early stages of TMC. Upon transforming into austenite at the As temperature, the reinforcement undergoes shape recovery, thereby placing the adjoining solder matrix in reverse shear, and thus reducing strain localization within the solder joint. In this paper, we present the results of experimental work on evaluating the impact of the martensite-to-austenite (M?¨A) transformation on matrix strain redistribution in the immediate vicinity of a reinforcement, using in situ thermo-mechanical loading experiments on a cross-sectioned sample inside an SEM. A comparison of in situ strain localization within monolithic Sn-4.7%Ag-1%Cu joints, joints reinforced with 'passive' Cu reinforcements, and joints reinforced with 'active' NiTi reinforcements will be presented. The results of concurrent finite element modeling (FEM) to elucidate the role of phase transformations within NiTi on the evolution of strain distribution within the joint will also be presented.

9:40 AM
Effect of Zn Content on the Vibration Fracture Behavior of Sn-Zn and Sn-Zn-Bi Solders: *Jenn-Ming Song*[1]; Yea-Luen Chang[2]; Truan-Sheng Lui[2]; Li-Hui Chen[2]; [1]National Dong Hwa University, Dept. of Matls. Sci. & Engrg., Hualien 974 Taiwan; [2]National Cheng Kung University, Dept. of Matls. Sci. & Engrg., Tainan 701 Taiwan

Given that plastic deformation, even failure, may occur due to vibration, the vibration fracture resistance of the solder should be taken into consideration during alloy design. To realize the effect of Zn content on the vibration properties of the Sn-Zn-Bi system, this study aimed to explore the vibration fracture behaviors of binary Sn-

xZn (x=7, 9, 11, 13wt%) solder alloys, as well as ternary Sn-xZn-3Bi(x=5, 8, 11wt%). Sn-9Zn has the poorest damping capacity and the lowest critical cycles to failure among the Sn-xZn alloys. On the other hand, the Sn-13Zn samples with massive primary Zn needles possess superior damping capacity and vibration life. As to Sn-xZn-3Bi alloys, both the damping capacity and vibration fracture resistance decrease in turn from Sn-11Zn-3Bi, Sn-8Zn-3Bi to Sn-5Zn-3Bi. The main crack of the Sn-5Zn-3Bi tends to propagate along the phase boundaries between proeutectic Sn and Sn-Zn eutectics.

10:00 AM
A Study on Oxidation of Sn and its Alloys by Electrochemical Reduction Analysis: *Sungil Cho*[1]; Jin Yu[1]; Sung K. Kang[2]; Da-Yuan Shih[2]; [1]Korea Advanced Institute of Science and Technology, Matls. Sci. & Engrg., 373-1 Gusung-dong, Yusung-gu, Daejon 305-701 S. Korea; [2]IBM, T. J. Watson Rsch. Ctr., 1101 Kitchawan Rd., Rte. 134, Yorktown Heights, NY 10598 USA

In a microelectronic system, solder joints are constantly exposed to temperature, humidity, stress and other reactive environments. Oxidation (and/or corrosion) of solder joints could be one of the major yield and reliability risk factors. Hence, an in-depth understanding on oxidation behaviors of solder interconnections becomes a critical issue in making solder interconnection technologies successful. However, the oxidation behaviors of Sn and Sn alloys have not been investigated in detail. Tin is a base material of Sn-Pb and Pb-free solders and little is known about its oxidation behavior at various conditions of processing or applications. In this study the solid-state oxidation of pure Sn, Sn-Pb alloys and Sn-base Pb-free solders was investigated by electrochemical reduction analysis method, which can provide the information on the chemistry and the amount of oxides. Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) were also employed to confirm the results obtained from the electrochemical analysis.

10:20 AM Break

10:30 AM
Unusual Spalling of Cu_6Sn_5 Induced by Au/Ni Surface Finish in Sn3.0Ag0.5Cu Solder Joints: *Cheng-En Ho*[1]; Wei-Chen Luo[1]; Eu-Wei Lin[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matls. Engrg., Chungli City 320 Taiwan

Gold/nickel bi-layer is one of the most common and important surface finishes for treating Cu pads in advanced packages. In this talk, a very interesting phenomenon induced by the Au layer of the Au/Ni surface finish in contact with the Sn3.0Ag0.5Cu solder will be presented. Experimentally, Sn3.0Ag0.5Cu spheres were reflowed on Au/Ni/Cu pads, and chemical interactions between the solder and the pads were investigated. Two kinds of intermetallics, $(Cu,Au,Ni)_6Sn_5$ and $(Ni,Cu)_3Sn_4$, coexisted at the solder/Ni interface after reflow. It was found that with increasing reflow time $(Cu,Au,Ni)_6Sn_5$ exhibited a very different morphology, from chunk-like to a continuous layer. Thick $(Cu,Au,Ni)_6Sn_5$ layer eventually spalled from the $(Ni,Cu)_3Sn_4$ into the molten solder as the reflow time reached 60 sec. In contrast, when Sn3.0Ag0.5Cu was reflowed on Ni/Cu, only two dense layers, $(Cu,Ni)_6Sn_5$ and $(Ni,Cu)_3Sn_4$, formed at the interface. The result indicated that the Au layer played an important role in this spalling phenomenon.

10:50 AM
Synthesis of Ni_3Sn_4 and Cu_6Sn_5 Nanoparticles in Deriving Lead-Free Composite Solders: *Li-Yin Hsiao*[1]; Szu-Tsung Kao[1]; Hsiang-Yi Lee[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101, Sect. 2 Kuang Fu Rd., Hsinchu 300 Taiwan

Intermetallic compounds (IMC) of Ni_3Sn_4 and Cu_6Sn_5 play important roles in the interfacial reaction between the lead-free solder and the Ni/Cu under-bump metallization during reflowing in electronics packaging. In this study, SnAgCu and SnAgNi composite solders were produced by mechanical alloying (MA) process with doping Cu_6Sn_5 and Ni_3Sn_4 nanoparticles, respectively. The nanoparticles of the Cu_6Sn_5 and Ni_3Sn_4 intermetallic compounds were synthesized by chemical precipitation with $NaBH_4$ in aqueous solutions. The nanoparticles were prepared by the precursor reacting with $NaBH_4$. A solution of appropriate metal-precursors was rapidly added to a $NaBH_4$/NaOH solution under strong stirring. After mixing these two solutions, black precipitates were immediately observed, which were washed with distilled water and then dried at room temperature. The structures of particles were characterized by X-ray diffraction (XRD), and field-emission scanning electron microscopy (FE-SEM) analysis was employed to analyze the morphology of the particles. The SnAgCu composite solder joint doped with the nanoparticles of Cu_6Sn_5 intermetallic compound formed thinner $(Cu, Ni)_6Sn_5$ layers at the solder/electroless Ni-P interface than that formed in the commercial solder joint and MA solder joint. The nanoparticles of Ni_3Sn_4 intermetallic compound doped into SnAgNi composite solder joint exhibited the similar effect.

11:10 AM
Effects of Volumetric Contraction of Electronic Solders: *Girish S. Wable*[1]; Srinivas Chada[2]; Bryan Neal[2]; Raymond A. Fournelle[3]; [1]SUNY, Dept. of Sys. Sci. & Industl. Engrg., PO Box 6000, Binghamton, NY 13902 USA; [2]Jabil Circuit, Inc., AMT/FAR Lab, 10800 Roosevelt Blvd., St. Petersburg, FL 33716 USA; [2]Jabil Circuit, Inc., AMT/FAR Lab, 10800 Roosevelt Blvd., St. Petersburg, FL 33716 USA; [3]Marquette University, Dept. of Mechl. & Industl. Engrg., 15151 W. Wisconsin Ave., Milwaukee, WI 53233 USA

Volumetric contraction during solidification is a characteristic that is exhibited by all metals, with the exception of gallium. Alloys that undergo solidification over a broad range of temperature generally exhibit a difference in the contraction behavior of their ensuing phases. Furthermore, dissolution of substrate metals during process reflow leads to meta-stable phases and volumetric contraction artifacts. The extent and frequency of surface roughness, shrinkage voids, fillet lifting and hot tearing seen in "lead-free" solders are significantly different than for "eutectic tin lead" solder. Shrinkage effects have been reported in Sn/Pb, Sn/Pb/Ag, Sn/Ag/Cu, Sn/Cu/Ni solders for various components, but few presented their impact on solder joint reliability. Nevertheless, they warrant a close observation and proper identification due to shift towards lead-free solders. This paper is a review of various defects resulting from shrinkage as well as the factors that contribute to their formation and a methodology to identify these defects effectively.

11:30 AM
Electrical and Mechanical Studies of the (Sn-Ag)eut+Cu+Sb+Bi Soldering Materials: *R. Kisiel*[1]; Gasior Wladyslaw[2]; Moser Zbigniew[2]; J. Pstrus[2]; K. Bukat[3]; J. Sitek[3]; [1]Warsaw University of Technology, Inst. of Microelect. & Optoelect., 00-662 Warszawa, Koszykowa Str. 75 Poland; [2]Polish Academy of Sciences, Inst. of Metall. & Matls. Sci., 30-059 Kraków, Reymonta Str. 25 Poland; [3]Tele and Radio Research Institute, 03-450 Warszawa, Ratuszowa Str. 11 Poland

Electrical (solder resistivity and solder joint resistance) and mechanical (tensile strength and shear strength of solder joints) parameters of quinary $(Sn\text{-}Ag)_{eut}$+Cu+Bi+Sb were investigated starting from binary eutectic Sn-Ag and close to ternary eutectic Sn-Ag-Cu alloys. The four-probe technique was used for electrical parameters measurements. Special equipment was constructed for the tensile strength measurement and also for determination of the shear strength calculation of solder joints between a typical circuit component and a Cu contact on a printed circuit board (PCB). It was found that electrical properties of the investigated alloys are comparable to the data from literature for eutectic Sn-Ag and the traditional tin-lead solders. The strong influence of Bi and Sb at the mechanical properties is observed.

Magnesium Technology 2005: Thermodynamics [Magnesium Alloys]

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Tuesday AM Room: 2004
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Bob R. Powell, General Motors Corp, R&D Ctr., Warren, MI 48090-9055 USA; Rainer Schmid-Fetzer, TU Clausthal, Inst. fuer Metall., Clausthal-Zellerfeld D-38678 Germany

8:30 AM
Experimental Investigation of the Equilibria in Mg-Al-(Ca, Sr) Systems: M. A. Parvez[2]; X. Wang[2]; *Elhachmi Essadiqi*[1]; Mamoun Medraj[2]; [1]CANMET- MTL, CANMET - Matls. Tech. Lab., 568 Booth St., Ottawa, ON K1A 0G1 Canada; [2]Concordia University, Montreal, Quebec Canada

The phase diagrams of Mg-Al-Sr and Mg-Al-Ca systems were investigated experimentally by differential scanning calorimeter (DSC) and X-ray Diffraction (XRD) techniques. The experimental work focused on the critical regions after reviewing the phase diagrams developed by thermodynamic modeling. Differential scanning calorimetry has permitted real time measurement of the phase change involved in these systems. The temperature ranges for the phase change peaks

have been determined. Enthalpy of melting and enthalpy of formation of the compounds are also reported. Comparison between these results and thermodynamic findings will be discussed. These results along with the XRD analysis are used to establish the equilibria in Mg-Al-Sr and Mg-Al-Ca systems. XRD was used to identify the phases in the studied samples. Al4Sr and Al2Ca were found to be the dominating phases in Mg-Al-Sr and Mg-Al-Ca systems, respectively.

8:50 AM
Laves Phase Stability in the Mg-Al-Ca System: *Zi-Kui Liu*[1]; Yu Zhong[1]; Alan Luo[2]; J. F. Nie[3]; Jorge O. Sofo[4]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Univ. Park, PA 16803 USA; [2]General Motors Research and Development Center, Matls. & Processes Lab., 30500 Mound Rd., Warren, MI 48090-9055 USA; [3]Monash University, Sch. of Physics & Matls. Engrg., Victoria 3800 Australia; [4]Pennsylvania State University, Dept. of Physics, Univ. Park, PA 16802 USA

Mg-Al-Ca alloys exhibit appealing creep strength at temperatures over 100C. It is believed that it is due to the substitution of the -Al12Mg17 phase by laves phases of higher thermal stability. In the literature, the C15-Al2Ca phase in the Al-Ca binary system and the C14-Mg2Ca phase in the Ca-Mg binary system have been reported. Their relative stability in the ternary system may play an important role in the development of the Mg-Al-Ca alloys. Furthermore, recent experimental studies and first-principles calculations in the literature have revealed the existence of a C36 laves phase in the Mg2Ca-Al2Ca pseudo-binary. In the present work, special quasirandom structures (SQS's) for the three laves phases, Al2Ca, Mg2Ca and (Mg,Al)2Ca, were developed to mimic their most relevant pair and multisite correlation functions in random solutions. First-principles calculations on the present SQS's were performed for the three laves phases to predict their stability over the whole composition range. Experimental investigations using individual alloys and diffusion couples were also carried out to examine the relationships and stability of the three laves phases obtained theoretically.

9:10 AM
Stress-Strain Response in Skin and Core Regions of Die Cast Magnesium Alloy AM60B Determined from Spherical Microindentation: *J. P. Weiler*[1]; J. T. Wood[1]; R. J. Klassen[1]; R. Berkmortel[2]; G. Wang[2]; [1]University of Western Ontario, Dept. of Mechl. & Matls. Engrg., London, Ontario N6A 5B9 Canada; [2]Meridian Technologies Inc., 25 MacNab Ave., Strathroy, Ontario N7G 4H6 Canada

Spherical indentation testing is used to analyze the dependence of the mechanical properties upon local microstructure of the high-pressure die cast magnesium alloy AM60B. Indentation testing is performed on two samples from the skin and the core regions of the die-casting. The difference in the indentation stress-strain flow curve between the skin and the core, and comparisons with results from uniaxial tensile testing are analyzed. It was found that results from the skin region compare well with the tensile stress-strain flow curve, while results from the core region produce a different strain hardening coefficient and a lower stress at each loading level. This study indicates that the skin region controls the flow properties of tensile samples cut from AM60B die-castings.

9:30 AM
Characterization of Local Deformation of Magnesium Alloys Using Micro-Indentation Techniques: *Lihong Han*[1]; Henry Hu[1]; Derek Northwood[1]; [1]University of Windsor, Dept. of Mechl., Auto. & Matl. Engrg., Windsor, Ontario N9B 3P4 Canada

The microstructure of die cast magnesium alloys varies significantly with the depth from casting surface. To characterize the behavior of local deformation within the vicinity adjacent to the casting surface, a micro-indentation technique has been employed in this study. The paper discusses the time-dependent mechanical response of Mg alloys subjected to a shallow indentation made by a 20μm radius spherical indenter and a three-sided pyramidal (Berkovich) diamond indenter at room temperature. Hardness, composite modulus and local stress-strain curve were obtained by measuring the penetration depth changing with time during testing. The creep characteristics under different constant load were also analyzed using this technique, and the possible mechanisms contributing to indentation creep have been identified.

9:50 AM
Thermodynamics of Mg-Zn-Zr: Implication on the Effect of Zr on Grain Refining of Mg-Zn Alloys: R. Arroyave[1]; *Z. K. Liu*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Univ. Park, PA 16803 USA

Due to their high specific strength, magnesium alloys have become increasingly attractive for automotive structural applications. In recent years it has been shown that Zr is a very effective grain refiner of Mg-Zn alloys. The grain refining properties of Zr are due to the presence of both undissolved Zr particles as well as Zr-containing intermetallics. Therefore, an accurate knowledge of the phase state of the system at any composition and temperature is required. In this work, we present a thermodynamic model of the Mg-Zn-Zr system by combining ab initio calculations and the CALPHAD method. Using this model, calculations on both stable and metastable isothermal sections as well as liquidus surfaces and equilibrium solidification are performed and related to experimental observations. The possible formation of metastable intermetallic phases during solidification is also discussed. Solubility of Zr in Mg-Zn-Zr alloys as well as phase fractions of intermetallic compounds are systematically studied as these parameters greatly affect grain refining.

10:10 AM Break

10:25 AM
Influence of Strong Static Magnetic Field on the Solution and Aging Behavior in AZ91 Magnesium Alloys: *Zhifeng Li*[1]; J. D.[1]; X. Q. Zeng[1]; Y. X. Wang[1]; C. Lu[1]; [1]Shanghai Jiao Tong University, Natl. Engrg. Rsch. Ctr. of Light Metal Forming, Shanghai 200030 China

A strong static magnetic field (SSMF) of about 10 T was applied when AZ91 magnesium alloy was undertaken solution and aging heat treatment for different time. Microstructures of conventional solutioned specimens and solutioned specimens under SSMF were investigated by Optical and SEM micrographs which indicated that the solution process of $Mg_{17}Al_{12}$ in the alloy was retarded under the SSMF condition. The microstructures of both conventionally and SSMF aged specimens at 453K were observed by OM and TEM. The results showed that $Mg_{17}Al_{12}$ discontinuous precipitates at grain boundary were promoted in the SSMF condition which resulted in a quick age hardening response in the first stage of aging. The acceleration of discontinuous precipitation may be attributed to the relatively lower body diffusion rate in the matrix under the SSMF condition which resulted in a higher chemical free energy driving force for the nucleation and growth of the discontinuous precipitate.

10:45 AM
Thermodynamics and Constitution of Ca-Zr and Mg-Al-Ca-Zr Alloys: Andreas Janz[1]; Djordje Mirkovic[1]; Joachim Gröbner[1]; *Rainer Schmid-Fetzer*[1]; [1]University of Clausthal, Inst. of Metall., Robert-Koch-Str. 42, Clausthal-Zellerfeld D-38678 Germany

This is a first report in our series of ongoing studies of quaternary Mg-Al-Ca-X systems. The addition of X = Zr is of interest in order to understand the limitations imposed by the alloy phase equilibria on the use of Zr for example as grain refiner. The practical problems in using Zr together with Al are ascribed to the formation of stable Al-Zr compounds. Our thermodynamic calculations reveal that at, say, 800°C in Mg-Al-Zr alloys up to 0.1 wt.% Al and 0.1 wt.% Zr the compounds AlZr, Al_3Zr_4, and Al_2Zr_3 may be formed. We studied the impact of Ca on these equilibria and also the impact of Zr on a wider range of Mg-Al-Ca alloys. One obstacle is that the Ca-Zr binary system is not known. Experimental work and thermodynamic modeling of the phase equilibria in this binary and the higher order alloys are performed. Selected phase diagram sections of the quaternary Mg-Al-Ca-Zr system are shown.

11:05 AM
Precipitation Hardening in Mg-Zn-Sn Alloys: *S. Cohen*[1]; G. R. Goren-Muginstein[1]; S. Avraham[1]; M. Bamberger[1]; R. Rashkova[2]; G. Dehm[2]; [1]Technion-Israel Institute of Technology, Technion City, 32000 Haifa Israel ; [2]Max-Planck-Institut für Metallforschung, Heisenbergstr. 3, 70569 Stuttgart Germany

New Al-free Mg-Zn alloys with alternative alloying elements are currently developed in order to overcome the poor creep behaviour of commercially available Mg-Alloys and to improve their corrosion resistance and castability at elevated temperatures. Based on thermodynamic calculations Zn and Sn additives are expected to form stable intermetallic phases with Mg and cause precipitation hardening. In the present work we studied a Mg alloy containing 4.46 wt.% Zn and 3.75 wt.% Sn. Precipitation hardening mechanisms were investigated in the temperature range of 175-200°C by Vickers hardness measurements and analysis of the microstructured evolution using XRD, SEM, and TEM. During aging at 175°C for 1 to 96 hours, two hardness maxima occurred: the first after 2 hours, where the hardness increased from 55 to 88 HV, followed by a rapid decrease to 73 HV and then the second peak at 87 HV after 16 hours. The hardness decreased after 48 hours to 70 HV and remained constant up to 96 hours. Corresponding XRD spectra and TEM analyse of the sample indicate that the occurrence of two hardness peaks can be related to a precipitation sequences. Firstly, MgZn2 precipitations form, and secondly, Mg2Sn particles precipi-

tate. The precipitates are uniformly distributed in the Mg-matrix with two morphologies: needle- and plate–like shapes. The study was partially supported by the German Israeli foundation for scientific research and development (GIF) under contract number I-704-43.10/2001. The foundation is acknowledged for its support.

Magnesium Technology 2005: Wrought Magnesium Alloys III

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Tuesday AM Room: 2006
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Robert E. Brown, Magnesium Monthly Review, Prattville, AL 36067-3806 USA; Karl Ulrich Kainer, GKSS Research Center, Ctr. for Mg Tech., Geesthacht D-21502 Germany

8:30 AM
Strain Path and Temperature Effects on Texture and Microstructure Evolution of AZ31: *Chris Huw John Davies*[1]; Fei Xiong[1]; [1]CRC for Cast Metals Manufacturing, Sch. of Physics & Matls. Engrg., Bldg. 26, Monash Univ., VIC 3800 Australia

The effect of strain path and temperature on texture and microstructure evolution in AZ31 was investigated by plane strain (channel die) compression, uniaxial tension, and uniaxial compression. Both as-cast and extruded structures were used as starting textures, and different strain paths were imposed upon the samples, including complete reversal, and 90° rotation in plane strain compression. After each step in the strain path, texture and microstructure were examined. The effect of thermal path has been examined in a similar manner. In this way, a picture of the evolution of texture and microstructure was compiled. Results will be presented showing the effect of strain path on flow stress, and of thermal path on microstructure, and these are interpreted in terms of texture evolution and the propensity for twinning. Simple models are used to show the types of twinning active during deformation.

8:50 AM
Prediction and Measurement of Residual Strains in a DC Cast AZ31 Magnesium Billet: Hai Hao[1]; *Daan M. Maijer*[1]; Mary A. Wells[1]; Steve L. Cockcroft[1]; Ron Rogge[2]; Steve G. Hibbins[3]; [1]University of British Columbia, Matls. Engrg., 309 - 6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]National Research Council of Canada, Steacie Inst. for Molecular Scis., Chalk River, ON K0J 1J0 Canada; [3]Timminco Metals Ltd., Haley, ON K0J 1Y0 Canada

As part of a research programme to improve casting efficiency, a thermo-mechanical finite element (FE) model of DC cast AZ31 magnesium billets has been developed to predict defect formation such as hot tearing and cold cracking. The model simulates the evolution of temperatures, stresses and strains inside the billet. Thermal boundary conditions have been selected based on knowledge of the physical process and through comparison with measured temperature data. The temperature predictions show good agreement with the measurements for both transient and steady state conditions. The constitutive behaviour of AZ31, a critical input to the thermal-stress model, was measured using a Gleeble 3500 thermo-mechanical simulator over a wide range of temperatures. In order to validate the predicted strain, residual strain measurements were carried out using neutron diffraction through the National Research Council of Canada's Neutron Program for Materials Research. Strain measurements in the radial, axial and hoop orientations were made at a variety of axial and radial locations in a billet. The measured strains were compared with the predicted results and used to validate the thermo-mechanical model.

9:10 AM
Evaluation of the Surface Heat Flux in the Secondary Cooling Zone During the Direct-Chill Casting: *Etienne J.F. R. Caron*[1]; Mary A. Wells[1]; Dimitry Sediako[2]; Steve G. Hibbins[2]; [1]University of British Columbia, Dept. of Matls. Engrg., 309 - 6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Timminco Metals, Tech. Dvlp. & Metall., Haley, ON K0J 1Y0 Canada

Accurate knowledge of the boundary conditions is essential when modelling the Direct-Chill (DC) casting process. Determining the surface heat flux in the secondary cooling zone, where the greater part of the heat removal takes place, is therefore of critical importance, particularly for the process start-up phase. Industrially DC cast AZ31 magnesium alloy samples were instrumented with thermocouples, preheated in an electrical furnace and sprayed with water jets to simulate the secondary cooling zone of the DC casting process. The surface heat flux in both the water jet impingement zone and the water film free-falling zone was then evaluated using the measured thermal history data in conjunction with a two-dimensional inverse heat conduction (IHC) model developed at the University of British Columbia. The effects of various parameters (water flow rate, impingement angle, surface morphology) on the rate of heat removal by the water jets was investigated.

9:30 AM
A Mathematical Model of Heat Transfer and Fluid Flow in the Direct Chill Casting of AZ31 Magnesium Billets: *E. H. Lu*[1]; D. M. Maijer[1]; D. Sediako[2]; [1]University of British Columbia, Dept. of Matls. Engrg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Timminco Metals, R&D, Haley, Ontario K0J 1Y0 Canada

A 2-D axisymmetric mathematical model of heat transfer and fluid flow during direct chill (DC) casting of AZ31 magnesium billets was developed using the finite volume modeling package FLUENT. Model boundary conditions were based on those used in a 2-D axisymmetric finite element model previously developed in ABAQUS, which approximates the effects of fluid flow by manipulating material properties. Materials properties were based on literature and knowledge of the physical phenomena occurring during the DC casting process. Temperature predictions from both models are compared with plant trial temperature data acquired from billets produced with different casting conditions at Timminco Metals in Haley, Ontario. Following this initial comparison, the high temperature material properties and boundary conditions of the FLUENT model were altered to fit the measured temperatures. The significance of fluid flow on the apparent material properties of AZ31 and the thermal boundary conditions is presented.

9:50 AM
Process and Alloy Development for Hydrostatic Extrusion of Magnesium: The European Community Research Project MAGNEXTRUSCO: *Jan Bohlen*[1]; Wim H. Sillekens[2]; Piet-Jan Vet[3]; Dietmar Letzig[1]; Karl Ulrich Kainer[1]; [1]GKSS Research Centre, Ctr. for Mg Tech., Max-Planck Str. 1, Geesthacht 21502 Germany; [2]TNO Industrial Technology, De Rondom 1, Postbus 6235, Eindhoven 5600 HE The Netherlands; [3]Outokumpu Copper HME b.v., Veerweg 14, Waalwijk 5145 NS The Netherlands

While magnesium cast products have been established in industrial lightweight applications, the development is now focussing on semi-finished wrought products. This leads to a broader variety of shapes as well as to improved material properties of magnesium alloys. In particular, extrusion as a shaping technology offers the possibility to produce such products. While the technology for processing magnesium alloys is available today, the industrial use is not well established because of technical and economical limitations of the direct and indirect extrusion process. The European Commission research project MAGNEXTRUSCO has been conducted as a GROWTH project within the 5th Framework programme in order to overcome these limitations in the production of magnesium wrought components. A consortium of 9 European partners from industry and research institutions focused on the use of the hydrostatic extrusion process for the production of magnesium structural components. The advantage of using this process is to increase the extrusion speed and to lower the extrusion temperature. Alloy development, process research, post-processing techniques and the development of magnesium demonstrators by end users are the main topics of the work programme along the complete process chain. This paper gives an overview on the project's outline regarding such issues as motivation, objectives and partnership. Results with respect to micro-structural evolution and mechanical properties due to the settings of the extrusion parameters will be presented and discussed.

10:10 AM Break

10:25 AM
An Efficient Route to Magnesium Alloy Sheet: Twin Roll Casting and Hot Rolling: *Lothar Loechte*[1]; Hakon Westengen[2]; John Rodseth[3]; [1]Hydro Aluminium, R&D Ctr., Georg-von-Boeselagerstr. 21, Bonn 53117 Germany; [2]Hydro Magnesium Competence Center, R&D Ctr., Porsgrunn 3908 Norway; [3]Hydro Aluminium, R&D Karmoy, Havik 4265 Norway

In the last decade a significant market pull, especially from the automotive industry, has been observed for magnesium alloy sheet. Two main production routes for this material are currently under world-

wide discussion; specifically, the classical route via DC casting plus hot rolling is competing with continuous casting techniques. A particularly flexible route for producing magnesium alloy sheet is provided by twin roll casting, followed by a hot rolling plus annealing sequence to the final gauge and temper. Early results and evaluations from trials with semi-scale (up to 700mm width) twin roll cast and hot rolled AZ31B are presented. Microstructures of as cast strip, as well as intermediate gauges and final thickness sheet, are discussed and related to production parameters such as casting speed and hot rolling process schemes. The promising mechanical properties which have been achieved are compared to those generated by a more conventional route and are discussed in terms of microstructural features, e.g., grain size and texture.

10:45 AM
Control of Wall Thickness Distribution in Magnesium Tube Gas Forming With Large Diameter Expansion: *Ramnath Krishnamurthy*[1]; Wuhua Yang[2]; Xin Wu[1]; Micheal L. Wenner[2]; [1]Wayne State University, Mechl. Engrg., 5050 Anthony Wayne Dr., Detroit, MI 48202 USA; [2]General Motor Corporation, Mfg. Sys. Rsch. Labs., Warren, MI 48090 USA

For developing lightweight structures for automotive applications, the feasibility of forming magnesium AZ31B tubes using Hot Metal Gas Forming process was conducted previously, and the results indicate that tube expansion with very large strain (above 100%) can be achieved. To take full advantages of the material formability enhancement and the new forming technique, the thinning of the tube wall thickness must be compensated, which is very challenging with very large deformation and with sensitive temperature dependence of the material. In this study, the understanding of metal flow and the strategy for thickness compensation are focused. The thickness control is achieved through combined control of temperature distribution, gas pressure and tube end-feeding velocity. Both experimental and analytical results are reported, and a comparison between them is provided. A concept of regional heating/forming and sequential multi-stage operation is proposed that allows local deformation control.

11:05 AM
An EBSD Study on Microstructural Evolution During Superplastic Deformation of a Fine-Grained AZ31 Magnesium Alloy: Yi Liu[1]; *Xin Wu*[1]; [1]Wayne State University, Mechl. Engrg., Detroit, MI 48202 USA

Superplastic deformation has been conducted on AZ31 magnesium alloy with initial fine grains (<10 micron) at 623-773K with the strain rate of 10^-3 to 1s^-1. A maximum elongation of over 500% was obtained at 773K with 10^-3 s^-1. Dynamic grain growth was found, that was temperature dependent. EBSD analysis indicated that, during deformation, the pre-existing strong (0001) fiber texture was not destroyed during deformation, although grain boundary sliding was observed. Together with optical microscope and SEM observations, the deformation mechanisms are discussed.

11:25 AM
Effects on Microstructures and Fracture Morphology of SiCp/AZ91D Composites by Equal-Channel Angular Pressing: *Yan Yin Biao*[1]; [1]Nanjing University of Science & Technology, Matls. Sci. & Engrg., 200 Xiaolingwei St., Nanjing, Jiangsu 210094 China

The equal channel angular pressing (ECAP) were performed for SiCp reinforced AZ91D based composites prepared by stirring-casting process. The structures of AZ91D based composites and fracture morphology were observed and analyzed by optical microscope and scanning electron microscope after ECAP. The results show that the casting defects such as pores were eliminated, the size of grains was be reduced and the uniformity of particle was improved after ECAP. The connection of matrix and particles, the harm accommodation to SiCp also were improved; SiCp masses were the main cause of fracture of the composites with tenacious fracture of the matrix alloy companied with some disconnection between particles and matrix and the cross-grain fracture exists. After ECAP, principal part was tenacious fracture of the matrix with little quantitative cross-grain fracture in the structures and disconnection between particles and matrix was the main form in the area of dense distribution of SiCp.

11:45 AM
High Strength Mg-Zn-Y Alloys with Long Period Stacking Structure: *Yoshihito Kawamura*[1]; Shintaro Yoshimoto[2]; [1]Kumamoto University, Matls. Sci. & Tech., Kurokami 2-39-1, Kumamoto 860-8555 Japan; [2]Kumamoto University, Grad. Sch. of Sci. & Tech., Kurokami 2-39-1, Kumamoto 860-8555 Japan

Mg-Zn-Y alloys have a long period stacking ordered (LPSO) structure of 18R. We have previously reported the Mg97Zn1Y2 rapidly solidified powder metallurgy (RS P/M) alloy exhibited high yield strength (0.2% proof stress) of 610 MPa, elongation of 5%, a high-strain-rate superplasticity and high corrosion resistance. In this study, the effect of RS P/M processing on mechanical properties of LPSO-type Mg-Zn-Y alloys was investigated by comparing with ingot metallurgy (I/M) alloys that were produced by extruding cast ingot. The tensile yield strength of the I/M Mg97Zn1Y2 alloy was 375 MPa and 278 MPa at ambient temperature and 473 K, respectively. It was found that the RS P/M processing improved the tensile yield strength by 63% at ambient temperature and by 40% at 473 K in comparison with the I/M processing.

Materials Processing Fundamentals: Smelting & Refining I

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control Committee
Program Organizers: Princewill N. Anyalebechi, Grand Valley State University, Padnos School of Engineering, Grand Rapids, MI 49504-6495 USA; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Tuesday AM Room: 3001
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Prince N. Anyalebechi, Grand Valley State University, Padnos Sch. of Engrg., Grand Rapids, MI 49504-6495 USA

8:30 AM
Phase Field Model of Electrochemistry for the Ti-Mg-Cl Ternary System: *Wanida Pongsaksawad*[1]; Adam C. Powell[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 4-043, Cambridge, MA 02139 USA

The mechanism of titanium product formation is investigated in the small-scale Electronically Mediated Reaction process (EMR). A Cahn-Hilliard model of electrochemistry for the Ti-Mg-Cl ternary system with composition-dependent mobility is applied to study morphology evolution of titanium sponge. The formulation used in this model assumes rapid charge redistribution in the electrolyte and transport-limited electrochemical reactions. Two-dimensional simulation results of a solid-liquid system with fluid flow are presented as a function of applied voltage, surface energy and viscosity, and as expected, the cathode interface is less stable under high electric field, low surface energy, and high viscosity. In addition, three dimensional results are presented illustrate the sponge formation relative to the two-dimensional case.

8:55 AM Cancelled
Mathematical Modeling of the Fray Farthing Chen (Ffc) Process for Titanium Extraction

9:20 AM
Dissolution of Copper Oxides in Molten Na2O- and CaO-Based Slags: *Peng Fan*[1]; Weol D. Cho[1]; [1]University of Utah, Dept. of Metallurgl. Engrg., Salt Lake City, UT 84112 USA

The dissolution rate and the solubility of copper oxides (CuO and Cu2O) in various Na2O-B2O3-SiO2 and CaO-B2O3-SiO2 slags have been studied at high temperatures. The effects of temperature and slag composition on the dissolution rate and the solubility have been determined for the two copper oxides. Based on the physical and chemical properties of the molten slags and the interaction between solid copper oxides and the molten fluxes, the mechanism of the dissolution is discussed.

9:45 AM
Main Parameters on the Performance of a Desulfurization of Ferro-Nickel by Slag Reaction: *Markus Hochenhofer*[1]; Helmut Antrekowitsch[1]; Matjaz Juhart[3]; [1]Christian-Doppler-Laboratory for Secondary Metallurgy of the Non-ferrous Metals, Franz-Josef-Strasse 18, Leoben 8700 Austria; [3]Treibacher Industrie AG, Althofen 9330 Austria

To produce ferroalloys like ferro-nickel for alloying purposes in a quality that meets the requirements of today's steel industry, refining processes like desulfurization and dephosphorization are indispensable. The performance of a desulfurization-treatment by slag reaction depends on various parameters, such as: sulfide capacity and viscosity of the slag, sulfur distribution between metal and slag, activity of sulfur

in the melt and movement of the melt. Within the framework of this work, the combined influences of these parameters on the desulfurization of a ferro-nickel alloy were examined. As a basis for this examination, a mathematical model, based on data from plant trials, was created. This model demonstrates the dependence of the desulfurization rate on the initial values of the above mentioned parameters. With this model the relevant parameters were evaluated and assessed concerning their influence on desulfurization.

10:05 AM Break

10:20 AM
Electro-Deoxidation of Solid Chromium Oxide in Molten Chloride Salts: *George Zheng Chen*[1]; Derek J. Fray[2]; Elena Gordo[3]; [1]University of Nottingham, Sch. of Chem. Environ. & Mining Engrg., Univ. Park, Nottingham NG7 2RD UK; [2]University of Cambridge, Dept. of Matl. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK; [3]University of Carlos III de Madrid, Dept. of Matl. Sci. & Metall. Engrg., Avda. de la Universidad 30, 28911 Madrid Spain

Chromium is industrially produced by aluminothermic reduction of Cr2O3 or electro-deposition from the aqueous NH4Cr(SO4)2 solution. The former makes more expensive products than what would be from an electrolytic method because aluminium is produced by electrolysis. In the aqueous electrolysis, the redox recycling of the multivalent chromium species between electrodes leads to low current efficiency (~45%) and high energy consumption (~18.5 kWh/kgCr). Recent reports have demonstrated that solid metal oxides can be directly electro-deoxidised to the respective metals/alloys in molten salts. For electrolysing solid Cr2O3, less than 0.2wt% oxygen could be achieved in the powdery product with the current efficiency and energy consumption being 75% and 5 kWh/kgCr, respectively. In this paper, previous work is reviewed in detail, followed by in-depth discussion on the mechanisms of (1) the electro-deoxidation process and (2) the formation of the cubic and nodular morphologies of the chromium powders obtained under different electrolysis conditions.

10:40 AM
Simulation of Reactive Pellets in a Pyrometallurgical Bath Using SPH: *Paul W. Cleary*[1]; Nick Stokes[1]; Joseph Ha[1]; Mahesh Prakash[1]; Geoff A. Brooks[2]; [1]CSIRO, Math. & Info. Scis., PB 10, Clayton S., Vic 3169 Australia; [2]CSIRO, Minls., Normanby Rd., Clayton S., Vic 3169 Australia

In several pyrometallurgical processes, reactive pellets are added to a melt. Heat is transferred to the pellets and reaction products such as gases, metal and slag are formed. For many pyrometallurgical processes traditional grid based CFD methods, such as using the Finite Element Method (FEM) and Finite Volume (or Control Volume methods), produce suitable predictions. However, the inclusion of solids in the bath represents significant challenges for these methods. Smoothed Particle Hydrodynamics (SPH), a Lagrangian simulation method, is able to simulate both the fluid component of the bath and any immersed solid materials, tracking the motion of these solids, and their interaction with the fluid, including gas generation. In this paper, we will summarize the key aspects of the SPH method and will show a series of structured examples illustrating the behaviour of the different physics sub-systems that are used in the model of the overall system.

11:00 AM
An Experimental Study on Floating Solids in a Liquid Bath: Mohamed Nabil Noui-Mehidi[1]; Hugh Blackburn[1]; Richard Manasseh[1]; *Geoff A. Brooks*[2]; Murray Rudman[1]; [1]CSIRO, Mfg. & Infrastruct. Tech., PO Box 56, Graham Rd., Highett, Melbourne, Victoria 3190 Australia; [2]CSIRO, Minls., PO Box 312, Clayton S., Melbourne, Victoria 3169 Australia

In some pyrometallurgical applications pelletised solids react in a bath of liquid metal at high temperatures. A key factor controlling the rate at which the reaction takes place is the extent and nature of the submergence process of the solids in the liquid bath. This study is concerned with an experimental investigation of the minimum speed and the associated power consumption to achieve the submergence of floating solids in a stirred vessel. The physical modeling was achieved by using water as liquid medium and pine wood pellets as buoyant particles. Particular attention was given to the effects of pellet size and pellets load on submergence. Power consumption was found to increased when larger pellets were used. The tests performed on the study of the load effect, have shown an increase in the power consumption when the total load increased; still higher power consumption was required to submerge larger pellet sizes.

11:20 AM
Reduction of Chromium Oxide from Liquid Slags: *Antonio Romero-Serrano*[1]; Juliana Gutierrez[1]; Victor Arredondo[1]; José Manuel Hallen[1]; [1]National Polytechnic Institute, Metall. & Matls. Dept., ESIQIE-IPN, Apdo. Postal 118-431, Mexico, D.F. 07051 Mexico

Experimental and theoretical analysis were carried out in this work to estimate the effect of slag basicity and amount of reducing agents on the reduction of chromium oxide from the slag which interacted with molten steel at 1600°C. The slag system contained CaO, MgO, SiO2, CaF2 and Cr2O3 together with Fe-alloys (Fe-Si or Fe-Si-Mg). Some experiments were also conducted to study the effect of initial Cr content in steel on the chromium oxide reduction. Three initial Cr contents were tested (0.15, 2 and 4 mass%). Argon was injected at the bottom of the furnace in these last experiments to increase the stirring of the system. Some estimations were made to determine the theoretical effect of temperature, slag basicity, (CaO+MgO)/SiO2, and amount of reducing agents in the slag on the chromium recovery. The FACT (Facility for the Analysis of Chemical Thermodynamics) computational package was used to determine the equilibrium between the slag and the molten steel.

11:40 AM
Studying Solvent Extraction Settler Process by Using CFD: *Timo Tapani Kankaanpää*[1]; [1]Helsinki University of Technology, Lab. of Matls. Procg. & Powder Metall., PO Box 6200, Espoo FIN-02015 HUT Finland

The liquid-liquid dispersion phases are separated by gravity in a solvent extraction settler. This separation step is made more effective by picket fences. The picket fence geometry and its physical placement have a significant effect on the separation process, because the purpose of a fence is to smoothen and control organic-aqueous dispersion flow in the first part of the settler and achieve a deep and dense dispersion layer in the front end of the settler resulting in a clean phase separation. In this study, physical phenomena in the solvent extraction process and phase separation in a solvent extraction settler with and without picket fences have been examined using a commercial CFX software package. It can be concluded that the used CFD-approach proved to be a complementary tool for optimizing and designing the solvent extraction settler.

12:00 PM
Mathematical Modeling of Molten Steel Flow in the Vacuum Circulation Refining Process: *Ji He Wei*[1]; Han Tao Hu[1]; Hui Fa Huang[1]; [1]Shanghai University, Dept. of Metallic Matls., 149 Yan Chang Rd., Shanghai 200072 China

A three-dimensional model for the flow of the molten steel in the whole unit during the RH refining process has been proposed and developed with considering the physical characteristics of the process, particularly the behaviors of gas-liquid two phase flow in the up-snorkel. The flow field of liquid, the gas holdup of the liquid phase in the up-snorkel and the circulation flow rate in a water model unit with an 1/5 linear scale of a 90 t RH degasser have been computed using this model. The results showed that the flow pattern in the whole RH unit could be well modeled by the model. The liquid can be fully mixed during the refining process except the area close to the free surface of liquid in the ladle and the zone between the two snorkels, but there is a boundary layer between the descending liquid stream from the down-snorkel and its surrounding liquid, which is a typical liquid-liquid two phase flow, and the molten steel in the ladle is not in a perfact mixing state. The lifting gas blown is rising mos tly near the up-snorkel wall, the flow pattern of the bubbles and liquid in the up-snorkel is closer to an annular flow. The calculated circulation flow rates are in good agreement with the measured values.

Mechanical Behavior of Thin Films and Small Structures: Stability, Strain and Stress

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior

Program Organizers: Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA; Brad L. Boyce, Sandia National Laboratories, Materials and Processes Sciences Center, Albuquerque, NM 87185 USA; Evan Ma, Johns Hopkins University, Department of Materials Science & Engineering, Baltimore, MD 21218 USA; Andrew Minor, Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Berkeley, CA 94720 USA; Christopher L. Muhlstein, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Judy A. Schneider, Mississippi State University, Department of Mechanical Engineering, Mississippi State, MS 39762 USA

Tuesday AM Room: 2024
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Judy Schneider, Mississippi State University, Dept. of Mechl. Engrg., Mississippi State, MS 39762 USA; Carl V. Thompson, Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139 USA

8:30 AM Invited
Stress Evolution During Deposition of Polycrystalline and Epitaxial Films of Cu and Ag: *Carl V. Thompson*[1]; Cody A. Friesen[2]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Cambridge, MA 02139 USA; [2]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., Currently at Arizona State University, Cambridge, MA 02139 USA

Polycrystalline films can develop very large tensile or compressive stresses during deposition. We have characterized stress evolution during evaporative deposition and during interruptions of evaporative deposition of both polycrystalline and epitaxial thin films of Cu and Ag. In all cases, a reversible stress change is observed when growth is interrupted and resumed. We have correlated this stress change with changes in the surface defect structure, through both modeling and experimental characterization with reflected high energy electron diffraction. These results demonstrate that kinetic roughening of film surfaces during deposition leads to a compressive surface stress, and may also be related to mechanisms that result in compressive residual stresses. The compressive stress associated with kinetic roughening is superimposed on mechanisms that lead to residual tensile stresses, such as the development of coalescence strains in polycrystalline films and misfit strains in epitaxial films.

8:55 AM
The Effect of Linewidth Scaling on the Stress State of Passivated Interconnects: Application to Stress Voiding: *Raju V. Ramanujan*[1]; D. Ang[1]; C. Wong[1]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Blk. N4.1, Nanyang Ave., Singapore 639798 Singapore

Critical dimensions of interconnects are in the nanometer range, such small dimensions can lead to stress induced diffusive voiding (SIDV) failure. Experimentally, a complex dependence of mean time to failure on linewidth dimensions is observed. To rationalize these experimental results and to correctly extrapolate reliability results to finer interconnects, the effect of interconnect dimensions on SIDV was studied. This scaling effect was examined by numerical analysis and the Eshelby model; the effect of linewidth scaling on the hydrostatic stress in passivated metal lines was determined. An increasing trend of hydrostatic stress with linewidth for narrow lines, and decreasing trend of hydrostatic stress with linewidth for wide lines, with a maximum hydrostatic stress at a critical linewidth was observed. The hydrostatic stress in copper lines for all linewidths is larger than that in aluminium lines. The effect of grain boundaries on SIDV was also examined using this model.

9:10 AM
Atomistic Simulation of Stress Evolution in the Early Stages of Volmer-Weber Growth: *Chun-Wei Pao*[1]; Mikhail I. Mendelev[1]; David J. Srolovitz[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Engrg., Olden St., Princeton, NJ 08544 USA

Wafer curvature experiments show that during Volmer-Weber growth, the stresses in the islands are compressive; the magnitude of which increases as the islands grow. The origin of these stresses is speculative. We performed a series of hybrid static-relaxation/molecular dynamics simulations of the growth of islands on an amorphous substrate that confirm that compressive stresses form and increase as the islands grow. To identify the origin of this effect, we perform a series of simulations as a function of island/substrate bond strength. Stronger interfacial bonding leads to smaller wetting angle and larger compressive stresses. Weaker bonding leads to slipping at the island/substrate interface and islands that are nearly stress-free. These observations are consistent with experimental results in which stronger interfacial bonding leads to larger compressive stresses in the islands and smaller film thicknesses at which the ubiquitous development of tensile stresses during film growth occurs.

9:25 AM Invited
Adatom Diffusion on Strained FCC (001) Surfaces: Wei Xiao[1]; P. Alex Greaney[1]; *D. C. Chrzan*[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., Berkeley, CA 94720-1760 USA

The growth of thin films often takes place in the presence of significant strains. These strains influence both the thermodynamics and kinetics of nucleation. Specifically, these strains may alter substantially the energy barriers associated with adatom diffusion. Further, under certain circumstances, surface strain can give rise to new types of adatom diffusion mechanisms. This talk considers the specific example of Cu adatom diffusion on strained Cu (001). Embedded atom method calculations employing the nudged-elastic-band method, are used to study adatom diffusion as a function of surface strain. These studies reveal interesting trends in adatom diffusion energy barriers, as well as the appearance of a surface crowdion mediated adatom transport mechanism. The structure and a rudimentary picture of the dynamics of these surface crowdions are presented. This work is supported by the National Science Foundation.

9:50 AM
Phase Transformation and Reorientation in Gold Nanowires: *Ken Gall*[1]; Jiankuai Diao[1]; Martin L. Dunn[1]; [1]University of Colorado, Dept. of Mechl. Engrg., Boulder, CO 80309 USA

Atomistic simulations with modified embedded atom method (MEAM), embedded atom method (EAM) and surface embedded atom method (SEAM) potentials reveal that, at certain sizes, a face centered cubic (fcc) gold <100> nanowire reorients into an fcc <110> nanowire. In MEAM simulations, the reorientation consists of two successive processes. First, surface stress and thermal vibrations cause the fcc <100> nanowire to transform into a body centered tetragonal (bct) nanowire. Second, the bct nanowire becomes unstable with respect to shear and transforms into an fcc <110> nanowire. In EAM and SEAM simulations a different reorientation mechanism exists. The surface stress in the fcc <100> nanowire induces slip on a {111}<112> system. Progressive slip on adjacent {111} planes changes the stacking sequence of these {111} planes from ABCABC to ACBACB, and the nanowire reorients into an fcc <110> nanowire. The difference in reorientation mechanism is rooted in the differences in the unstable stacking fault energy and orientation dependence of electron density in the potentials. In spite of these differences, the final structures of the reoriented nanowires are the same, which helps to explain why uniform fcc <110> nanowires are observed much more often in experiments than nanowires of other orientations. Results of preliminary first principal calculations are presented in an effort to better understand the semi-empirical results.

10:05 AM Break

10:20 AM Invited
Strain-Induced Coarsening in Nanograined Films: *John William Morris*[1]; Miao Jin[1]; Andrew M. Minor[2]; [1]University of California, Matls. Sci. & Engrg., 210 Hearst Mining Bldg., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, 1 Cyclotron Rd., Berkeley, CA 94720 USA

In-situ nanoindentation within a transmission electron microscope has been used to study the deformation mechanisms in ultrafine-grained Al films. An initially surprising result of these studies was the frequent observation of spontaneous grain growth during indentation, which appears to be triggered by the deformation. This deformation-induced coarsening is clearly observed in micrograined Al films. In situ studies of nanograined films suggest that the same mechanisms are operative, though the difficulty of imaging nanosized grains makes the evidence less clear. The coarsening is promoted by two driving forces: decreasing the surface energy and increasing plastic work. The apparent mechanism is associated with the role of deformation in enhancing grain boundary mobility. The results suggest that grain growth and coalescence are important modes of response in the deformation of ultrafine- and nanograined materials.

10:45 AM
Thermomechanics of Thin Film Au for Micro/Nano Scale Engineering: *Ken Gall*[1]; David Miller[1]; Nancy Yang[1]; Cari Herrmann[1]; Hans Maier[1]; Steve George[1]; Conrad Stoldt[1]; [1]University of Colorado, Dept. of Mechl. Engrg., Boulder, CO 80309 USA

Gold is a promising material in emerging micro and nano systems owing to the ease of fabricating one- and two- dimensional Au nanostructures, its biocompatibility and capacity for biofunctionalization, and its favorable electrical properties. However, the thermomechancial properties of Au in various small-scale forms are not well characterized or understood, presenting a potential roadblock to the use of Au in devices. We have examined the thermomechanical behavior and microstructural evolution of gold thin films adhered to silicon microcantilevers with a chrome barrier layer. Such microcantilevers have application in optics, electronics (DC and RF), chemical and biological sensors, actuators, and fabrication process monitoring. Arrays of Au/Cr/Si microcantilever specimens subject to isothermal hold conditions exhibited dimensional instability that is dynamic in nature. We have characterized the thermomechanical response of our gold thin film based structures using curvature-temperature-time experiments in the temperature range from 50 to 225ºC. Drastic changes in curvature were observed for specimens annealed in air at 225ºC for less than 24 hours. The changes observed were significant and are certain to influence design performance as well as device reliability. Cross-sectional TEM evidenced an initial microstructure containing twins and dislocations that evolved during annealing. The free surface of the gold layer was observed to increase in roughness. FE-SEM imaging revealed extreme grain boundary grooving and grain growth. The observed changes in microstructure and morphology may be influenced by the diffusion and oxidation of the chrome layer. EDX analysis of the TEM foil specimens qualitatively suggests diffusion of the Cr barrier layer to the Au surface. Furthermore, it was found that the use of nanometer thick Atomic Layer Deposition (ALD) grown alumina coatings can greatly influence the evolution of the gold mcirostructure. Differences in mechanical and structural behavior were observed when surface coatings were applied. In particular, alumina coatings were observed to limit surface evolution, mitigate the migration of chrome, and suppress changes in curvature.

11:00 AM
Mechanical Property Enhancement of Silicon Component with Laser Shock Peening: *Gary J. Cheng*[1]; D. Pirzada[1]; Dave Bahr[1]; [1]Washington State University, Sch. of Mechl. & Matls. Engrg., Pullman, WA 99163-2920 USA

This paper will investigate a fundamental and general problem about processing of brittle material: How can we generate plastic deformation in silicon crystal to favorable stress/strain distribution for mechanical property enhancement? Silicon-based micro-components are dominant structural materials for micro-machines. However, silicon has the disadvantage of brittle material behavior. Fracture toughness of silicon is several orders lower than that of metals. Silicon thin film often fails under cyclic loading conditions even in ambient air and at room temperature. These result in serious problems when silicon-based materials are subjected to aggressive mechanical, thermal and chemical environments. However, it has been shown that silicon displays “metal like” stress-strain relationship, and high dislocation mobility at elevated temperature. This work will explore the silicon’s plastic behavior by laser shock peening and its benefit to mechanical property enhancement. Laser shock peening (LSP) experiments and thermomechanical simulation will be conducted. Mechanical properties (fatigue life and fracture toughness) will be predicted and compared to experiments. Nanoindetation testing will be used to investigate the changes in mechanical property. TEM will be employed to investigate the dislocation structure after LSP. X-ray diffraction will be used to measure the residual stress.

11:15 AM Invited
Thermo-Mechanical Stability of Metallic Nanolaminates: *Amit Misra*[1]; Richard G. Hoagland[1]; [1]Los Alamos National Laboratory, MST Div., MS G755, Los Alamos, NM 87545 USA

Sputter deposited metallic nanolaminates exhibit unusually high hardness when the bilayer periods approach nanometer dimensions. In this presentation, we report on the thermal and mechanical stability of sputter deposited Cu-Nb nanolaminate foils. The nanolaminates exhibit extraordinary plastic stability upon room temperature rolling undergoing uniform reduction in layer thickness to high levels of plastic strain. Large reduction in thickness is accomplished without the formation of the classical dislocation cell structures. Furthermore, no out-of-plane lattice rotations are observed. These results are interpreted in terms of symmetric slip occurring by the glide of single dislocations on multiple slip systems in both layers. The interface stability under mechanical straining is compared to the morphological stability of these nanolaminates following elevated temperature annealing. The effect of nanolayering length scale on the thermal and mechanical stability is also discussed. This research is funded by DOE, Office of Science, Office of Basic Energy Sciences.

11:40 AM
High Temperature Mechanical Properties of Cu/Nb Nanoscale Multilayers at Diminishing Length Scales: *Nathan A. Mara*[1]; Alla V. Sergueeva[1]; Tammy Tamayo[1]; Xinghang Zhang[2]; Amit Misra[3]; Amiya Mukherjee[1]; [1]University of California, Div. of Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Texas A&M University, Dept. of Mechl. Engrg., College Sta., TX 77843-3123 USA; [3]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

The microstructure and high temperature mechanical properties of textured, polycrystalline Cu-Nb nanolayered composites prepared by magnetron sputtering were evaluated. The layer thicknesses less than or equal to 75 nm were tested in an effort to investigate high temperature mechanical properties of freestanding thin film multilayers. Effects of decreasing layer thickness on high temperature properties are presented, and show a dependence of strength and ductility on layer thickness and test temperature. The deformed specimens were characterized using transmission electron microscopy. The role of elevated-temperature deformation mechanisms such as interlayer and grain boundary are discussed. This investigation is supported by the National Science Foundation, grant # NSF-DMR-0240144 and LANL CARE grant #69757. Work at LANL is supported by DOE, Office of Basic Energy Sciences.

11:55 AM
Stability of Nanoscale Twins in Sputtered 330 Stainless Steel Thin Films: *Xinghang Zhang*[1]; Amit Misra[2]; Haiyan Wang[2]; Richard G. Hoagland[2]; [1]Texas A&M University, Dept. of Mechl. Engrg., College Sta., TX 77843-3123 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

We have recently discovered that sputter-deposited austenitic 330 stainless steel (330 SS) thin films have a nanoscale twinned structure. These twins are of {111} type with an average twin spacing of a few nanometers and the twin planes have a preferred orientation normal to the growth direction. The as-sputtered films have hardness values approaching 7 GPa, about an order of magnitude higher than that of bulk 330 SS. The unusually high strength originates from the high resistance of twin interfaces to slip transmission, as revealed by molecular dynamics simulations. In this paper we report on the thermal stability of these nanoscale twins. The evolution of mechanical behavior and electrical transport properties during annealing are correlated to the variation of microstructure such as the average twin spacing and columnar grain sizes. In addition, we explore the effect of residual stress on the formation of nanoscale twins in 330 SS.

12:10 PM
Crystal Growth and Superhardness Effect of Nano-Scale Multilayers: Nan Shao[1]; *Qianxi Lai*[1]; Yunshan Dong[1]; Fanghua Mei[1]; Geyang Li[1]; [1]Shanghai Jiao Tong University, Sch. of Matls. Sci. & Tech., Shanghai 200030 China

A comprehensive introduction is given to the coherent growth of stable phases, the stabilization of metastable phases and the crystallization of amorphous phases in magnetron sputtered nanomultilayers due to coherent interfaces by summarizing the recent work of authors. The superhardness effect resulting from this coherent growth is also discussed.

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Diffusion and Atomistic Modeling

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Fuqian Yang, University of Kentucky, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA; C. C. Chau, Pactiv Corporation, Canandaigua Technology Center, Canandaigua, NY 14424 USA; Sung Nee George Chu, Multiplex Inc, South Plainfield, NJ 07080 USA; M. Ashraf Imam, Naval Research Laboratory, Materials Science & Technology Division, Washington, DC 20375-5343 USA; Teh-Ming Kung, Eastman Kodak Company, Rochester, NY 14650 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; B. B. Rath, Naval Research Laboratory, Materials Science and Component Technology Directorate, Washington, DC 20375-5341 USA

Tuesday AM Room: 3000
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: B. B. Rath, Naval Research Laboratory, Matls. Sci. & Component Tech. Direct., Washington, DC 20375-5341 USA; S. Ankem, University of Maryland, Matls. Sci. & Engrg., College Park, MD 20742 USA

8:30 AM Invited
Atomistic Models of a Grain Boundary: *F. R.N. Nabarro*[1]; [1]University of the Witwatersrand, Sch. of Physics, PB 3, WITS 2050, Johannesburg S. Africa

The classical theory of grain boundaries is based on Bollmann's coincidence site and O lattice analysis. Special significance is given to those atomic sites in the lattice of grain 2 which would also be lattice sites or be close to lattice sites in grain 1 if that grain was extended into the domain of grain 2. No attention is given to the orientation of the grain boundary with respect to the lattices of the two grains. J.F. Nie and others have shown that in some systems the interface between two phases is governed by the requirements that lattice planes in the two phases should meet along a line lying in the interface. The physical basis is not clear, because there will generally be no atoms lying on this line. Moreover, the model does not predict discrete sets of relative orientations of the two grains and of the orientation of the boundary between them. Using the simple model of a square two-dimensional lattice, we attempt to relate these two models. For a pair of grains in contact, a low value of Bollmann's S indicates that special boundaries probably exist between the two grains. For such a case, we introduce a parameter S which depends on the orientation of the grain boundary. Boundaries with a low value of S/S are usually special. None of these models allow for physical considerations such as the directionality of bonding.

8:55 AM Invited
Coupling Grain Boundary Motion to Shear and Grain Rotation: John W. Cahn[2]; *Yuri Mishin*[1]; Akira Suzuki[1]; Jean E. Taylor[3]; [1]George Mason University, MSN 5C3, Sch. of Computatl. Scis., 4400 Univ. Dr., Fairfax, VA 22030 USA; [2]National Institute of Standards and Technology, Matls. Sci. & Engrg. Lab., Gaithersburg, MD 20899-8555 USA; [3]New York University, Courant Inst. of Math. Scis., 251 Mercer St., New York, NY 10012 USA

We present a theory in which motion of grain boundaries (GBs) results in a coupled tangential shear of the region swept by the motion. This coupling of normal motion to tangential motion has been studied by molecular dynamics simulations on high- and low-angle tilt GBs. The coupling shear is found to be a discontinuous function of misorientation with a change in sign at a critical tilt angle. An understanding of the atomic mechanism has led to a prediction and verification of the misorientation dependence of the shear. Many disparate phenomena, and interrelationships between applied shear stress, normal GB motion, GB sliding, tangential motion of grains, and grain rotation, can be explained or reinterpreted with this theory. This work also sheds some light on some of J.C.M. Li's early results on subgrain rotation and the relationship between the curvature of a tilt GB and its velocity.

9:20 AM Invited
Grain Growth and Deformation in Nanocrystalline Materials: *Chandra Shekhar Pande*[1]; Robert A. Masumura[1]; [1]Naval Research Laboratory, Physl. Metall. Branch, Code 6325, Washington, DC 20375 USA

In case of nanocrystalline materials several additional features need to be taken into account in considering grain growth and deformation whose existence was first anticipated by Prof Li years ago. Grains can increase their size by grain rotation as well as by curvature driven motion. Grain rotation has been considered in detail by Li (J. C. M Li, J. Appl. Phys., 1962, 33, 2958.) theoretically and recently other researchers by simulation. We show that in nanocrystalline materials this mode is quite possible especially if the grain growth is retarded by finite triple junction mobility. New deformation modes occur in these materials leading to the so called Inverse Hall-Petch Effect which will be discussed in detail.

9:45 AM Invited
Spontaneous Sn Whisker Growth and Impression Creep: *K. N. Tu*[1]; [1]University of California, Matls. Sci. & Engrg., Los Angeles, CA 90095-1595 USA

Spontaneous Sn whisker growth is a creep phenomenon. Because it is spontaneous, stress relaxation is accompanied by stress generation. The latter is due to the room temperature reaction between Cu and Sn to form Cu6Sn5 in the grain boundaries of Sn. The in-diffusion of one Cu atom into Sn to grow Cu6Sn5 requires the out-diffusion of one Sn atom, roughly speaking, otherwise, compressive stress will build up in the Sn. The growth rate of a whisker from the diffusion of Sn atoms to the base of the whisker driven by a stress gradient is similar to the impression creep rate due to the out-diffusion of atom under the plunger at a very slow impression rate. On the other hand, the free surfaces of Sn and Sn whiskers have a protective oxide which tends to eliminate the source of vacancies needed for diffusion, hence the oxide affects greatly the spontaneous Sn whisker growth. In this talk, a mechanism of spontaneous Sn whisker growth will be presented.

10:10 AM Break

10:15 AM Invited
Atomistic Modeling for Heat Capacities of Carbon Nanotubes: Chunyu Li[1]; *Tsu-Wei Chou*[1]; [1]University of Delaware, Dept. of Mechl. Engrg., 126 Spencer Lab., Newark, DE 19716 USA

Carbon nanotubes have many potential applications, such as nanotube-reinforced composites, nanodevices, and nanowires. These applications take advantage of their remarkable mechanical and physical properties. Recently, increasing attention has been paid to the thermal behaviors of carbon nanotubes. Some experimental and theoretical studies have been devoted to the determination of their heat capacity. However, some important issues, such as the effects of tube diameter and tube chirality, and the differences in the heat capacity among isolated SWNTs, SWNT bundles and MWNTs, are still not clear. In this paper, we study the heat capacity of carbon nanotubes by using an atomistic modeling technique, namely, the molecular structural mechanics method. The vibrational modes of the nanotube are quantized according to the theory of quantum mechanics. The dependence of the heat capacities of carbon nanotubes on temperature, tube diameter and tube chirality is investigated and our modeling predictions are compared with existing results.

10:40 AM Invited
Molecular Potential Finite-Element Method (MPFEM) for Cell Mechanics: Ju Li[2]; Ming Dao[1]; *Subra Suresh*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg. & Div. of Biologl. Engrg., Rm. 8-303, 77 Mass. Ave., Cambridge, MA 02139 USA; [2]Ohio State University, Dept. of Matls. Sci. & Engrg., 494 Watts Hall, 2041 Coll. Rd., Columbus, OH 43210 USA

Akin to the interatomic potential finite-element method (J. Mech. Phys. Solids 52, 691), we have developed an on-the-fly homogenization scheme for studying the mechanics of living cells that comprise two-dimensional/three-dimensional molecular networks as structural bases. For the particular case of the spectrin network undercoat of the human red blood cell (RBC) wall that provides most of its shear elasticity, we use the worm-like chain (WLC) potential for single spectrin molecular response. However more accurate molecular potentials, once measured from single-molecule stretching experiments, can be employed as well. Multiple local energy minima in RBC shape are found, that include the biconcave, the cup, and even randomly crumpled shapes. Optical tweezers stretching up to 100% elongation (mimicking RBC deformation in capillaries) are simulated and compared with experiments. MPFEM provides a bridge between whole-cell mechanics and single-molecule stretching response and the underlying structure of the spectrin network. Triangular, cubic and hexagonal spectrin networks with various defects and disorder are studied. We are also starting MPFEM calculations using real AFM scanned spectrin network micrographs as the homogenization area element for cell wall.

11:05 AM Invited
The Effect of Geometry on Chemical Stresses in Boundary Layer Diffusion: *Sanboh Lee*[1]; Sun-Chien Ko[1]; Y. T. Chou[2]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101 Kuang Fu Rd., 2nd Sec., Hsinchu, Taiwan 300 China; [2]University of California, Dept. of Chemi. Engrg. & Matls. Sci., Irvine, CA 92606 USA

Consider a layer A of thickness 2a sandwiched between two semi-infinite layers B of different chemical compositions. Three geometries of cross section are considered: slab, solid circle and square. Two types of diffusant sources are studied. One is the constant surface concentration source and the other is instantaneous surface concentration source. Assume that the diffusivity of diffusant in layer A is much greater than that in layer B. This problem has similar mathematical equations to the grain boundary diffusion. The concentration distribution in layer A of slab cross section was obtained using Fourier Laplace transform technique. This solution was applied to GaAs/AlAs/GaAs layer system. The stress distribution in layer A was solved using Hsueh method. Hsueh proposed three equations of total force, bending force and moment to solve bending axis, uniform strain and curvature. The stress distribution in layer A of circular cross section was solved the force equilibrium equations. The tangential and radial stresses were obtained in a close form. The force equilibrium equations of layer A of square cross section were digitized. The numerical solutions of stresses developed in layer of square cross section were obtained. They have common characterization that the maximum stresses occur near the free surface at a short time. A comparison among the chemical stresses in slab, solid circle and square cross sections are made.

11:30 AM
Interaction Between Diffusion and Chemical Stresses: *Fuqian Yang*[1]; [1]University of Kentucky, Chem. & Matls. Engrg., Lexington, KY 40506 USA

The interaction between chemical stresses and diffusion is studied. Based on the results of Li[1] and Larch and Cahn,[2,3] a new relation between hydrostatic stress and the concentration of solute is established. For a solid free of the action of body force, the Laplacian of the hydrostatic stress is proportional to the Laplacian of the concentration of solute - that is, deviation of the hydrostatic stress from its local average is proportional to deviation of the local concentration of solute. A general relationship among the surface concentration, the normal stress and the surface deformation of a solid is obtained, in which the normal stress is a function of the mean curvature of the undeformed surface and tangential components of the surface displacement. Using the new relationships, the evolution of chemical stresses in a thin plate is discussed. A closed-form solution of the steady state concentration of solute is derived. It turns out that linear distribution of solute is non-existe nt due to the interaction between chemical stresses and diffusion. [1]J.C.M. Li, Metall. Trans. 9A(1978) 1353-1380; [2]F.C. Larche and J.W. Cahn, Acta Metall. 30(1982) 1835; [3]F.C. Larche and J.W. Cahn, J. Res. Natl. Bur. Stand. 89(1984) 467.

11:50 AM Invited
Molecular Dynamics Simulation of Barnacle Cement: *Ya-Pu Zhao*[1]; Jun Yin[1]; [1]Chinese Academy of Sciences, State Key Lab. of Nonlinear Mech. (LNM), Inst. of Mech., Beijing 100080 China

Barnacle cement is an underwater adhesive that is used for permanent settlement. Its main components are insoluble protein complexes which have not been fully studied. In present article, we chose two proteins of barnacle cement for study, 36-KD protein and Mrcp-100k protein. In order to investigate the characteristics of above two proteins, we introduced the method of molecular modeling. And the simulation package GROMACS was used to simulate the behavior of these proteins. In this article, we mainly focused on two properties of these two proteins: structure stability and adhesive ability. First we simulated the structure stability of two proteins in vacuum, then the stability of 36-KD protein in solutions, in water and seawater environment was investigate. We found that the stability varies in the different environment. Next, to study adhesive ability of two proteins, we simulated the process of peeling the two proteins from the substrate (graphite). Then, we analyzed the main reason of these results. We fo und that hydrogen bond in proteins play an important role in the protein stability. Of course there are also other factors such as van der Waals (vdW) interactions and electrostatic interactions. In the process of the peeling, we used Lennard-Jones 12-6 formula to calculate the vdW interactions and found that it is vdW interactions that work effectively between proteins and substrate. At last, we introduced some theories to explain the results, compared the theoretical results with the results obtained by molecular dynamics (MD) simulations.

12:10 PM
Three-Dimensional (3D) Microstructure Visualization and Finite Element Modeling of the Mechanical Behavior of Heterogeneous Materials: Vasudevan Ganesh[1]; Rajen S. Sidhu[1]; *Nik Chawla*[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287-6006 USA

The mechanical behavior of materials is inherently controlled by microstructure. In particular, heterogeneous materials, consisting of two or more components or phases, have complex microstructures. This makes modeling of the mechanical behavior a challenge. We have developed a three dimensional (3D) approach to (a) constructing a "virtual microstructure" in 3D by serial sectioning technique, and (b) finite element modeling using the 3D microstructure as a basis. In this talk we will explore the fundamentals of the 3D virtual microstructure modeling methodology. This methodology was used to study the deformation behavior of two important systems, (i) SiC particle reinforced metal matrix composites, and (ii) Sn-3.5Ag solder alloys. The role of second phase fraction, morphology, and aspect ratio on deformation was quantified and will be discussed. Results from the microstructure based 3D simulations were found to be in good agreement with the experimental observations, indicating the importance and effectiveness of 3D microstructure-based simulations.

Microstructural Processes in Irradiated Materials: RPV Embrittlement and Oxide Dispersion Strengthened Alloys

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Tuesday AM Room: 3011
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Steve Zinkle, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6138 USA; Graeme J. Ackland, University of Edinburgh, Sch. of Physics, Edinburgh, Scotland EH9 2LZ UK

8:30 AM
Precipitation in Neutron Irradiated Copper Free RPV Steels: *G. Robert Odette*[1]; Mike K. Miller[2]; K. F. Russell[2]; Brian D. Wirth[3]; [1]University of California, Dept. of Mechl. Engrg., Santa Barbara, CA 93106 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6136 USA; [3]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720-1730 USA

The formation of Mn-Ni-Si phases that would produce large and unanticipated hardening and embrittlement in irradiated Cu-free reactor pressure vessel (RPV) steels was predicted more than a decade ago. Since large incubation doses were expected, these features were dubbed "late blooming phases" (LBP); however, LBP have proved to be an elusive quarry. Recent small angle neutron scattering, atom probe tomography and combined electrical resistivity Seebeck coefficient measurements have now provided unequivocal evidence of LBP in Cu-free alloys containing 1.6%Mn, 1.6%Ni, 0.25%Si and 0.005 to 0.040%P irradiated at 270°C at intermediate flux to ≅ 0.025 dpa. All techniques showed the Mn-Ni-Si rich features, with nominal volume fractions up to ≅ 0.6%, in the 0.005%P alloy; however, solute clusters were not observed in the atom probe tomography examinations of the alloy with 0.040%P. Hardening ranged from ≅ 160 to 190MPa, consistent with the estimated precipitate volume fractions.

8:50 AM
The Effects of Irradiation, Annealing and Reirradiation on an A533B RPV Steel: *Michael K. Miller*[1]; Randy K. Nanstad[1]; Mikhail A. Sokolov[1]; Kaye F. Russell[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, MS 6136, Oak Ridge, TN 37831-6136 USA

The mechanical properties, number density, size, and composition of precipitates in an A533B RPV steel have been characterized through two irradiation and annealing cycles. Atom probe tomography re-

vealed that irradiation (fluence = $5x10^{23}$ m^{-2}) (E>1MeV) produced a high number density of copper-, manganese-, nickel- and silicon-enriched precipitates. After annealing (168 h at 460°C), their number density significantly decreased. After irradiation (fluence = $0.85x10^{23}$ m^{-2}), annealing and re-irradiation (fluence = $0.85x10^{23}$ m^{-2}), a high number of copper-enriched precipitates was observed. A low number density of some large copper-rich precipitates was observed after the second annealing treatment. Research at the Oak Ridge National Laboratory SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the U. S. Department of Energy.

9:10 AM
Investigation of Irradiation-Induced Hardening in Low-Alloy Steels by STEM-XEDS Spectrum Imaging: *Masashi Watanabe*[1]; David B. Williams[1]; [1]Lehigh University, Dept. of Matls. Sci. & Engrg., 5 E. Packer Ave., Bethlehem, PA 18015 USA

Properties and microstructures of materials can significantly be modified after irradiation. Irradiation-induced hardening is frequently observed in low-alloy steels. This phenomenon can be due to the formation of ultra-fine solute-enriched "clusters/precipitates." Previously, X-ray mapping using a scanning transmission electron microscope (STEM) equipped with an X-ray energy dispersive spectrometer (XEDS) has been employed and 2 to 3 nm Ni-enriched "precipitates" have been detected in the matrix. In this study, further quantitative STEM-XEDS mapping has been performed by using a spectrum imaging (SI) technique, which stores a whole spectrum at an individual pixel. In order to enhance weak signals from the fine precipitates, multivariate statistical analysis (MSA) has been applied to the measured SI data and the presence of Mn and Cu has been confirmed besides Ni in the fine precipitates. The combination of SI with MSA can be the ideal approach for investigation of fine features in complex microstructures.

9:30 AM
On the Correlation Between Matrix Damage and Cu-Precipitation: *Abderrahim Almazouzi*[1]; [1]SCK.CEN, Reactor Matls. Rsch., LHMA, Boeretang 200, Mol 2400 Belgium

Within the framework of the european PERFECT project, binary Fe-Cu model alloys are used to assess the contribution of matrix damage and Cu-precipitates to the total strength hardening of the material. Positron annihilation techniques in conjunction with tensile testing are used to identify the effect of the microstructure on the materials behaviour. Both thermal aging and neutron irradiation have been performed in order to separate and quantify the obstacles as function of temperature, time, dose and flux.

9:50 AM
Solute Interaction with Point Defects in a Fe Under Irradiation: A Combined Ab Initio and Kinetic Monte Carlo Approach: *Edwige Vincent*[1]; Charlotte S. Becquart[1]; Christophe Domain[2]; [1]Université des Sciences et Technologies de Lille, Lab. de Métall. Physique & Génie des Matériaux, UMR 8517, Bat. C6, Villeneuve d'Ascq Cédex F-59655 France; [2]EDF-R&D, Dépt. MMC, Les Renardières, Moret sur Loing cédex F-77818 France

Solute Cu plays a major role in the embrittlement of pressure vessel steels under radiation. In RPV steels and dilute FeCu alloys, the Tomographic Atom Probe has revealed the formation of Cu atmospheres under neutron flux. More recently the role of other solutes such as Ni, Mn and Si which are also within the atmospheres have been put forward. It is thus very important to characterise the interactions of these solutes with radiation induced point defects in order to understand the elementary mechanisms behind the formation of these atmospheres. We have investigated by ab initio calculations based on the density functional theory the interactions of point defects and solute atoms in dilute FeX alloys (X = Cu, Mn, Ni or Si). The different possible configurations of small solute clusters, solute-vacancy complexes and solute-dumbbell complexes have been studied. Their formation and binding energies are discussed, as well as the migration energies for the most interesting configurations. These data have been used to derive a parameterisation to couple the diffusion of point defects with solute atoms, in order to simulate the evolution of solute atoms by Kinetic Monte Carlo. First results will be presented and compared to some experimental observations.

10:10 AM Break

10:40 AM Invited
Nano-Mesoscopic Structural Control in 9Cr-ODS Ferritic/Martensitic Steels: *Shigeharu Ukai*[1]; [1]Japan Nuclear Cycle Development Institute, Oarai Engrg. Ctr., Sys. Engrg. Tech. Div., 4002, Narita, Oarai-machi, Higashi-Ibaraki-Gun, Ibaraki-Prefecture 311-1393 Japan

The equi-axial grain structure of 9Cr-oxide dispersion strengthened (ODS) steels with a composition of 9Cr-0.13C-2W-0.2Ti-0.35Y2O3 is controlled by the alpha and gamma phase transformations. The appropriate selection of titanium and excess oxygen contents induces residual alpha grain formation, which is network-like shape and contains the ultra fine and dense Y2Ti2O7 type complex oxide particles with 1.5 nm size. The thermodynamic model calculation quantitatively revealed the condition of the residual alpha grain formation. In addition, for 9Cr-ODS steels containing the residual alpha grains, the critical cooling rate to induce martensitic phase transformation is as high as 10,000 K/h, due to one micron size of prior gamma grains. Prevention of martensitic phase transformation should suppress sliding among packet grain boundaries. It is concluded that excellent high temperature strength of 9Cr-ODS steels is significantly enhanced by the formation of network-like residual alpha grains containing ultra fine Y2Ti2O7 particles and packet-free grain boundaries.

11:20 AM
Precipitation and Stability of Nanometre Particles in ODS Alloys: Philippe Pareige[1]; *Mike Miller*[2]; David T. Hoelzer[2]; Emmanuel Cadel[1]; Roger E. Stoller[2]; [1]CNRS, ERT, Inst. des Matériaux, Ave. de l'Univ., St. Etienne du Rouvray BP12 76801 France; [2]Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6136 USA

The microstructures of commercial mechanically-alloyed oxide dispersion strengthened alloys (MA957 and 12YWT) have been characterized in the as-received condition, after annealing up to 20h at 1300°C and after ion irradiation (Fe ions at 300°C up to 0.5 dpa for 12YWT). A comparison of the behaviour of these two materials before and after ageing is reported. Studies were performed by three dimensional atom probes that give access to information on the microstructure at the atomic scale. It has been revealed, that the Ti-,Y- and O- enriched particles are stable under thermal ageing or irradiation in the 12YWT. A slight coarsening of the particles is observed in the MA957 material after 24h at 1300°C and a high oxygen content was measured in the ferrite matrix. The lower molybdenum content in MA957 was found to be less effective in trapping oxygen than the higher tungsten content in 12YWT alloy.

11:40 AM
Molecular Dynamics Simulation of Primary Irradiation Defect Formation in Fe-Cr Alloys: *Jae-Hyeok Shim*[1]; *Brian D. Wirth*[1]; [1]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720 USA

Ferritic-martensitic Fe-Cr alloys represent a technologically important class of candidate materials for fusion first wall and blanket structures facilities. These alloys will experience severe irradiation environments including the creation of atomic displacements in high-energy displacement cascades and damaging concentrations of helium and hydrogen. We present the results of molecular dynamics simulations to investigate high energy displacement cascade evolution and the properties of point defect clusters in an Fe-10%Cr alloy. Finnis-Sinclair potentials for Fe and Cr are used to describe the interatomic behavior. A previously published Fe-Cr cross-potential, was slightly modified to fit the heat-of-mixing and lattice constant data of these alloys. Displacement cascade simulations with PKA energies of 20 and 40 keV were performed. The morphology, energetics and mobility of primary irradiation defects were characterized in terms of PKA energy and temperature, and compared to the cascade evolution and defect production in pure Fe.

Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Experimental Methods for Determining Diffusion Mechanisms

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee, MPMD-Solidification Committee, ASM/MSCTS-Atomic Transport Committee

Program Organizers: Carelyn E. Campbell, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Ursula R. Kattner, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD 20899-8555 USA; Afina Lupulescu, Rensselaer Polytechnic Institute, Materials Science & Engineering, Troy, NY 12180-3590 USA; Yongho Sohn, University of Central Florida, Advanced Materials Processing & Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816-2455 USA

Tuesday AM Room: 3007
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Yongho Sohn, University of Central Florida, Dept. of Mechl. Matls. & Aeros. Engrg., Orlando, FL 32816-2455 USA; Richard D. Sisson, Worcester Polytechnic, Ctr. for Heat Treat Excellence, Worcester, MA 01609 USA

8:30 AM Invited
Effects of Composition on Kinetics of Intermetallic Layer Growth for Soldered Copper Alloys: *Evan K. Ohriner*[1]; [1]Oak Ridge National Laboratory, PO Box 2008, Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

The formation of intermetallic compounds on a number of copper alloys soldered with a variety of tin-containing solders has been studied for the temperature range of 150 to 250 C for times of up to 4000 hours. Tin-containing intermetallic layers exhibit parabolic layer growth under all conditions studied. Pure copper and Cu-5Sn-0.2P behave similarly, forming layers of Cu_3Sn and Cu_6Sn_5. A series of Cu-Ni-Sn alloys with nickel contents ranging from 6 to 23% form only a $(Cu,Ni)_6Sn_5$ intermetallic. The parabolic rate constant for layer growth is very sensitive to nickel content of the alloy, increasing rapidly to peak rate with additions of up to 9% nickel and then decreasing to a minimum rate with the highest nickel contents. The intermetallic layer thickness constant varies by a factor of 30 among the nickel-containing alloys. This unusual phenomenon is discussed.

9:00 AM
A Novel Double-Layered Titanium Boride Coating on Titanium: Kinetics of Boron Diffusion and Coating Development: *Nishant M. Tikekar*[1]; K. S. Ravi Chandran[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Increasing the surface performance of titanium and its alloys critically depends on finding an effective coating concept that can prevent galling, seizure and contact deformation of titanium surfaces. While there have been several externally applied coating concepts for titanium, none have been quite reliable. In this study, we performed solid-state diffusion experiments in a variety of boron-containing mediums to explore the possibility of making novel titanium boride coatings on titanium. The coating consisted of a double layer that involved a titanium diboride (TiB_2) top layer and a titanium monoboride (TiB) sublayer, which was primarily made of long, pristine TiB whiskers growing into titanium. Maximum thicknesses of about 10 μm TiB_2 and about 40 μm TiB whisker layer have been achieved by careful control of diffusion kinetics. The kinetics of diffusion was studied and was found to be governed by the ratio of the ingredients of the powder pack, the temperature and the time of the diffusion process. A two-layer diffusion model was developed to illustrate the nature of coating formation.

9:25 AM
Diffusion Mechanism in Two-Phase Intermetallic Titanium Aluminde Alloys: *Fritz Appel*[1]; [1]GKSS Research Centre, Inst. for Matls. Rsch., Geesthacht D-21502 Germany

Intermetallic titanium aluminides exhibit attractive thermo-physical properties, which give them the potential for extensive use as lightweight structural components. Engineering alloys are multiphase assemblies with complex constitution and microstructures. In the present paper the diffusion mechanisms occurring in these material will be investigated. Particular emphasis is placed on the effect of off-stoichiometric deviations, which produce a significant chemical disorder due to the formation of antisite defects. These defects gives rise to fast diffusion via antistructural bridges, which is particularly effective at low homologous temperature. The implication of this diffusion mechanism on the structural and mechanical properties of TiAl-alloys will be investigated. The major areas of the study involve: phase transformations, static and dynamic strain ageing, and creep.

9:50 AM
Grain Boundary Diffusion of Fe and Co in High Purity Iron: *Akiko Inoue*[1]; Koichi Takasawa[1]; Hiroyuki Nitta[2]; Junichi Koike[1]; Yoshiaki Iijima[3]; [1]Tohoku University, Matls. Sci., 02 Arakamaki Aobaku, Sendai, Miyagi 980-8579 Japan; [2]Tohoku Univerisity, Inst. for Matls. Rsch., 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi 980-8577 Japan; [3]Iwate University, Matls. Sci. & Tech., 3-18-8 Ueda, Morioka, Iwate 020-8551 Japan

It has been well known that the bulk self-diffusion in iron depends on the magnetic spin ordering. This study focuses on the influence of it on the grain boundary diffusion. The radioisotopes of ^{59}Fe and ^{57}Co were electroplated on the mirror-like surface of a high purity iron(C:0.7, S:1.0, N:0.7, O:2.0, P:1.0 mass ppm). The specimens were annealed under 10^{-4} Pa in the temperature range 553-1173K. A serial sectioning method was employed. At lower temperature range, the type C kinetics regime was applied. The grain boundary self-diffusion coefficient, D_{gb}^{Fe}, obtained in the present work was much larger than those by other authors. The activation energy of D_{gb}^{Fe} in the paramagnetic α-Fe was obtained to be 55.7kJ/mol which was only 0.22 of that for the bulk self-diffusion. Below the Curie temperature the decrease in both D_{gb}^{Fe} and D_{gb}^{Co} is remarkably larger than that in the bulk diffusion coefficients.

10:15 AM Break

10:30 AM
Interdiffusion in the Iron-Rich Part of the Ternary Fe-Cr-Al System: Experimental and Simulation Studies: *Guenter Borchardt*[1]; [1]TU Clausthal, Metall., Robert-Koch-Strasse 42, Clausthal-Zellerfeld 38678 Germany

Interdiffusion experiments were carried out on model alloys FexCryAlz with 0.57 x 0.80, 0.06 y 0.32, 0.01 z 0.29 in the temperature range 800°C T 1300°C. From the resulting concentration profiles the two main and two cross-interdiffusion coefficients required for the description of ternary diffusion were determined. For the calculation of the ternary diffusivities, a recent numerical approach was used in which the four ternary interdiffusion coefficients are determined over a selected composition range from a single diffusion couple experiment. The on-diagonal coefficients on both sides of the Matano interface were determined in the high and low Al and Cr concentration part of the diffusion couples. They show an Arrhenius behaviour with activation enthalpies between 158-203 kJ/mol for Cr-high regions, 144-228 kJ/mol for Cr-low regions, 171-221 kJ/mol for Al-high regions and 170-223 kJ/mol for Al-low regions.

10:55 AM
Precipitation-Strengthened Al-Sc-Ti Alloys Studied by Three-Dimensional Atom-Probe Microscopy: *Marsha E. van Dalen*[1]; David C. Dunand[1]; David N. Seidman[1]; [1]Northwestern University, Matls. Sci. & Engrg. Dept., 2220 N. Campus Dr., Evanston, IL 60208 USA

Currently, most precipitation-strengthened aluminum alloys are limited to usage at relatively low temperatures, because of the rapid coarsening and/or dissolution of their precipitates. Al-Sc alloys represent an exception, because they contain nanosize, coherent Al_3Sc precipitates (L12 structure) with low coarsening rates. In the present study, titanium was added as a ternary alloying element because it diffuses more slowly than scandium in aluminum and it has a high solubility in Al_3Sc. Al-Sc-Ti alloys are cast, solutionized and aged at temperatures in the range of 300-450°C to form nanosize, coherent $Al_3(Sc,Ti)$ precipitates within coarse aluminum grains. The Ti additions are found to decrease the coarsening kinetics as compared to Al-Sc alloys. The diffusion coefficient of Ti in Al is so small, however, that only a tiny concentration of Ti is detected in the precipitates by three-dimensional atom-probe microscopy. The resulting creep properties of the Al-Sc-Ti alloys are also discussed.

11:20 AM
Diffusion in Al-Ni-Ce Melts: *Axel Griesche*[1]; Michael-Peter Macht[1]; Günter Frohberg[2]; [1]Hahn-Meitner-Institute, Matls., Glienicker Str. 100, Berlin 14109 Germany; [2]Technical University Berlin, Inst. for Matl. Scis. & Tech., Hardenbergstr. 36, Berlin 10623 Germany

We investigate the influence of the melt's structure and the influence of thermodynamic forces on diffusion in the liquid state of ternary Al-based alloys. The long-capillary method was used to measure

self- and interdiffusion in liquid Al-Ni-Ce alloys at temperatures above liquidus. The interdiffusion coefficients Dik of all element pairs i-k were determined for a mean composition of Al87Ni10Ce3 at 1273 K and for a mean composition of Al77Ni20Ce3 at 1373 K. The self diffusion coefficients of Ni and Ce in liquid Al87Ni10Ce3 were measured by use of the penetration of enriched stable 62Ni- and enriched stable 142Ce-isotopes. Convective contributions to the mass transport were detected by measuring the time dependence of the diffusion coefficients and by direct observation with in-situ x-ray radioscopy. The concentration profiles were analyzed after solidification by means of energy-dispersive x-ray spectroscopy (EDS) in the case of chemical diffusion and by means of inductively coupled plasma mass spectrometry (ICP-MS) in the case of self diffusion. The chemical potentials of the elements in the melt were calculated using the commercial software Pandat. The influence of thermodynamic forces on diffusion is discussed in the framework of Darken's approach, which connects e.g. for a (solid) binary system the interdiffusion coefficient Dik with the self diffusion coefficients D'i and D'k by the equation Dik=(Nk·D'i+Ni·D'k)·F with F the thermodynamic factor representing a force due to gradients of the chemical potential and Ni and Nk the mole fractions of both elements. The influence of the structure of the melt on diffusion is discussed together with results of diffusion experiments from literature done by quasi-elastic neutron scattering and done by molecular dynamics simulations.

Neutron Diffraction Characterization of Mechanical Behavior: Deformation II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Hahn Choo, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

Tuesday AM Room: 3004
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Mark A.M. Bourke, Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA; Ersan Ustundag, Iowa State University, Dept. of Matls. Sci. & Engrg., Ames, IA 50011 USA

8:30 AM Invited
Modeling Grain Interactions in Polycrystals with Crystals Discretized with Finite Elements: *Paul R. Dawson*[1]; Tong-Seok Han[1]; [1]Cornell University, Sibley Sch. of Mechl. & Aeros. Engrg., 196 Rhodes Hall, Ithaca, NY 14853 USA

Neutron diffraction data provide detailed information on the lattice strains during loading of polycrystalline solids that offer insight into the characteristics of load sharing among crystals that comprise the aggregate. Such data are particularly useful when combined with finite element modeling of polycrystals in which the influence of crystallographic neighborhood can be explicitly included in a simulation. This presentation summarizes the comparisons between diffraction experiments and finite element simulations of a two-phase (iron-copper) alloy. During loading into the fully plastic regime, the alloy demonstrates several stages in load sharing as each phase makes the transition from elastic to elastic-plastic behavior. Simulation results compare well with lattice strain histories for several combinations of scattering vector and crystal plane. From the simulations, the nature of the grain interactions that lead to the various stages can be explained.

8:50 AM
Effects of Grain Size on the Micromechanics of Deformation in Ni: *J. W.L. Pang*[1]; R. R. Rogge[2]; R. L. Donerberger[2]; W. Liu[3]; G. E. Ice[1]; [1]Oak Ridge National Laboratory, Metals & Ceram., PO Box 2008, MS6118, Oak Ridge, TN 37831 USA; [2]National Research Council Canada, Steacie Inst. for Molecular Sci., Chalk River Lab., Chalk River, Ontario K0J 1J0 Canada; [3]University of Illinois, Frederick Seitz Matls. Rsch. Lab., 104 S. Goodwin Ave., Urbana, IL 61801-2902 USA

Capability of synergistic combination of neutron and synchrotron diffraction to provide the information for crystal plasticity modeling is presented. A series of Ni tensile samples of different grain sizes were measured by both techniques to investigate deformation behavior on the microscopic and mesoscopic length scales. Samples of grain size ranging from hundreds to few micrometers have been measured at different deformation levels with neutron diffraction. Results indicate that the trends in the microstrain evolution are similar for all grain sizes; however the strain magnitudes are generally larger for samples with smaller grain sizes. The deformed samples were then examined by the 3D X-Ray Crystal Microscope at the Argonne Photon Source. Variations in strains and misorientations within grains, subgrains and grain boundaries were determined. The experimental results were compared with the finite element deformation model.

9:10 AM
Evolution of Intergranular Stresses During In Situ Straining of IF Steel with Different Grain Sizes: *João Quinta da Fonseca*[1]; Pete S. Bate[1]; [1]University of Manchester, Sch. of Matls., Grosvenor St., Manchester, Greater Manchester M1 7HS UK

The heterogeneous nature of plastic deformation of polycrystalline metals at the microscopic scale gives rise to intergranular stresses, which influence fatigue, stress corrosion cracking and also skew residual stress measurements made by diffraction. Grain size is known to have a significant effect on the plastic deformation of metals, but the effect of grain size on the development of intergranular stresses has yet to be investigated. This article presents diffraction measurements obtained during in-situ uniaxial tensile straining of interstitial free (IF) steel with different grain sizes. These are compared to predictions made using crystal plasticity finite element modelling (CPFEM). As well as comparing mean elastic strains by tracking peak position, changes in peak width are correlated with slip activity and predicted spreads in elastic strains.

9:30 AM
Deformation Behavior of Fe-Cu Alloy Composites With a Bimodal Grain Size Distribution: *Jin-woo Jeon*[1]; Hahn Choo[1]; Peter K. Liaw[1]; Guojiang Fan[1]; [1]University of Tennessee, Matl. Sci. & Engrg., 318 Dougherty Hall, Knoxville, TN 37996 USA

Strengthening of an alloy is usually achieved at the expense of the ductility. However, recently developed alloys with so-called "bimodal grain size distribution" exhibit a unique mechanical properties, i.e., high strength with appreciable ductility. To investigate the micromechanics responsible for the unique mechanical behavior of the bimodal-grain-sized (BGS) alloys, immiscible Fe-Cu alloy composites were fabricated by sinter-forging of a mixture of ball-milled ultrafine Fe powder and coarse Cu powder. The microstructure and deformation behavior of the BSG Fe-Cu alloy composites were investigated using x-ray diffraction, transmission electron microscopy, tensile test, and nanoindentation test. Furthermore, in-situ tensile loading measurements were performed using neutron diffraction to investigate the intergranular strain evolution and load partitioning between the ultrafine (Fe) and coarse (Cu) grains.

9:50 AM Invited
Constitutive Behavior of Ferroelectrics: *Ersan Ustundag*[1]; Robert C. Rogan[1]; S. Maziar Motahari[1]; Mark R. Daymond[2]; [1]Iowa State University/Ames Laboratory, MSE Dept., 2220 Hoover Hall, Ames, IA 50011 USA; [2]ISIS Facility, Rutherford-Appleton Lab., Chilton, Didcot OX11 0QX UK

Ferroelectric materials exhibit a unique response to electromechanical loading and can be used as both sensors and actuators. This presentation will present recent results from in-situ uniaxial compression experiments on various Pb(Zr,Ti)O3 or PZTs using neutron diffraction to determine their constitutive behavior. PZTs near the edge of the morphotropic phase boundary as well as single phase (tetragonal and rhombohedral) specimens were investigated. Then a new self-consistent micromechanics model will be presented. This model considers different domain variants and attempts to estimate strain and texture (or domain switching) evolution during the loading of ferroelectrics. Finally, the diffraction data will be compared to the predictions of this model.

10:10 AM
In-Situ Tensile Loading Study of Nanocrystalline Nickel: *Xun-Li Wang*[1]; A. D. Stoica[1]; J. Almer[2]; C. T. Liu[3]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, Bldg. 8600, Oak Ridge, TN 37831-6474 USA; [2]Argonne National Laboratory, Advd. Photon Source, 9700 Cass Ave., Argonne, IL 60439 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S, Oak Ridge, TN 37831 USA

The deformation behavior in nanocrystalline nickel was investigated using high energy synchrotron radiation. The first five full diffraction rings were recorded allowing a complete grain orientation analysis of diffraction line shift and broadening. The evolution of

lattice strains was determined during the continuous tensile loading up to failure, as well as during the loading-unloading cycles. For a sample with a grain size of 15 nm, the fracture strength was found to be 1.3 GPa and the maximum recorded elongation was 3.8%. Besides the characteristic lattice strain behavior, the diffraction lines do not show any irreversible broadening after unloading, since the broadening accumulated during the plastic deformation is released upon unloading. In nanocrystalline materials the peak broadening due to strain heterogeneity is overshadowed by the grain size contribution. However, the quite high accuracy in peak profile analysis allows us to conclude that, in nanocrystalline nickel, the immobile dislocations are not accumulating during the plastic deformation, contrast to the coarse grained materials. This research was sponsored by Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC05-00OR22725 with UT-Battelle, LLC. Use of the Advanced Photon Source was supported by the U. S. Department of Energy, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38.

10:30 AM Break

10:50 AM Invited
Atomistic Mechanism of Plastic Deformation in Metallic Glasses: *Takeshi Egami*[1]; [1]University of Tennessee/Oak Ridge National Laboratory, MSE/Physics, 208 S. Coll., 1413 Cir. Dr., Knoxville, TN 37996 USA

While it is well established that mechanical deformation of crystalline materials occurs through the motion of dislocations, the atomistic mechanism of deformation in metallic glasses is poorly understood. It is often phenomenologically described in terms of free volume model, it is becoming clear that the reality of "free volume" is not vacancy-like as originally imagined, but is more collective involving a large number of atoms. On the other hand experimentally it has been observed that homogeneous anelastic deformation results in bond-orientational anisotropy. We propose an atomistic mechanism of plastic deformation in metallic glasses based upon stress-induced bond exchange mechanism. Even though the each element of space used for deformation is much smaller than the atomic size, thus not vacancy-like as free volume, because of the long-range stresses involved the total activation energy is large, comparable to that in crystalline solids. The implication of this model on ductility will be discussed.

11:10 AM Invited
Formation of Free Volume During Mechanical Deformation: *Matthew J. Kramer*[1]; Bulent Biner[1]; Dan J. Sordelet[1]; [1]Iowa State University, Ames Lab., 37 Wilhlem, Ames, IA 50011 USA

The atomic scale deformation in bulk metallic glasses has received considerable attention recently. At low temperature, deformation is concentrated in localized shear bands. At temperatures approaching the glass transition temperature up to the crystallization temperature, deformation appears to be homogeneous. Using high energy synchrotron radiation (~100 keV), we investigated the changes in the total scattering function and shifts in the average bond lengths to attempt to quantify the changes in free volume associated with the mechanical deformation. Room temperature indentation on Vitroloy showed a small decrease in the free volume ranging from 0.1 to 0.23%. High temperature creep experiments at 250 and 400 MPa showed about 0.35% increase in free volume. Experimental results will be compared to molecular dynamic calculations.

11:30 AM Invited
Icosahedral Order in Undercooled Metallic Liquids and the Influence on the Nucleation Barrier: *Kenneth Franklin Kelton*[1]; [1]Washington University, Dept. of Physics, Campus Box 1105, One Brookings Dr., St. Louis, MO 63130 USA

Over a half-century ago, Frank argued that liquid metals can be undercooled because of the development of icosahedral short-range order (ISRO) in the liquid. The existence of this ISRO (though often distorted) is supported by recent in-situ neutron and x-ray scattering studies of undercooled liquids that are levitated by electrostatic and electromagnetic methods. In a TiZrNi alloy, the developing ISRO favors the transformation of the liquid to a metastable icosahedral quasicrystal phase, instead of the stable tetrahedrally-coordinated crystal C14 Laves phase, demonstrating a clear connection between the nucleation barrier and the local structure of the liquid and verifying Frank's hypothesis. The consequences of this coupling between the order parameter characterizing the structure of the liquid and that for nucleation are discussed. Supported by NASA under contract NAG 8-1682, and by the NSF under grant DMR 03-07410.

11:50 AM Invited
The Mechanical Behavior of Ceramic Membranes in Oxygen Partial-Pressure Gradients: *James W. Richardson*[1]; Yaping Li[1]; Evan R. Maxey[1]; [1]Argonne National Laboratory, Intense Pulsed Neutron Source, 9700 S. Cass Ave, Argonne, IL 60439 USA

Dense ceramic components with mixed conduction properties and high oxygen permeability are important as membranes for oxygen separation and solid oxide fuel cell applications. Many of the most promising are perovskite-derived oxides, due to their structural stability over large ranges of chemical composition and oxygen vacancy concentration. Membranes are typically operated at elevated temperatures (800-1000°C) and exposed to large oxygen partial pressure (pO_2) gradients. A number of factors limit the lifetime of a membrane, including chemical decomposition, phase transformation and mechanical instability associated with internal strain generated by lattice parameter gradients. In-situ neutron diffraction, with a large beam, high penetrating power and sensitivity to scattering from oxygen, is extremely effective at characterizing ceramic oxide membranes in operational conditions. Studies of materials in the La-Sr-Fe-Co-O system under static reducing conditions show dramatic lattice expansion ($\Delta a/a$ up to $6x10^{-3}$) as a response to Co/Fe ionic radius changes. Measurements from membranes exposed to large oxygen partial pressure gradients (e.g., $pO_2 = 10^{-1}$ and 10^{-20} on opposing sides) - with corresponding structural gradients - across thin (~1mm thick) membrane tubes provide structural representations integrated across the structural gradients. As expected, the average structure has composition and lattice dimension intermediate between the two extremes represented by the surface environments. Unexpected, though, is the small distribution of composition and lattice dimension, with average near the conditions at the oxidizing surface. The implications for mechanical stability of ceramic membranes and importance of surface properties will be discussed. The Intense Pulsed Neutron Source at Argonne National Laboratory is funded by the U.S. Department of Energy under Contract W-31-109-ENG-38.

Neutron Scattering in Materials Research: Diffraction, Phases, and Micromechanics

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Brent T. Fultz, California Institute of Technology, Department of Materials Science, Pasadena, CA 91125 USA; Michael Atzmon, University of Michigan, Department of Materials Science & Engineering, Ann Arbor, MI 48109 USA

Tuesday AM Room: 3022
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Brent Fultz, California Institute of Technology, Pasadena, CA 91125 USA; Michael Atzmon, University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109-2136 USA

8:30 AM Welcome and Introduction: Brent Fultz and Michael Atzmon

8:35 AM Invited
Neutron Diffraction in Engineering Research: *Ersan Ustundag*[1]; [1]Iowa State University/Ames Laboratory, MSE Dept., 2220 Hoover Hall, Ames, IA 50011 USA

Neutron powder diffraction offers unique opportunities in engineering research by allowing in-situ studies of material deformation. In addition to phase information, one can also collect data on texture and lattice strain as a function of stress, temperature and sample environment. The diffraction data are then complemented with micromechanics modeling for full interpretation and to obtain the in-situ constitutive behavior of the material. The latter is very difficult to deduce from ex-situ tests and is crucial for predicting the long term performance in service. The recent construction of dedicated engineering diffractometers such as SMARTS and ENGIN-X has elevated the engineering neutron diffraction field to a new level of sophistication. Stresses exceeding 3 GPa, temperatures above 1500°C and numerous environments are now accessible. This presentation will describe recent work on bulk metallic glass composites, structural ceramics and metal matrix composites. It will also offer insight into exciting future developments.

9:05 AM Invited
Neutron Diffraction Studies of Mechanical Behavior: *Xun-Li Wang*[1]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, Bldg. 8600, Oak Ridge, TN 37831 USA

Since the 1980's, neutron diffraction has been used to study the mechanical behavior of materials. Much of the research in the early days was strongly oriented towards mechanical engineering, involving the determination of residual stress distribution in components and the use of these data in design and life-time predictions. Opportunities for fundamental research began to emerge when it became evident that some of the experimental data could not be understood within the framework of continuum theory and simple thermal-mechanical simulations. I will use recent experiments to illustrate what today's instruments are capable of, and their limitations. Examples will include fatigue behavior, deformation in nano-structured materials, and annealing-induced cracking in intermetallic composites. A number of new instruments are being built world-wide, increasingly adding new capabilities. The VULCAN diffractometer at the SNS is a new generation of diffractometers dedicated for users in materials science and engineering communities. Scientific opportunities with VULCAN will be discussed. This research was supported by U.S. Department of Energy, Basic Energy Sciences, Division of Materials Science and Engineering, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:35 AM
Finite Element Analysis for a Distributed Neutron Scattering Data Analysis Framework: *Cahit Can Aydiner*[1]; Ersan Ustundag[1]; [1]Iowa State University, Matls. Sci. & Engrg., 2220 Hoover Hall, Ames, IA 50011-2300 USA

Integration of data analysis and simulation tools used in neutron diffraction (ND) experiment evaluation offers unique opportunities in ND research. In this study, a scheme for integrating finite element analysis (FEA) to accompany engineering ND experiments is presented using the ABAQUS package. This scheme relies on modularizing the three stages of FEA: preprocessing (model definition), simulation and postprocessing. For the former, most commonly encountered samples and loading procedures in engineering ND experiments are used to form a model library. On the other hand, the advanced user is allowed to integrate a custom model into the framework. A similar module library and extension capability is provided for postprocessing. In addition, a user-defined subset of the model parameters are linked to a generic inverse solver that optimizes these parameters with the experimental output. This approach will be presented in conjunction with a new effort to perform distributed analysis of ND experiments.

10:05 AM Break

10:25 AM
Low Temperature Transformation of NiAlM Alloys: *Ling Yang*[1]; Xun-Li Wang[2]; Chain T. Liu[3]; Jaime A. Fernandez-Bacad[4]; James W. Richardson[5]; [1]University of Cincinnati, Cheml. & Matls. Sci., Cincinnati, OH USA; [2]Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN USA; [4]Oak Ridge National Laboratory, Condensed Matter Scis. Div., Oak Ridge, TN USA; [5]Argonne National Laboratory, Intense Pulsed Neutron Source, Argonne, IL USA

A systematic study of NiAlM (M=Ni, Fe, Co) alloys is carried out using neutron diffraction. The result shows that when Al atoms are partially replaced by transitional metal atoms ($Ni_{60-x}M_xAl_{40}$), there is a phase transition at very low temperature(~20K), while alloys with transitional metal atoms at Ni sites ($Ni_{50-x}M_xAl_{50}$) maintain a stable B2 structure down to 10K. Clearly, the addition of transitional metal atoms (Ni+M) at Al sites destabilizes the B2 structure and the transition depends on the fraction of (Ni+M) on Al sites. However, the observed transition does not seem to be related to magnetism, and the new phase does not match any of the known martensite. Instead, we found that the new peaks can be well indexed with a slightly distorted double-lattice superstructure. These observations suggest that the new phase is formed by re-ordering of the local atoms, which is fascinating to occur at such low temperatures.

10:45 AM
Evolution of Nanocluster, Dislocation and Grain Structure in Nanostructured Ferritic Alloy MA957 at Elevated Temperatures: *Matthew J. Alinger*[1]; G. Robert Odette[1]; Hirotatsu Kishimoto[2]; [1]University of California, Dept. of Matls., Santa Barbara, CA 93106 USA; [2]Kyoto University, Inst. of Advd. Energy, Gokasho, Uji, Kyoto 611-0011 Japan

So-called Nanostructured Ferritic Alloys (NFAs) containing 12-14Cr and a high density of nanoscale clusters (NCs) of Y-Ti-O exhibit superior creep strength and potential for high resistance to radiation damage. The coarsening-oxide transformation kinetics of the nm-scale precipitates in MA957 were characterized for anneals for various combinations of times from 1/3 to 480h and temperatures from 1150 to 1400°C. Small angle neutron scattering (SANS) was used to quantify the NC evolution, and TEM was used to observe the corresponding changes in the dislocation and grain structure and the formation of oxide phases. The NC coarsening and transformations can be approximately described by a pipe diffusion-type kinetics mechanism, with a time exponent of ≅ 1/5 and a high effective activation energy of ≅ 660 kJ/mole. This very high activation energy is believed to be due to the very low solubility of Y.

11:05 AM
A Neutron Diffraction Study of Phase Transformation by Tracking Texture Evolution with Temperature in Ti-6Al-4V: *Dhriti Bhattacharyya*[1]; G. B. Viswanathan[1]; S. C. Vogel[2]; D. J. Williams[2]; V. Venkatesh[3]; H. L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Los Alamos National Laboratory, LANSCE, Los Alamos, NM USA; [3]TIMET, R&D, 8000 W. Lake Mead Dr., Henderson, NV 89015 USA

The two phase (alpha/beta) Ti alloy Ti-6Al-4V is considered to be a workhorse alloy for its many applications in these diverse fields. One of the major aspects of this alloy which is not yet well understood is, whether the beta that forms at high temperatures is nucleated freshly from the alpha phase or whether it grows from the beta preexisting at room temperature. Another important, and as yet unresolved, question is whether the alpha phase maintains the Burgers Orientation Relationship (BOR) with the beta phase even after cold work and recrystallization. A unique way to address these issues is to monitor the development of texture in this microstructure as a function of temperature. The present study attempts to do this by carrying out, for the first time, high temperature in-situ texture measurements of TI-6Al-4V using the HIPPO instrument at LANSCE. This instrument enables texture measurements by neutron diffraction at various temperatures with the sample located inside a vacuum furnace. The textures of the alpha and the beta phases measured at room temperature, 800°C and 1020°C provided fresh evidence which indicate definite answers to the questions posed above.

11:25 AM
Orientation Distributions of Diamond Particles in Polycrystalline Diamond Synthetic Cells: *Jun-Yun Kang*[1]; Suk Hoon Kang[1]; Kyu Hwan Oh[1]; Hu-Chul Lee[1]; [1]Seoul National University, Sch. of Matls. Sci. & Engrg., Shinrim 9-dong, Kwanak-gu, Seoul 151-744 Korea

The orientation distributions of diamonds in two types of synthetic cells were examined. From the orientation distribution function (ODF) calculated from EBSD data, it was concluded that the diamonds in layered cell exhibited weak texture of <111> parallel to the stacking direction. And from the neutron diffraction spectrum of powdered cell, the texture indices of diamond {111}, {220} and {311} planes were calculated as 1.18, 0.89 and 0.90, respectively, which implied that the orientation distribution of the diamonds approached random distribution. In layered cell, there exists concentration gradient of carbon around growing diamonds along the stacking direction. By contrast, in powdered cell, diamonds are surrounded by homogeneous carbon solution. This discrepancy was considered to lead to the different orientation distributions of diamonds in the two cells. The favored alignment of {111} planes perpendicular to the stacking direction in layered cell was thought to originate from their lowest surface energy.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Aging, Crystallographic Texturing and Characterization of Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee

Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Katsuaki Suganuma, Osaka University, Department of Nanomaterials and Environmental Conscious Technology, Ibaraki, Osaka 567-0047 Japan

Tuesday AM Room: 3016
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Iver E. Anderson, Ames Laboratory/Iowa State University, Matls. & Engrg. Physics, Ames, IA 50011 USA; Lawrence P. Lehman, Binghamton University, Dept. of Physics, Binghamton, NY 13902 USA

8:30 AM Invited
Void Suppression in Thermal Aging of Tin-Silver-Copper-X Solder Joints: *Iver Eric Anderson*[1]; Joel L. Harringa[1]; Sung K. Kang[2]; Bruce A. Cook[1]; [1]Ames Laboratory (USDOE), Iowa State University, Matls. & Engrg. Physics, 222 Metals Dvlp. Bldg., Ames, IA 50011 USA; [2]IBM T. J. Watson Research Center, Microelect. Packing Tech., 1101 Kitchawan Rd., Rte. 134, PO Box 218, Yorktown Hgts., NY 10598 USA

Recent work demonstrated the strategy of modifying a strong (high Cu) Sn-Ag-Cu (SAC) solder alloy with a substitutional alloy addition (X=Co, Fe) for Cu to retain solder joints with strength and ductility after aging at 150°C for up to 1000 h. This is critical to Pb-free assembly of portable electronics with potential for drop impact failure of interconnects. The SAC + X solder alloy joints appear to suppress the interdiffusion of Sn and Cu through Sn-Cu intermetallic layers to inhibit the formation and linkage of voids at the Cu substrate/Cu3Sn interface and, thus, to prevent embrittlement on aging. Other recent work identified Cu as the fast diffusing species that leads to voiding/embrittlement, albeit at lower aging temperatures. Void distributions in the Cu/solder interface region were analyzed on aging (at 150°C) in SAC and SAC + X solder joints to gain understanding of the limiting diffusion species and mechanism. Supported by ISU Research Foundation and USDOE-BES (W-7405-Eng-82).

9:00 AM
Microstructure and Shear Strength Evolution of Sn-Ag-Cu Solder Bumps During Aging at Different Temperatures: Dezhi Li[1]; *Changqing Liu*[1]; Paul P. Conway[1]; [1]Loughborough University, Wolfson Sch. of Mechl. & Mfg. Engrg., Loughborough, Leicestershire LE11 3TU UK

Flip chip devices with Sn-3.8Ag-0.7Cu solder on electroless Ni UBM were studied after aging at both 80°C and 150°C. The shear strength of the solder bumps was tested and the microstructure evolution was studied by SEM with EDX and EBSD technique. When the chips were aged at 80°C, a (Cu,Ni)6Sn5 IMC layer grew very slowly during the aging and the increase of the IMC thickness was not significant. Also, no Kirkendall voids were found under SEM examination. However, when the chips were aged at 150°C, the increase of the IMC thickness became very clear. Some Kirkendall voids appeared even after the chips were aged at 150°C for only 2 days. During the aging, the (Cu,Ni)6Sn5 IMC changed from a scallop-like formation to a faceted shape. The shear tests showed that the shear strength of solder bumps dropped at some point during the aging and then almost kept at a consistent value during the later aging periods. All the solder bumps were found to fracture in the bulk solder.

9:20 AM
Microstructure Evolution of Gold-Tin Eutectic Solder on Cu and Ni Substrates: *Jui-Yun Tsai*[1]; C. W. Chang[1]; C. E. Ho[1]; Y. L. Lin[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matl. Engrg., No 300, Jung-da Rd., Chungli City Taiwan 320

The microstructures of Au20Sn/Ni and Au20Sn/Cu that had been reacted in different bonding conditions first, and then aged were studied. An Sn/Au/Ni sandwich structure (2.5/3.75/2 μm) and an Sn/Au/Ni sandwich structure (1.83/2.74/5.8 μm) were deposited over the Si wafer, respectively. The overall composition of the Au and Sn layers corresponded to the Au20Sn binary eutectic (wt.%). The microstructures of Au20Sn solders on Ni and Cu were controlled by the bonding conditions. When the reaction condition was 290°C for 2 min, the microstructure of and Au20Sn/Ni and Au20Sn/Cu was both a two-phase (Au5Sn and AuSn) eutectic microstructure. The difference between Au20Sn/Cu and Au20Sn/Ni is that (Au, Ni)Sn formed next to Ni and (Au, Cu)5Sn formed next to Cu. When the bonding condition was at 240°C for 2 min, an layered microstructure was produced (AuSn/Au5Sn/Ni and AuSn/Au5Sn/Cu). The thermal stability of the as-bonded samples were also studied. Many interesting microstructure evolutions were observed. In general, the thermal stability for samples on Ni was better than that of on Cu when the bonded samples were subjected to long-term aging at 240°C.

9:40 AM
Interfacial Reaction Between 42Sn/58Bi Solder and Electoless Ni-P/Immersion Au UBM During Thermal Aging: *Moon Gi Cho*[1]; Kyung Wook Paik[1]; Hyuck Mo Lee[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Engrg., Kusung-Dong 373-1, Yusung-Gu, Taejon 305-701 Korea

The interfacial reaction between the 42Sn/58Bi solder and the electoless Ni-P/immersion Au UBM has been investigated before and after thermal aging, with a focus on formation of intermetallic compound (IMC), interfacial reaction rate, and bump shear strength. The immersion Au layer with a thickness of 0 (bare Ni), 0.1 and 1 μm was plated on the electroless Ni-5~6at.%P (6 μm) layer. Then, 42Sn/58Bi solder balls were fabricated on UBM by screen-printing and reflow. The IMC layer composed of Ni3Sn4 grains was formed at the join interface after reflow. On aging at 125°C, a ternary IMC phase was observed on the Ni3Sn4 layer in case of Au plating, which was identified as (Au,Ni)Sn4. Its thickness reached 7 μm and higher after aging for 1000 hr, and the Ni-P UBM was consumed by more than 1.2 μm. The thick (Au, Ni)Sn4 IMC layer deteriorated the integrity of the solder joint and thus the shear strength of the solder bump decreased by about 30% compared with non-aged joints.

10:00 AM Invited
Formation and Growth of IMC During Thermomechanical Fatigue of Sn-Based Solders: J. G. Lee[1]; *K. N. Subramanian*[1]; [1]Michigan State University, Dept. of Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48824 USA

Sn-based solder joints exhibit significant aging from thermomechanical fatigue (TMF) treatments with longer dwell times at elevated temperature extreme. In the TMF cycles employed in this study the total accrued aging time at the high temperature extreme of 150°C could be about 2000 hours for about 1000 TMF cycles. Formation and growth of IMC phases in ternary and quaternary eutectic Sn-Ag based alloys with Cu and (Cu+Ni) additions due to such treatments, and their relative importance in the damage accumulation that results in deterioration of properties will be discussed. Work supported by the National Science Foundation under grant NSF DMR-0081796 and NSF DMI-0339898.

10:20 AM Break

10:30 AM
Distinct Reaction Morphologies of Two High Lead Solders, 90Pb10Sn and 95Pb5Sn, on Cu UBM During Solid-State Aging: Jin-Wook Jang[1]; Lakshmi N. Ramanathan[1]; Jong-Kai Lin[1]; *Darrel R. Frear*[1]; [1]Freescale Semiconductor, FMTC, 2100 E. Elliot Rd., MD EL725, Tempe, AZ 85284 USA

The solid-state reaction of two high lead solders, 90Pb10Sn and 95Pb5Sn, on Cu UBM were examined. Upon reflow, the Cu3Sn intermetallics formed on Cu UBM for both solder alloys, but solid-state aging produced significantly different reaction morphologies. For 90Pb10Sn solder, the Cu3Sn intermetallics continued to grow during solid-state aging at 170°C for 1500 hrs whereas 95Pb5Sn solder showed very limited intermetallic growth and the subsequent spalling of Cu3Sn from Cu UBM was observed. Such a difference is explained by a two-step mechanism: the Sn diffusion from bulk solder to the solder/Cu3Sn interface and the subsequent intermetallic formation by interdiffusion of Cu and Sn atoms. It was postulated that the competition between

these two kinetic factors leads to such a big morphological difference. The spalling phenomenon of Cu3Sn intermetallics in 95Pb5Sn solder was due to the loss of chemical adhesion between the Cu3Sn intermetallics and Cu UBM. Thermodynamic interpretation of spalling phenomenon showed that the interfacial free energy without spalling is greater than that with spalling after solid-state aging.

10:50 AM
Cyclic Twin Nucleation in Tin Based Alloys: *Lawrence P. Lehman*[1]; Yan Xing[1]; Ju Wang[1]; Kara Mather[1]; Thomas R. Bieler[2]; Adwait U. Telang[2]; Eric J. Cotts[1]; [1]Binghamton University, Dept. of Physics, Sci. II, PO Box 6000, Binghamton, NY 13902-6000 USA; [2]Michigan State University, Dept. Cheml. Engrg. & Matls. Sci., 2527 Engrg. Bldg., E. Lansing, MI 48824-1226 USA

Most Pb free solders are based on tin, with small additions of silver and copper. The mechanical and physical properties of these solders depend upon the slip and recovery/creep properties of the tin microstructure. The anisotropic nature of tin's mechanical properties means that the number and relative orientation of the tin grains in a solder joint will strongly influence its mechanical response. SnAgCu solder joints are often comprised of essentially one tin grain, or at most, a few dominant orientations. Twinning during Sn solidification is common, resulting in highly preferred, ~60° misorientations between neighboring Sn grains. We explore the source, crystallography and growth of cyclic twinned microstructures in tin based solders. Models of the geometry of twinning on different crystallographic planes can account for different growth morphologies, and different physical manifestations of cyclic twinning.

11:10 AM
Microtextural Analysis of Lead Free Solder Alloys: *Vineet Kumar*[1]; Zhigang Zak Fang[1]; Jin Liang[2]; Nader Dariavach[2]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA; [2]EMC2 Corp., 176 South St., Hopkinton, MA 01748 USA

There is a thrust to develop lead free solder alloys, having better or at least similar properties to conventional tin-lead solder alloys. Tin based eutectic and near-eutectic compositions similar to Sn-4Ag-1Cu are most promising alloys. However, there is still a lack of comprehensive understanding of the microstructure of these materials, hence their dependence on processing variable. In order to achieve best possible properties a comprehensive physical metallurgical analysis is required. In this direction, for alloy Sn-3.8Ag-0.7Cu, microstructural and microtextural analysis was done using Scanning Electron Microscope (SEM) and Electron Back-Scattered Diffraction (EBSD) technique. Composition and crystallographic details for different phases were measured and it was found that the intermetallics had a different composition from that of reported in literature. Orientation of different microstructural components was found. Misorientation relationship between different microstructural constituents including copper substrate was found. It is found that intermetallics have a definitive misorientation relationship with tin matrix and copper substrate.

11:30 AM
Effect of Cu on Microstructure and Grain Boundary Character in Sn-3.0Ag-Cu Solder Balls Solidified at 1/s: Adwait U. Telang[1]; *Thomas R. Bieler*[1]; Yan Xing[2]; Larry P. Lehman[2]; Eric J. Cotts[2]; [1]Michigan State University, Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48824-1226 USA; [2]Binghamton University, Physics, Matls. Sci. Prog., Binghamton, NY 13902 USA

The solder alloy composition and cooling rate is known to have a large effect on microstructure of lead free solder joints. However, the mechanisms by which alloy composition influence solidification microstructures are unknown. In this study, 1 mm diameter solder balls having 3.0 wt% Ag with Cu levels of 0, 0.27, 0.59, 0.86, 1.1, and 1.4 wt% Cu were remelted and solidified at a cooling rate of 1/s, and examined in cross polarized light microscopy. With increasing Cu content, the size of the crystalline domains increased from about 50 microns with no Cu, to about 250 microns with 1.4 Cu. Using orientation imaging microscopy, grain orientation maps and misorientation statistics will be presented to identify the grain boundary character, and from this, the effect of Cu on possible solidification mechanisms will be discussed.

11:50 AM
Novel Characterizing Technique in Phase Distribution with Sample Preparation for Application in Solder Joint, Wire Bonding Chip and Li-Ion Battery Assembly: *Hung-Kai Chen*[1]; Shih-Hai Li[1]; *Jenq-Gong Duh*[2]; [1]National Tsing Hua University, Dept. of Engrg. & Sys. Sci., 101 Sec.2 Kunag-Fu Rd., Hsinchu 300 Taiwan; [2]National Tsing Hua University, Dept. of Matl. Sci. & Engrg., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan

Characterization of solder joint microstructure is essential in the evaluation of microelectronic packaging reliability. Analyzing technique and sample preparation play important roles in the material characterization. This study demonstrates several novel approaches in the microstructure evaluation of solder joint. By utilizing the precision etching and coating technique, the interface of the IC packaging chip between the solder and under bump metallization (UBM) would be observed much more precisely and the intermetallic compounds could be revealed in details under field-emission scanning electron microscope (FE-SEM). For the interface of Sn-Pb/Ni/Cu, the intermetallic compound, defined as Ni_3Sn_4 and Cu_6Sn_5 by electron probe microanalyzer (EPMA), would form between the nickel and the solder. By adjusting the operating variables, such as etching energy, etching period, incident angle between the ion beam and sample, the time for sample preparation was significant reduced. For an example, when the cross-section surface of Sn-Pb solder specimen was perpendicular to the incident ion beam, the best quality of the SEM micrographs could be obtained either at 2.5 keV with an etching time for 7 min or at 4.5 keV for 3 min. In addition, the diffusion phenomenon of zinc in ZnO-coated $LiCoO_2$ powder and the redistribution of boron in LiBO-coated $LiMn_2O_4$ powder could be clearly revealed and evidenced by field-emission electron probe microanalyzer (FE-EPMA) with special sample preparation technology.

TUESDAY AM

Phase Transformations Within Small-Size Systems: Phase Separation, Precipitation and Displacive Transformations

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee, EMPMD-Nanomaterials Committee
Program Organizers: Vijay K. Vasudevan, University of Cincinnati, Department of Chemical and Materials Engineering, Cincinnati, OH 45221-0012 USA; Robert D. Shull, National Institute of Standards and Testing, Metallurgy Division, Gaithersburg, MD 20899-8552 USA; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375-5000 USA; Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA

Tuesday AM, February 15, 2005 — Room: 3002, Location: Moscone West Convention Center

Session Chairs: David N. Seidman, Northwestern University, Matls. Sci. & Engrg., Evanston, IL 15229-3180 USA; Vijay K. Vasudevan, University of Cincinnati, Cheml. & Matls. Engrg., Cincinnati, OH 45221-0012 USA

8:30 AM Invited
Effect of Surface Stress on the Coherent Phase Equilibria of Nanoparticles: *Joo-Youl Huh*[1]; William C. Johnson[2]; James M. Howe[2]; [1]Korea University, Div. of Matls. Sci. & Engrg., 5-1, Anam-Dong, Sungbuk-Ku, Seoul 136-701 Korea; [2]University of Virginia, Dept. of Matls. Sci. & Engrg., PO Box 400745, Charlottesville, VA 22904-4745 USA

As motivated by the recent observations on the stabilization of an ordered phase ($L1_2$) in Ag-Cu-Ag nanoparticles at temperatures more than 250°C above the bulk ordering temperature, we investigated the influence of particle size on coherent phase equilibria of a binary, spherical particle by considering the alteration of elastic fields caused by surface stress. The coexisting phases in a spherical particle are assumed to be configured as concentric shells. In the stress-free state, the binary system exhibits a miscibility gap ($\alpha_1 + \alpha_2$) at high temperatures and a eutectoid transition to form an ordered phase ($L1_2$) at a low temperature. Phase diagram constructions show that, depending on the particle size and materials parameters, the interaction between surface stress and misfit strains can stabilize either the $\alpha_1 + \alpha_2$ two-phase state by suppressing completely the $L1_2$ phase formation or the $\alpha_1 + L1_2$ two-phase state until it transforms to a single-phase solid solution at high temperatures. This talk will also discuss possible effects of compositional strains on the phase stability of nanoparticles.

9:05 AM
Phase Transformations Within Nanoparticles of Aluminum Alloys: Jixiong Han[1]; Martin J. Pluth[1]; Kazuo Furuya[2]; Jainagesh A. Sekhar[1]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Cheml. & Matls. Engrg., 401 Rhodes Hall, ML 0012, Cincinnati, OH

45221-0012 USA; [2]National Institute for Materials Science, Nanocharacterization Lab., 3-13 Sakura, Tsukuba, Ibaraki 305-0003 Japan

Phase transformations within nanoparticles of Al alloys were studied. Nanoparticles of binary Al-Cu and Al-Zn alloys were synthesized by plasma ablation of precursor ingots and the structure of these particles as well structural changes in these on aging at temperatures between 65-190°C for times to 100h studied by electron diffraction, nanoprobe energy dispersive x-ray spectroscopy and HRTEM. The particles were supersaturated fcc state in both cases, but displayed a variation in the individual particle composition when compared with the precursor bulk alloys. A 3-5 nm thick oxide layer was present around all the particles. On aging the Al-Cu nanoparticles, a precipitation sequence consisting of nearly pure Cu precipitates to θ' to the equilibrium θ was observed, with all three forming only along the outer oxide-particle interior interface. The structure of θ' and its interface with the Al matrix was characterized in detail. In the Al-Zn alloy, a spinodal structure was noted in the as-synthesized nanoparticle, which coarsened on aging into a fine scale structure composed of f.c.c twin-related platelets within which were contained platelets with a hcp structure. This morphology led to relatively complicated diffraction effects, which were analyzed in detail. Nearly-pure Zn precipitates, with an hcp structure, also formed along the oxide-particle interface and consumed the spinodal structure with time. Details of the precipitation sequence, nature and structure of second phase precipitates and interphase interfaces and formation mechanisms will be reported. In addition, the synthesis of and precipitation behavior in ultrafine (5-25 nm) Al-Cu nanoparticles will be presented. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

9:30 AM
Microstructure and Mechanical Behavior of Fe-20Ni-25Mn-25Al: *Ian Baker*[1]; Markus W. Wittmann[1]; James Hanna[1]; Paul R. Munroe[2]; [1]Dartmouth College, Thayer Sch. of Engrg., 8000 Cummings Hall, Hanover, NH 03755 USA; [2]University of New South Wales, Elecron Microscope Unit, Sydney, NSW 2052 Australia

An alloy of composition (in atomic percent) Fe-20Ni-25Mn-25Al was cast and its microstructure examined in both the as-cast condition and after anneals for one hour at various temperatures. The as-cast microstructure, which appears to form by spinodal decomposition, consists of alternating, coherent ~50nm wide B2 and b.c.c. plates aligned along <100>. The microstructure is remarkably resistant to coarsening below 1000 K, but precipitation of a f.c.c. phase occurs in the b.c.c. plates at higher temperatures. Mechanical testing revealed a yield strength of 1.3-1.5 MPa from 300-600 K and a strength greater than 200 MPa above 1073K. Room temperature hardness testing was also performed after various anneals. This presentation will attempt to correlate the observed microstructure with the mechanical properties, and will briefly discuss the effects of changes in composition on the microstructure and mechanical properties. Research supported by NIST grant 60NANB200120.

9:55 AM Break

10:10 AM Invited
The Chemical Evolution of Gamma Prime Precipitates in Ni-Al-Cr Base Alloys on a Subnanoscale to Nanoscale: *David N. Seidman*[1]; Chantal K. Sudbrack[1]; Kevin E. Yoon[1]; Ronald D. Noebe[2]; [1]Northwestern University, Matls. Sci. & Engrg., Cook Hall, 2220 Campus Dr., Evanston, IL 60208-3108 USA; [2]NASA, MS 49-3, Glenn Rsch. Ctr., 21000 Brookpark Rd., Cleveland, OH 44135 USA

The temporal evolution of the gamma (FCC) and gamma prime (L12 structure)phases in Ni-Al-Cr alloys, with additions of W or Re is followed on a sub- to nanoscale employing three-dimensional atom-probe (3DAP)microscopy. The emphasis in this presentation is on the chemistry within nanoscale gamma prime precipitates. It is demonstrated experimentally that, for example, in a ternary Ni-Al-Cr alloy the compostion of the gamma prime precipitates evolve temporally and they do not follow the compositions given by tie line, whereas in contrast the compostion of the gamma matrix follows the tie line. This is consistent with the Kuehmann-Voorhees model of coarsening in a ternary alloy. Additionally, 3DAP microscope evidence is presented, which shows the existence of concentration gradients within the gamma prime precipitates that evolve toward their equilbrium values. Thereby proving that transient composition profiles exist before the quasi-steady state profiles are achieved.

10:45 AM
Application of the Cluster/Site Approximation to the Calculation of Coherent Interphase Boundary Energy: *W. W. Cao*[1]; J. Zhu[1]; F. Zhang[2]; W. A. Oates[3]; M. D. Asta[4]; Y. A. Chang[1]; [1]University of Wisconsin, Dept. of Matl. Sci. & Engrg., Madison, WI 53706 USA; [2]CompuTherm LLC, 437 S. Yellowstone Dr., Madison, WI 53719 USA; [3]University of Salford, Inst. for Matls. Rsch., Salford M5 4WT UK; [4]Northwestern University, Dept. of Matl. Sci. & Engrg., Evanston, IL 60208 USA

The coherent interphase boundary (IPB) energies are calculated as a function of temperature thermodynamically using the Cluster/Site Approximation (CSA) for binary fcc-based alloy systems. CSA calculated results are compared with those obtained from the tetrahedron-octahedron approximation of the Cluster Variation Method (TO-CVM). We have demonstrated that the CSA offers comparable accuracy as the TO-CVM in calculating the IPB energies. Moreover, the CSA-calculated IPB energies for (Al)/Al_3Li in Al-Li and γ / γ' in Ni-Al are also in accord with experimental data. One advantage of CSA is that it is computational less demanding.

11:10 AM
Differential Role of Nanoscaled Oxide Dispersoids (Y2O3 vs Al2O3) in the High Temperature Structural Stability of NiCr Alloys: *Dheepa Srinivasan*[1]; P. R. Subramanian[2]; Reed R. Corderman[2]; [1]GE India Technology Centre, Matls. Rsch. Lab., EPIP-II, Whitefield Rd., Bangalore, Karnataka 560 066 India; [2]GE Global Research, Ceram. & Metall. Tech., PO Box 8, K1-MB265, Schenectady, NY 12301 USA

Microstructural stability studies were conducted on nanoscale yttria and alumina-reinforced NiCr alloys that were fabricated by electron-beam physical vapor deposition (EB-PVD). The nanoscale yttria dispersoids were far more effective in contributing to the structural stability of the NiCr alloys as compared to the alumina particles. The fine yttria particles share a coherent interface with the NiCr matrix and exert a Zener pinning force on the matrix grains to inhibit rapid grain growth at elevated temperatures. Coarsening of yttria takes place by diffusion of the metallic species through the NiCr matrix, whereas the nano sized alumina particles undergo a hierarchy of phase transformations (gamma-Al2O3 to delta-Al2O3) leading to rapid coarsening. Phase selection at the nanoscale has been examined on the basis of free energies of formation of the metastable vs stable phases at the respective size ranges. The thermal stability of these alloys, as mapped via microstructural examination, has been correlated to room temperature hardness. Further, the different mechanisms responsible for strengthening (Hall-Petch, Orowan and solid solution strengthening) have been examined. The impact due to the most dominant mechanism is brought out for the two types of dispersoids.

11:35 AM
Observations of Reverse Martensitic Transformations in NC Pearlitic Steel: *Yu. Ivanisenko*[1]; I. MacLaren[2]; R. Z. Valiev[3]; H.-J. Fecht[1]; [1]Forschungszentrum Karlsruhe, Inst. für Nanotech., Karlsruhe 76021 Germany; [2]University of Glasgow, Dept. of Physics & Astron., Glasgow G12 8QQ UK; [3]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., Ufa 450000 Russia

Unusual phase transformations in nanocrystalline (NC) materials, such as extension of solid solubility and alteration of phase boundaries, etc. may be related with the shift of thermodynamic equilibrium in a small system due to strong increase of interfacial energy and interfacial stresses. At the same time an important part can play also kinetic factors during preparation of NC structure, such as cooling rate, or high stresses and dislocation density at severe deformation. Here we report about observations of a shear ferrite-austenite transformation taking place at severe deformation of NC pearlitic steel, something which never occurs in conventional deformation of coarse-grained iron and steels. Orientation relationship between parent ferrite and new austenite are either Kurdjumov-Sachs or possibly Nishiyama-Wassermann, i.e. the same as those observed for temperature-driven martensitic transformations in Fe and steels. A mechanism of reverse martensitic transformation is discussed in terms of sliding of transforming partials 1/6<1-10>(110) in bcc lattice.

Shape Casting — The John Campbell Symposium: Solidification

Sponsored by: Light Metals Division, LMD-Aluminum Committee, MPMD-Solidification Committee
Program Organizers: Murat Tiryakioglu, Robert Morris University, Moon Township, PA 15108 USA; Paul N. Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday AM Room: 2008
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Srinath Viswanathan, Sandia National Laboratory, Albuquerque, NM 87185-1134 USA

8:30 AM
The Role of the Eutectic in Porosity Formation in Al-Si Foundry Alloys: *Arne K. Dahle*[1]; Stuart D. McDonald[2]; Liming Lu[1]; Kazuhiro Nogita[2]; [1]University of Queensland, CRC for Cast Metals Mfg., Matls. Engrg., Brisbane, Qld 4072 Australia; [2]University of Queensland, Matls. Engrg., Brisbane, Qld 4072 Australia

It is now well-established that three different eutectic solidification patterns can operate in Al-Si foundry alloys. Furthermore, the selection of each solidification mechanism can be manipulated through additions of typical modifier elements, such as Sr and Na, but also less common elements. The spatial distribution of evolving eutectic is very different for each eutectic solidification pattern, therefore resulting in large differences in permeability in the last stages of solidification of the castings. This paper presents a coherent approach to solidification mechanisms in Al-Si alloys and the relationship to the porosity formation in this important alloy system.

8:55 AM
Mechanism of Eutectic Solidification of Aluminum-Silicon Alloys: *Sumanth Shankar*[2]; *Makhlouf Makhlouf*[1]; [1]Metal Processing Institute - Worcester Polytechnic Institute, Matls. Sci. & Engrg., 100 Inst. Rd., Worcester, MA 01609 USA; [2]McMaster University, Mechl. Engrg., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canda

A mechanism is presented to explain the formation of the eutectic phases in Al-Si hypoeutectic alloys. The mechanism is supported with results of non-equilibrium thermal analyses, and microstructure evidence obtained from optical, scanning and transmission electron microscopy, as well as selected area electron diffraction analyses and elemental x-ray mapping, in addition to results of high temperature rheological measurements performed on Al-Si alloy samples of precisely controlled chemistry.

9:20 AM
Chemical Modification of the Morphology of the Eutectic Phases in Hypoeutectic Aluminum-Silicon Alloys: Sumanth Shankar[1]; *Makhlouf Makhlouf*[2]; [1]McMaster University, Mechl. Engrg., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada; [2]Metal Processing Institute - Worcester Polytechnic Institute, Matls. Sci. & Engrg., 100 Inst. Rd., Worcester, MA 01609 USA

A mechanism is presented to explain the chemical modification of the morphology of the eutectic phases in Al-Si hypoeutectic alloys. The mechanism is supported with results of non-equilibrium thermal analyses, and microstructure evidence obtained from optical, scanning and transmission electron microscopy, as well as selected area electron diffraction analyses and elemental x-ray mapping, in addition to results of high temperature rheological measurements performed on Al-Si alloy samples of precisely controlled chemistry.

9:45 AM
An Overview of Heterogeneous Nucleation Mechanisms in Al Alloys: *Brian John McKay*[1]; Peter Schumacher[1]; [1]University of Leoben, Lehrstuhl für Gießereikunde, Franz-Josef Str. 18, Leoben, Styria Austria

The benefits of grain refining wrought and foundry Al alloys are widely acknowledged. To induce grain refinement, TiBAl and TiCAl master alloys are commonly added to the melt prior to casting. Understanding the nucleation mechanisms of these refiner particles is important with respect to optimizing current commercial casting practices and improving future refining potency. However, using conventional solidification casting techniques, heterogeneous nucleation is difficult to investigate as α-Al grain growth obscures the nucleation sites. A metallic glass technique has been successfully applied allowing the nucleation mechanisms to be examined using Transmission Electron Microscopy (TEM). The technique freezes the crystalline growth of α-Al at an early stage in a glass and allows the study of the orientation relationship between nucleant and nuclei using diffraction and chemical analysis. The effects of Si and Zr which at higher concentrations can have a detrimental effect on the ultimate grain size (UGS) achieved by the grain refiner addition were examined. It has been found that the chemical composition of the refiner particles and their interfaces play an important role in the nucleation mechanism, which has implications for current casting practices.

10:10 AM Break

10:20 AM
Nucleation of Primary Al_5FeSi in an Al-11.6Si-1.5Fe-0.37Mg-0.29Mn Alloy: *David N. Miller*[1]; Liming Lu[1]; Arne K. Dahle[1]; Graham B. Schaffer[1]; [1]University of Queensland, Div. of Matls. Engrg., Brisbane, Queensland 4072 Australia

Recent research published by Cao and Campbell[1] has provided strong evidence to support the theory that the iron containing 'sludge' phases, Al_5FeSi and $Al_{15}(FeMn)_3Si_2$, nucleate on oxide films entrained in aluminium casting alloys. This is evidenced by the presence of crack-like defects within these iron intermetallics. In an attempt to verify the role of oxide in iron intermetallic formation, experiments have been conducted under conditions of low melt agitation and significant agitation to alter the oxide levels of cast samples with primary Al_5FeSi formation. The results are in general agreement with the theory of Cao and Campbell where the crack-like defects are only evident in Al_5FeSi particles from alloys that are agitated during solidification, ie. conditions that lead to the likely entrainment of oxide films. More detailed electron microscopy studies have been carried out into the nature of the oxide-Al_5FeSi system. [1]Cao, X. and J. Campbell, Metallurgical and Materials Transactions, 2003. 34A(7): p. 1409-1420.

10:45 AM
Observation of Hot Tearing Crack Propagation: Cameron Davidson[2]; *David Michael Viano*[1]; Liming Lu[1]; David StJohn[3]; [1]University of Queensland, Div. of Matls., Sch. of Engrg., St. Lucia, Queensland 4072 Australia; [2]CSIRO Manufacturing & Infrastructure Technology, PO Box 883, Kenmore 4069 Australia; [3]University of Queensland, CRC for Cast Metals Mfg., UDP No. 055, Brisbane, QLD 4072 Australia

Hot tears are generally thought to occur at high solid fractions between 0.95 and 0.99. While the general mechanisms of hot tearing are understood, i.e. the inability of liquid to feed strain imposed on the mushy material, work continues on improving the understanding of the mechanisms at play. A hot tear test rig that measures the temperature and load imposed on the mushy zone during solidification has been successfully used to study hot tearing in Al-Cu alloys. The mould has been modified to incorporate a window above the hot spot region allowing visual observation of hot tear formation and growth. Combining information from visual observation with load and temperature data has led to a better understanding of the mechanisms of hot tearing. It was found that hot tearing started at extremely low loads. The development of load with time was surprisingly insensitive to the presence, or not, of a crack.

11:10 AM
Prediction Hot Tearing Tendency for Multicomponent Aluminum Alloys: *Xinyan Yan*[1]; Jen C. Li[1]; [1]Alcoa, Alcoa Techl. Ctr., 100 Technical Dr., Alcoa Ctr., PA 15069 USA

It is of practical importance to be able to predict the hot tearing tendency for multicomponent aluminum alloys, as hot tearing is one of the most common and serious defects occurred during casting of commercial aluminum alloys that are essentially all multicomponent systems. For many years, the main criterion applied to characterize the hot tearing tendency of an alloy system was based on solidification interval. However, this criterion cannot explain the susceptibility-composition relation between the limits of the pure base metal and the eutectic composition. Clyne and Davies [T. Clyne and G. Davies, Brit. Found., 74(1981), 65] correlated the susceptibility/composition relationship in binary systems based on the concept of the existence of critical time periods during the solidification process when the structure is most vulnerable to cracking. The Scheil equation was used in their model with constant partition coefficient and constant liquidus slope estimated from the phase diagram. In the present study, we followed Clyne and Davies' general idea, and directly coupled the Scheil solidification simulation with phase diagram calculation via PanEngine, a multicomponent phase equilibria calculation interface, and extended the model to higher order systems. Experimental results of hot tearing tests on some ternary and quaternary systems will be presented. The predicted hot tearing tendencies correlated very well with the experimental results of multicomponent aluminum alloys.

11:35 AM
Influence of Temperature and Alloying Elements on Fluidity of Al-Si Alloys: *Marisa Di Sabatino*[1]; Sumanth Shankar[2]; Diran Apelian[3]; Lars Arnberg[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., A.Getz vei, 2B, Trondheim 7491 Norway; [2]McMaster University, Dept. of Mechl. Engrg., Hamilton, Ontario L8S 4L7 Canada; [3]Worcester Polytechnic Institute, Metal Procg. Inst., Worcester, MA 01609 USA

The goal of the work is to study the influence of casting temperature and four alloying elements: Mg, Ti, Fe and Sr, on fluidity of Al-7wt.% Si alloys. Fluidity of the alloys was measured using a fluidity mould produced by N-Tec Ltd., U.K. The experiments were designed using three orthogonal L8 Taguchi matrices. Each of the four alloying elements and the casting temperature was an independent variable with two levels. Three interactions between the variables were identified and analyzed. The two levels of Mg were 0.03wt.% and 0.45 wt.%; Ti levels were 0 and 0.2wt.%; Sr at 0 and 0.023wt.%; and Fe levels were 0.06wt.% and 0.24wt.%. Superheats were 70°C and 130°C over the respective liquidus temperatures of the experimental alloys. The main effect of each of the independent variables on the fluidity was quantified and ANalysis Of VAriance (ANOVA) was performed on the experiment matrix. The results were verified and validated to ensure robustness of the experiment design. In addition to the Taguchi design of experiments, fluidity evaluations were carried out on five melt systems: pure Al, Al-17wt.%Si, 356, 390 and 520. The results of the Taguchi design of experiments show that casting temperature had the most pronounced influence on fluidity of the molten metal. Among the alloying elements chosen, only Mg had an appreciable effect on fluidity. Increasing Mg in the melt from 0 to 0.45wt.% showed a decrease in fluidity of the molten metal. The results of the fluidity experiments on the additional five melt systems show that silicon has a significant effect on fluidity. The variation of fluidity among families of Al-Si alloys is more pronounced than the variation within a particular family of alloy such as 356, which implies that minor changes in composition within a family of Al-Si alloy does not influence fluidity significantly.

Superalloys and Coatings for High Temperature Applications: Oxidation Behaviour - I

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), High Temperature Materials Committee of IoM3

Program Organizers: Roger C. Reed, University of British Columbia, Department of Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; John Nicholls, Cranfield University, Bedfordshire MK43 OAL UK; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Tuesday AM Room: Nob Hill A/B
February 15, 2005 Location: San Francisco Marriott

Session Chairs: Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; Roger C. Reed, University of British Columbia, Dept. of Metals & Matls. Engrg., Vancouver, BC V6T 1Z4 Canada

8:30 AM Invited
Desk Top TBC Spallation and Interfacial Hydrogen Embrittlement of Alumina Scales: *James L. Smialek*[1]; [1]NASA Glenn Research Center, Matls., 21000 Brookpark Rd., 106-1, Cleveland, OH 44135 USA

Plasma sprayed 8YSZ coatings were deposited on PWA 1484 single crystal superalloys, without bond coats, and oxidized in 1100°C furnace tests, using 1-hr and 100-hr cycles. Depending on the alloy sulfur content, lives ranged from about 200 to 2000 hr. Often failure did not occur immediately upon cooldown but only after a considerable period at room temperature or upon water immersion. Also, Rene-N5 was oxidized at 1150°C for 1000 1-hr cycles. Scale spallation, before and after water immersion, was monitored by weight change, macrostructure, and acoustic emission. While exhibiting excellent cyclic oxidation resistance, exposure to humidity at room temperature often produced additional interfacial spallation events, occurring in rapid succession or bursts and continuing for hours. Using electrochemical hydrogen charging techniques, cathodic polarization in 1N H2SO4, at -2V and only 1 mA, caused massive scale delamination in a matter of minutes, with minimal N5 dissolution. Conversely, anodic polarization, at +2V and 100 mA, produced no delamination, but only gradual chemical dissolution. A new phenomenon, moisture-induced hydrogen embrittlement of the scale-metal interface, is proposed to explain these results.

9:00 AM Invited
Beneficial Aspects of Pt+Hf-Modified γ'-Ni_3Al+γ-Ni Alloys and Coatings for High-Temperature Oxidation and Corrosion Resistance: *Brian Gleeson*[1]; Daniel J. Sordelet[1]; [1]Iowa State University, Matls. Sci. & Engrg., 2220 Hoover Hall, Ames, IA 50011-2300 USA

Many high-temperature alloys and coatings rely on the formation of a continuous and adherent thermally grown oxide (TGO) scale of α-Al_2O_3 for extended resistance to degradation. For instance, the durability and reliability of thermal barrier coating (TBC) systems in gas turbines are critically linked to the oxidation behavior of the alumina-forming β-NiAl-based bond coat. It has been found that certain unique alloy compositions based on the Ni-Al-Pt-Hf system, yet sufficiently low in aluminum content to be free of β-NiAl, are excellent candidates for the development of novel bond coats for significantly improved TBC reliability and durability. Specifically, it has been found that γ'-Ni_3Al+γ-Ni alloys modified with 10-30 at.% Pt and up to 2 at.% Hf form highly adherent, slow-growing TGO scales during both isothermal and cyclic oxidation at high temperature (maximum temperature studied was 1200°C). Moreover, the thermodynamic activity of aluminum in the alloys is below that in Ni-base superalloys used for areo-engine turbine applications. As a consequence, and in complete contrast to the typically used β-based bond coat compositions, aluminum diffuses from the substrate alloys to the novel γ'+γ coatings. Thus, the novel coating compositions offer the advantages of (1) forming a slow-growing and adherent TGO scale of α-Al_2O_3, (2) not depleting in aluminum due to coating/substrate interdiffusion during service, and (3) being compatible with superalloy substrate in terms of phase constitution and, hence, coefficient of thermal expansion. This paper will review recent findings on the oxidation, hot-corrosion and interdiffusion behaviors of Pt+Hf-modified γ'+γ alloys and coatings.

9:30 AM Invited
Oxidation Behavior of Ni-Al-Hf-(Pt or Pd) Bond Coating Compositions: *Bruce A. Pint*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., PO Box 2008, MS 6156, Oak Ridge, TN 37831-6156 USA

Interdiffusion problems for conventional Pt-modified aluminide coatings can limit their long-term durability as the Al content in the coating decreases. Recent work has suggested that two-phase aluminide coatings with less than 25% Al may be more compatible with single-crystal superalloys. The oxidation resistance of this class of coatings is critically dependent on a precious metal addition to improve selective oxidation of Al and inhibit Ni-rich oxide formation. The oxidation behavior of model Ni-22.5Al+Hf alloys containing either Pt or Pd are being investigated at 1000°-1200°C in order to better understand the role of composition on oxidation resistance of this class of coatings.

10:00 AM Invited
Cyclic Oxidation Behavior of Ru-Containing Single Crystal Superalloys: *Q. Feng*[1]; B. Tryon[1]; T. K. Nandy[1]; T. M. Pollock[1]; [1]University of Michigan, Dept. of Matls. Sci. & Engrg., 3062 H. H. Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

Single crystal superalloys containing Ru as a new alloying addition have recently been of interest since Ru additions may further improve high temperature properties. Cyclic oxidation experiments were conducted on a series of experimental high Ru-containing single crystal superalloys as a function of temperature (900-1200°C) and alloying content. The experimental results indicated that high levels of Ru (3.5-9 at.%) and Cr (8 at.%) additions exhibit good oxidation behavior, equivalent to the second generation single crystal alloys. But high Ru containing alloys without Cr additions displayed poor oxidation resistance. The oxides present and the morphology of oxidized surface were characterized by X-ray diffraction and scanning electron microscopy (SEM). The possible oxidation mechanisms will be discussed.

10:25 AM Break

10:45 AM
Effects of Applied Stresses on Oxidation of Superalloys: *Bryan Randall Barnard*[1]; Peter K. Liaw[1]; Ray A. Buchanan[1]; Peter F. Tortorelli[2]; Bruce A. Pint[2]; Weiju Ren[2]; Dwaine L. Klarstrom[4]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., 427-B Dougherty

Engrg. Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6156 USA; [4]Haynes International, Inc., Tech. Engrg., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The purpose of this research is to examine the effects of applied stresses on the elevated-temperature oxidation behavior of superalloys. Elevated-temperature corrosion is a leading cause of failures among high-temperature structural components. Superalloys, particularly those that contain large amounts of chromium, are often the chosen materials for these components. This is because chromium forms oxide scales that protect the alloy from further damage due to elevated-temperature corrosion. These oxide scales have been studied extensively. However, the effects that applied stresses have on oxide layers have not been examined as thoroughly, and, thus, are the subject of this research. Preliminary results comparing the isothermal oxidation behavior of Haynes 75® and Haynes 230® alloys with and without an applied (creep) load will be presented for temperatures of 700 and 800°C. A model for predicting the oxidation behavior under various conditions of load and temperature will be developed, based upon the results of testing.

11:10 AM Invited
Oxidation and Partitioning Characteristics of Advanced Ni-Base Superalloys with Ru and Pt: L. Zhang[1]; *S. Tin*[1]; M. K. Miller[2]; S. S. Babu[2]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge, Cambridgeshire CB2 3QZ UK; [2]Oak Ridge National Laboratory, Microscopy, Microanalysis, Microstruct. Grp., Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, Oak Ridge, TN 37831 USA

The structural properties and temperature capabilities of advanced Ni-base single crystal superalloys can potentially be extended by utilizing Pt-group metal additions. Recent investigations have shown single crystal Ni-base superalloys containing additions of both Pt and Ru exhibit unusually good high temperature oxidation characteristics. Partitioning characteristics of these elements within the constituent phases of the microstructure have been investigated using atom probe and high resolution transmission electron microscopy. Results of cyclic oxidation tests on bare and aluminized specimens are presented. Environmental implications of incorporating Pt-group elements in Ni-base superalloys will be discussed.

11:35 AM
Oxidation Characteristics of the Third Generation Superalloy CMSX-10: *Ainul Akhtar*[1]; Roger C. Reed[1]; [1]University of British Columbia, Metals & Matls. Engrg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

The oxidation resistance of the single crystal superalloys is an important consideration, since it is not always the case that coatings can be employed, for reasons of practicality and cost. Here, the oxidation of bare CMSX-10 is studied. Since the Cr content is relatively low, it is found that the oxidation behaviour differs from earlier alloys in some important respects. Although an external scale of NiO containing spinels is formed, transformation into the beta phase preceeds the creation of an internal oxidation zone which contains (Ni,Co)Ta2O6 and (Ni,Co)WO4. We have found no evidence of alumina formation. The kinetics of oxidation are interesting: oxidation at higher temperatures can be slower than at lower temperatures, and it is postulated that this is due to the formation of the aluminide phase.

12:00 PM
Yttrium Doping for Oxidation Resistance of 4th Generation Ni-Base Superalloys: *Atsushi Sato*[1]; Kyoko Kawagishi[2]; Kenji Nishida[3]; Toshiharu Kobayashi[2]; Hiroshi Harada[2]; Hachiro Imai[1]; [1]Shibaura Institute of Technology, Dept. of Matls. Sci. & Engrg., 3-9-14, Shibaura, Minato-ku, Tokyo 108-0023 Japan; [2]National Institute for Materials Science, High Temp. Matls. Grp., 1-2-1, Sengen, Tsukuba, Ibaraki 305-0047 Japan; [3]National Institute for Materials Science, Surface Analy. Grp., 1-2-1, Sengen, Tsukuba, Ibaraki 305-0047 Japan

The 4th generation Ni-base single crystal (SC) superalloys with platinum group metals, e.g., Ru, have superior creep strengths and recessive oxidation resistance. In this study, yttrium doping was applied to a 4th generation Ni-base SC superalloy, TMS-138, to improve the oxidation resistance. TMS-138 base samples with yttrium contents ranging from 0 to 1000 wppm were examined by a 1100°C cyclic oxidation test. The weight change was found to be smallest with a sample containing 100 wppm yttrium. Microstructure observation with this sample showed that yttrium sulfide particles were dispersed in the gamma/gamma prime matrix, whereas other additional yttrium compounds were observed in samples with higher yttrium contents. Thus, it was considered that the right amount of yttrium capable enough of removing sulfur from the matrix to form sulfides improved the adhesiveness of oxide scales to the matrix and consequently, the oxidation resistance.

Surface Engineering in Materials Science - III: Nanocoatings

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Craig Blue, Oak Ridge National Laboratory, Materials Processing Group, Metals and Ceramic Division, Oak Ridge, TN 37831 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Tuesday AM Room: 2022
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sudipta Seal, University of Central Florida, Advd. Matls. Procg. & Analy. Ctr. & Mechl., Oviedo, FL 32765-7962 USA; John J. Moore, Colorado School of Mines, Dept. of Metall. & Matls. Engrg., Golden, CO 80401 USA

8:30 AM Invited
Nanocomposite Coatings: Structure and Properties: *Jeff T. De Hosson*[1]; [1]University of Groningen, Appl. Physics, Nijenborgh 4, Groningen 9747 AG The Netherlands

This contribution deals with fundamental and applied concepts in nano-structured coatings, in particular focusing on the characterization with high-resolution (transmission) electron microscopy. Both balanced and unbalanced magnetron-sputtering systems were used to deposit nc-TiC/a-C nanocomposite coatings with hydrogen-free DLC matrix. The contents of Ti and C in the coatings were varied in the whole range of interest (5~45 at.%Ti) by changing the configuration of the targets. The size and distribution of the nanoparticles were determined with High-Resolution (HR) and Energy-Filtered (EF) Transmission Electron Microscopy (TEM); their size varies between 2 and 20 nm diameter and particle clustering was observed. Electron microscopy was further employed to determine the chemistry of the a-C matrix (sp3/sp2 ratio), through Electron Energy Loss Spectroscopy (EELS). It was demonstrated that both TiC particle size and a-C chemistry could be directly controlled by change of deposition parameters such as applied bias and deposition pressure.

8:55 AM
Interfacial Phenomena in Carbon Nanotube Reinforced Al-Based Composite Coatings Deposited by Air Plasma Spray and High Velocity Oxy-Fuel Techniques: *Arvind Agarwal*[1]; *Tapas Laha*[1]; [1]Florida International University, Mechl. & Matls. Engrg., 10555 W. Flagler St., EC 3464, Miami, FL 33174 USA

Employment of carbon nanotubes (CNT) as reinforcement in metal matrix composite coatings potentially improves the fracture toughness, wear resistance and hardness. In the present effort, CNT reinforced Al-based composite coatings have been deposited by atmospheric plasma spray and high velocity oxy-fuel spray deposition techniques. The distribution of CNT reinforcement in the composite coatings has been investigated by scanning electron microscopy, X-ray diffraction, Raman spectroscopy and transmission electron microscopy. The characterization confirms presence of physically and chemically stable carbon nanotubes in both the composite coatings. Besides, TEM investigation has been carried out to understand the interfacial phenomena. The effect of high temperature and rapid solidification processing on the interfacial reactions, as well as on the wettability of aluminum on carbon nanotubes has been studied. Appreciable wetting of CNT by molten aluminum has been noticed in the composites.

9:10 AM
Role of Surface Treatment of Carbon Fibers on Properties: *G. V. Prabhu Gaunkar*[1]; S. P. Sharma[1]; S. C. Lakkad[1]; [1]IIT Bombay, Dept. of Metallurgl. Engrg. & Matls. Sci., Powai, Mumbai 400076 India

A process of modifying surface characteristics of fibers by incorporating nano and micro constituents has been developed. Carbon fibers of different strength levels and subjected to surface modification treatments have been used to make polymer matrix composites. Role of

surface modification of fibers on mechanical properties. Damage tolerance under impact loading has been studied using ultrasonic C scan technique and scanning electron fractography. Surface modification treatments are found to give significant improvements in mechanical properties and well as daamage tolerance under impact loading.

9:25 AM
Nano-Coatings on Carbon Structures for Interfacial Modification: *Rajasekhar Venkata Pulikollu*[1]; Pratik Joshi[1]; Sharmila Mitra Mukhopadhyay[1]; [1]Wright State University, Dept. of Mechl. & Matls. Engrg., 209 Russ Engrg. Ctr., 3640 Col. Glenn Hwy., Dayton, OH 45435-0001 USA

Microwave plasma has been used to deposit 1-5 nanometer thin films on surfaces of carbon foam and nano-fibers, and the resulting changes in interfacial properties are investigated. In this presentation, interfacial issues related to carbon-polymer, and carbon-metal composites will be discussed. Two types of coatings: One for enhancing the surface reactivity and another for surface inertness has been compared with uncoated carbon structures. It is observed that the stress-strain behavior, as well as fracture paths of foam-epoxy and nanofiber-epoxy composites is significantly altered by the nano-coatings. Additionally, the influence of these films on the microstructure and growth mechanism of metallic (copper) thin films on graphite has been investigated, and will be presented. The significance of these nano-coatings to formation of metal-matrix and polymer-matrix composites will be discussed.

9:40 AM Break

10:10 AM Invited
Nanostructured Coatings: Properties and Fundamentals: *Enrique J. Lavernia*[1]; [1]University of California, Coll. of Engrg., John D. Kemper Hall of Engrg., One Shields Ave., Davis, CA 95616 USA

The application of nanocrystalline materials used as powder feedstock for thermal spraying in recent years has been mainly facilitated by the wide range of powder sources available, including: vapor condensation, solution precipitation, combustion synthesis, sol-gel processing, thermochemical synthesis, and mechanical alloying/milling. The resultant thermal sprayed coatings have been shown to exhibit unique and often enhanced physical and mechanical performance properties in comparison to the coatings produced by current technology. Improvements in physical have been documented for several metallic and cermet based nanostructured coatings. However, the behavior of a nanostructured material during thermal spraying is rendered complex by factors such as morphology of feedstock powders; thermal stability of nanostructured powders; and thermal and momentum behavior of nanostructured powder. Optimization of chemistry, morphology and coating thickness, for example, should lead to the attainment of physical performance heretofore unattainable with conventional coatings. The present paper is to provide an overview of recent advancements in the field of high performance nanostructured coatings, paying particular attention to underlying fundamental issues. Examples of several metallic and cermet coatings and bulk samples will be used to demonstrate the influence of the morphology of nanostructured powders on performance.

10:35 AM
The Effect of the Surface Nanocrystallization-and-Hardening (SNH) Process on the Fatigue Behavior of a 316 Stainless Steel: *J. W. Tian*[1]; W. Yuan[1]; G. Y. Wang[1]; D. E. Fielden[1]; J. Jeon[1]; H. Choo[1]; J. C. Villegas[2]; L. L. Shaw[2]; P. K. Liaw[1]; [1]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996-0001 USA; [2]University of Connecticut, Metall. & Matls. Engrg., Storrs, CT 06269 USA

Using the surface-nanocrystallization-and-hardening (SNH) process, a nano-structured layer was found on the surface of the 316 stainless steel disk, while the coarse grain below the surface remained unchanged. A grain-size gradient from the surface nano-layer to the interior coarse grain, a micro-hardness gradient, and a residual compressive to tension stress distribution were introduced through the cross section of the samples. Microstructural features of the cross section near and below the surface were investigated by means of the X-Ray diffraction (XRD), scanning-electron microscope (SEM), and transmission-electron microscope (TEM). The microhardness of the cross section was measured by the Vicker's-hardness tester and nanoindentation, and the residual stresses distributions were determined by XRD. Four-point-bend-fatigue experiments were employed to study the fatigue behavior of the specimens. The results show that the nano-structured layer on the surface has high resistance to the fatigue-crack initiation, and the interior coarse grain can retard the growth of fatigue cracks, both of which evidently improved the fatigue lives of the specimens. However, with the further increase of the processing time, fatigue properties become worse because of the surface roughness and/or surface damage. The present work is supported by the National Science Foundation under DMR-0207729 with Dr. K.L. Murty and Dr. J. Akkara as the contract monitors.

10:50 AM
Nano-Engineered Multiwall Carbon Nanotube-Copper Composite Thermal Interface Material for Efficient Heat Conduction: *Quoc Xuan Ngo*[1]; Brett A. Cruden[2]; Alan M. Cassell[2]; Gerard Sims[2]; Jun Li[2]; M. Meyyappan[2]; Cary Y. Yang[1]; [1]Santa Clara University, Ctr. for Nanostruct., 500 El Camino Real, Santa Clara, CA 95050 USA; [2]NASA Ames Research Center, Ctr. for Nanotech., Moffett Field, CA 94035 USA

Efforts in integrated circuit (IC) packaging technologies have recently been focused on management of increasing heat density associated with high frequency and high density circuit designs. While current flip-chip package designs can accommodate relatively high amounts of heat density, new materials need to be developed to manage thermal effects of next-generation integrated circuits. Multiwall carbon nanotubes (MWNT) have been shown to significantly enhance thermal conduction in the axial direction and thus can be considered to be a candidate for future thermal interface materials by facilitating efficient thermal transport. This work focuses on fabrication and characterization of a robust MWNT-copper composite material as an element in IC package designs. We show that using vertically aligned MWNT arrays reduces interfacial thermal resistance by increasing conduction surface area, and furthermore, the embedded copper acts as a lateral heat spreader to efficiently disperse heat, a necessary function for packaging materials. In addition, we demonstrate reusability of the material, and the absence of residue on the contacting material, both novel features of the MWNT-copper composite that are not found in most state-of-the-art thermal interface materials. Electrochemical methods such as metal deposition and etch are discussed for the creation of the MWNT-Cu composite, detailing issues and observations with using such methods. We show that precise engineering of the composite surface affects the ability of this material to act as an efficient thermal interface material. A thermal contact resistance measurement has been designed to obtain a value of thermal contact resistance for a variety of different thermal contact materials.

11:05 AM
The Effect of Gas Pressure on the Structure and Tribological Performances of Nanometer Diamond-Like Carbon Films Prepared by Plasma-Based Ion Implantation: Liao Jiaxuan[1]; *Liu Weimin*[2]; Xu Tao[2]; Yang Chuanren[1]; Chen Hongwei[1]; Fu Chunlin[1]; *Leng Wenjian*[1]; [1]University of Electronic Science and Technology of China, Sch. of Microelect. & Solid-State Elect., Chengdu 610054 China; [2]Chinese Academy of Sciences, State Key Lab. of Solid Lubrication, Lanzhou Inst. of Cheml. Physics, Lanzhou 730000 China

Nanometer diamond-like carbon (DLC) films were prepared on Si wafers by plasma-based ion implantation at 18kV for 30min. The effect of gas pressure ranging from 0.5Pa to 2.0Pa on the DLC films has been investigated. Dry sliding tribological experiments against alumina balls have also been carried out on a ball-on-disc tester. The results show that the films range from 30nm to 70nm in thickness, and are firmly adhered to Si substrate owing to a C-implanted layer. A critical gas pressure of 0.5Pa corresponds to no films but a C-implanted layer. The DLC films exhibit an average roughness of less than 0.50nm, a content of sp3 bonds of more than 50% and an improvement in tribological performances. Meanwhile, an increasing gas pressure corresponds to an increasing film thickness, a decreasing content of sp3 bonds and an improvement in tribological performances.

Texture and Microstructure in Thin Films and Coatings: Coatings

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee
Program Organizers: David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; John E. Sanchez, Advanced Micro Devices, Sunnyvale, CA 94088 USA; J. A. Szpunar, McGill University, Department of Metallurgical Engineering, Montreal, Quebec H3A 2A7 Canada

Tuesday AM Room: 3010
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Bae-Kyun Kim, McGill University, Mining, Montreal, Quebec H3A 2B3 Canada

8:30 AM
Texture and Microstructure in Oxide Films Formed During Oxidation of Pure Iron: *Bae-Kyun Kim*[1]; *Jerzy A. Szpunar*[1]; [1]McGill University, Mining, Metals & Matls., 3600 Univ. St., Montreal, Quebec H3A 2B3 Canada

In order to improve the quality of steel surface, it is important to understand descaling process that take place in hot rolling. For better understanding of the scale removal, iron oxide texture, microstructure and structure of interphones should be characterized. Electron backscattered diffraction (EBSD) become an important tool for studying local texture and microstructure of materials. The orientation imaging microscopy (OIM) based on the EBSD technique can be used to examine transformation of texture and microstructure during high temperature oxidation. OIM can also be used to study oxide defects, which may affect the cracking of the oxide scale. In order to simulate industrial hot rolling, high temperature oxidation tests of pure iron were performed in the tube furnace up to 950°C with continuous heating under atmospheric pressure. The iron oxide microstructure can be described using OIM maps representing the image quality (IQ) and the inverse pole figure (IPF) of the cross-sectional area of oxidized iron. The three different iron oxides phases, namely the wüstite (FeO), magnetite (Fe3O4), and hematite (Fe2O3), are distinguished and the characteristics of oxides developed during different oxidation treatment are compared. Additionally, interfacial structure between different iron oxide layers is analyzed in relation to nucleation of microcracks and mechanism of oxide removal from the substrate.

8:50 AM
The Effects of Substrate Bias, Substrate Temperature and Pulse Frequency to the Microstructures of CrN Coatings Deposited by Pulsed DC Magnetron Sputtering: *Jyh-Wei Lee*[1]; Shih-Kang Tien[2]; Chih-Hsiung Lin[2]; Jenq-Gong Duh[2]; [1]Tung Nan Institute of Technology, Dept. Mechl. Engrg., #152, Sec.3 Pei-Shen Rd., Shen-Ken, Taipei County 222 Taiwan; [2]National Tsing Hua University, Dept. Matls. Sci. & Engrg., #101, Sec.2, Kuang-Fu Rd., Hsin-Chu 300 Taiwan

The chromium nitride coatings have been deposited by the bipolar symmetric pulsed DC magnetron reactive sputtering process at different pulse frequency, substrate bias power and substrate temperature. The substrate bias was applied with constant pulse frequency of 50 KHz. The surface and cross sectional morphologies of the coatings were analyzed by the field emission scanning electron microscopy. Phase analysis of the coatings was performed by the XRD. For the CrN coatings deposited with pulse frequency of 2 and 20 KHz, the preferred orientation of coatings changed from (111) to (200) as substrate temperature and bias increased. The deposition rate of the CrN films decreased with increasing pulse frequency, whereas the substrate temperature and bias showed almost no influence. It was concluded that the pulse frequency, substrate bias and temperature played important roles to the texture, microstructure and surface roughness of the CrN coatings deposited by the pulsed DC magnetron sputtering.

9:10 AM
Simulation of Texture Development in Oxide Films: *Hualong Li*[1]; Jerzy A. Szpunar[1]; [1]McGill University, Mining, Metals & Matls., 3600 Univ. St., Montreal, Quebec H3A 2B2 Canada

A methodology that can be used to simulate the texture formation in oxide formed on metal substrate is proposed. Graphically enhanced discrete computer simulation incorporates Orientation Imaging Microscopy (OIM) experimental data to characterize the texture and microstructure of the substrate that used as an input to simulation. It also allows user to generate input data based on X-ray measurements of texture. The abundant information contained in OIM measurement allows the computer model to incorporate many structural characteristics of polycrystalline materials such as, texture, grain boundary character, grain shape and size, phase composition, chemical composition, stored elastic energy, and residual stress. The input data to the simulation characterize the interfacial energy between the oxide and substrate, state of the stress in oxide film and oxide diffusivity in the bulk and also the grain boundary diffusion coefficients. These data can be obtained from the experiments or from the first principle calculations. Graphical representation allows the user to monitor each simulation step of the structural transformation of oxide film and to perform virtual experiments. In this paper, we will present the results of simulation of oxide layer in Zr and Ni alloys.

9:30 AM
Significant Improvements in Ni-B Coatings for Metal Alloys: *Yancy W. Riddle*[1]; Ed McComas[1]; Wynn Atterbury[2]; [1]Universal Chemical Technologies, Inc., 7825 SW Ellipse Way, Stuart, FL 4997 USA; [2]UCT Defense, 7825 SW Ellipse Way, Stuart, FL 34997 USA

Significant improvements to the properties associated with early generations of Ni-B coatings for metal alloys have been made. When compared to other industrial metal coatings such as hard chrome new Ni-B coatings can outperform. Unfortunately many engineers and scientists refer to the properties of early generation Ni-B coatings during investigative or comparative studies. This paper serves as a technical and scientific update of Ni-B coatings based on an extensive research program. These new Ni-B coatings are of major industrial importance due to improved properties of; hardness, lubricity, ductility, coefficient of friction, wear resistance, corrosion resistance, environmental friendliness, safety, processing, thermal compatibility, and more. A wide variety of carbon and stainless steels, aluminum alloys, copper alloys, titanium alloys, and other metal alloys can be successfully coated by these new Ni-B coatings. Included with this paper are comparative performance data between old and new generation Ni-B coatings reinforced by microstructural evidence and examples of successful application in industry.

9:50 AM
Crystallographic Texture of EB-PVD TBCs on Stationary Flat Surfaces in a Multiple Ingot Chamber: Jeremy Bernier[1]; G. Levan[2]; Sudha Bose[2]; Md. Maniruzzaman[1]; *Richard D. Sisson*[1]; [1]Worcester Polytechnic Institute, Mfg. & Matls. Engrg., Mechl. Engrg. Dept., 100 Inst. Rd., Worcester, MA 01602 USA; [2]Pratt & Whitney, Matls. & Process Engrg., E. Hartford, CT USA

The crystallographic texture of EB-PVD TBCs (7 wt% Y2O3) deposited in a two ingot chamber has been experimentally determined by comparing pole figure data with measured column growth angles. It was found that the coating deposited on a flat surface directly above an ingot exhibited <220> texture. Coatings deposited between the ingots and off of the centerline exhibited <311> texture>. Coating deposited at the far corners of coating chamber revealed a <110> fiber texture or <311> single crystal type texture. The microstructures of specimen cross sections reveal that column growth angle and vapor incidence angles are in reasonable agreement and that the columns grow towards the closest ingot. These results are discussed in terms of substrate temperature and vapor incidence angles.

10:10 AM Break

10:40 AM
The Metallurgy of High-Strength and Thermally Stable Nanostructured Ni-Mn Alloys: *Albert Alec Talin*[1]; [1]Sandia National Laboratories, 7011 East Ave., Livermore, CA 94550 USA

Electroplated NiMn alloy is a promising material for applications requiring high strength, thermal stability and low residual stress. In this paper we report on the variation of crystallite diameter, crystallographic texture, and yield strength of NiMn electrodeposits as a function of current density and Mn content. Over the concentration range of 0 wt. % to 1 wt. %, Mn reduces the average crystallite size from hundreds of nanometer to ~20 nm, dramatically alters the texture dependence on current density, and increases the yield strength from ~ 300 MPa to over 1 GPa. We also show how small additions of Mn solute affects the recrystallization temperature and texture of Ni, and how these results can be understood in terms of the spatial distribution of Mn in Ni, obtained using three dimensional atom probe technique.

11:00 AM
Texture and Corrosion Resistance of Electrodeposited Tin Coatings: *Shixue Wen*[1]; Jerzy A. Szpunar[1]; [1]McGill University, Dept. of Mining, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

Due to its non-toxicity, good corrosion resistance and excellent solderability, tin is widely used in food packaging and electronic industries. Tinplate, mild steel coated with tin, is a material of great economic importance. Tin electrodeposition on mild steel substrates using stannous sulfate and sulfuric acid was studied under different current densities, temperature and organic additives. The macrotexture of the tin deposits was measured using an x-ray texture goniometer. The orientation imaging microscopy (OIM) was used to measure the orientations and sizes of individual grains. The corrosion resistance of electrodeposited tin coatings was measured using a standard corrosion cell kit according to the ASTM standard G3. It was found that tin coatings with three different texture, (110), (100) and (301) fiber texture can be produced by electrodeposition. At a temperature 20ºC and a current density of 50A/m2, a fibre (110) was obtained. As the current density increased to 100 and 200 A/m2, the texture of tin coatings was changed to (100) fibre. With a different organic additive, tin coatings with (100) fibre and (301) fibre textures were obtained at the current densities of 200A/m2 and 50A/m2 respectively at 20ºC. An increase in current density leads to a decrease in grain sizes. At the same current density, the grain sizes of tin coatings increase with increased temperature. The influence of temperature (20, 40, 60 and 80ºC) on texture is relatively negligible. The corrosion resistance of tin coatings increases with a decrease in grain size. The corrosion resistance of tin coatings with (301) fibre was 1.5 times higher than that of the tin coating with (100) fibre texture and the corrosion current density was 0.2092 µA/cm2 for the former sample and 0.3011µA/cm2 for the latter sample. The results suggest that texture and microstructure (grain size) play an important role in controlling corrosion rate of tin based coatings. It also can be concluded that texture's influence on corrosion resistance is much higher than that of grain size.

11:20 AM
Microstructure and Mechanical Properties Evaluation of Chromium Nitride/Tungsten Nitride Superlattice Coatings: Fan-Bean Wu[1]; *Jyh-Wei Lee*[2]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Matls. Sci. Ctr., #101, Sec. 2, Kuang-Fu Rd., Hsin-Chu 300 Taiwan; [2]Tung Nan Institute of Technology, Dept. Mechl. Engrg., #152, Sec.3 Pei-Shen Rd., Shen-Ken, Taipei County 222 Taiwan

Chromium nitride and tungsten nitride superlattice coatings have been fabricated by a dual-gun rf magnetron reactive sputtering system. The superlattice period of the coatings varies in the range 6 to 24 nm. The microstructures of the CrN/WNx coatings were evaluated by the field emission scanning electron microscopy and transmission electron microscopy. Elemental distributions of the coatings were revealed by the Auger electron spectroscopy depth profiling analysis. Nanoindentation technique was employed to evaluate the mechanical properties including hardness and Young's modulus. The phase identification indicated that the coatings were composed of the CrN and W2N phases. The CrN/WNx superlattice coatings exhibited a high hardness over 30 GPa, which was superior to that of the single CrN coating. The nanolayered structure that confined the grains of the nitrides in the nano range was beneficial to the enhancement of the mechanical performance of the multilayer coating.

11:40 AM
Effect of Process Current Density and Temperature on Electrochemical Boriding of Steel in Molten Salts: *Guldem Kartal*[1]; Servet Timur[1]; [1]Istanbul Technical University, Metall. & Matls. Sci. Engrg., ITU Chmst-Metall. Faculty, Maslak, Istanbul 34469 Turkey

In this study, the boriding of steels by molten salt electrolysis in borax-base electrolyte at various current densities (50-700mA/cm2), was investigated. The influence of the electrolysis parameters (current density) on thickness and morphology are present. Different borided phases can form depending on the amount of the diffused boron from surface to matrix as follows: FeBx(x>1)?¨FeB?¨Fe2B?¨Fe3B?¨FeyB(y>3)?¨Fe. Determination of optimum current density for the boriding of DIN EN 10130-99 DC04 low carbon steel are 1 hour and at constant bath temperature (900°C), and % 10 NaCl + % 90 Na2B4O7 bath composition.

The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session III

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; John William Morris, University of California, Department of Materials Science and Engineering, Berkeley, CA 94720 USA

Tuesday AM Room: 3003
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: David E. Laughlin, Carnegie Mellon University, MSE, Pittsburgh, PA 15213 USA; Peter W. Voorhees, Northwestern University, MSE, Evanston, IL 60208 USA

8:30 AM Opening Remarks

8:35 AM Invited
Elastic Stability and the Limits of Strength: *John William Morris*[1]; [1]University of California, Matls. Sci. & Engrg., 210 Hearst Mining, Berkeley, CA 94720 USA

The upper limit of strength (the "theoretical strength") has been an active subject of research and speculation for the better part of a century. The subject has recently become important, for two reasons. (1) Given recent advances in ab initio techniques and computing machines, the limits of strength can be calculated with considerable accuracy, making this one of the very few problems in mechanical behavior that can actually be solved. (2) Given recent advances, the limits of strength are being approached in some systems, such as hardened or defect-free films, and their relevance is becoming recognized in others. The present paper discusses results from recent research on the limits of strength. Topics include: criteria for elastic stability, the inherent nature of {100} cleavage in bcc metals, the source of strength in steel, resistance to cleavage in fcc metals, the difference between "hard" and "soft" carbonitrides, and the possibility of measuring ideal strength with nanoindentation.

9:00 AM Invited
Linking Phase Field Method to Ab Initio Calculations: Modeling Dislocation - Precipitate Interactions in Real Alloys: Chen Shen[1]; *Y. Wang*[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210 USA

In an effort to understand effects of microstructural characteristics on deformation mechanisms observed in Ni-based superalloys, we incorporate directly information from ab initio calculations into phase field model of dislocations. This is accomplished by formulating appropriate invariant forms of the crystalline energy, which relates explicitly to the generalized stacking fault (GSF) energy. The use of ab initio GSF energy in the phase field model allows for quantitative studies of core structures of both stationary and moving dislocations in individual phases as well as at interphase interfaces, and the effects of size and spatial distribution of gamma prime particles on deformation mechanisms. The work is supported by the National Science Foundation.

9:25 AM
Phase-Field Modeling of ä?-Precipitation at Dislocations in Al-Li Alloys: *Y. H. Wen*[1]; A. W. Zhu[2]; G. J. Shiflet[2]; [1]UES Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]University of Virginia, Dept. Matls. Sci. Engrg., Charlottesville, VA 22904 USA

A phase-field model is formulated to study the precipitation process at the presence of various dislocations. The evolution of the particular morphology of ä? precipitates are demonstrated to be associated with interaction between the stress fields around edge dislocations and the particle in Al-Li alloys. The results are compared with TEM observation where the ä? heterogeneous precipitation occurs through isothermal aging with small undercoolings. Work completed under Air Force Contract # F33615-01-C-5214.

9:40 AM
Dislocation-Induced Crossover Scaling During Spinodal Decomposition: *Mikko P. Haataja*[1]; Jennifer Mahon[2]; *Nikolas Provatas*[2];

Francois Leonard[3]; [1]Princeton University, Mechl. & Aeros. Engrg., Olden St., Princeton, NJ 08544 USA; [2]McMaster University, Matls. Sci. & Engrg., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada; [3]Sandia National Laboratories, Livermore, CA 94551 USA

In this talk we address the role of mobile dislocations on the phase separation process of a binary alloy. We employ a phase-field model which explicitly incorporates the dynamics of the composition and dislocation fields in two spatial dimensions, coupled through their elastic fields. We show both analytically and numerically that the effects of mobile dislocations on domain coarsening kinetics can be expressed via a crossover scaling function. We also discuss experimental ramifications of such a crossover scaling function.

9:55 AM
The Influence of Interstitial Oxygen and Peak Pressure on the Shock Loading Behavior of Zirconium: *E. K. Cerreta*[1]; G. T. Gray[1]; R. S. Hixson[2]; R. A. Rigg[2]; D. W. Brown[1]; B. L. Henrie[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, DX-2, Los Alamos, NM 87545

The pressure of the a-w phase transition in zirconium has been quantified as a function of interstitial content. The pressure increases with increasing interstitial oxygen content and for the high purity (HP) material occurs at 7.1GPa. Increasing the interstitial oxygen content increases the number of octahedral sites occupied; this is postulated to increase the pressure for the phase transformation. Deformation behavior and substructural evolution of as-annealed HP zirconium under quasi-static conditions has been compared to its response following shock prestraining at 5.8 and 8 GPa. The reload stress-strain response of HP zirconium shock prestrained to 8GPa was found to exhibit enhanced hardening when compared to its quasi-static constitutive behavior. The reload yield behavior of zirconium specimens shocked to 5.8 GPa did not exhibit enhanced shock hardening.

10:10 AM Break

10:35 AM Invited
Modeling of Cyclical Phase Transformations in Mechanical Alloying: *Jong K. Lee*[1]; [1]Michigan Technological University, Dept. of Matls. Sci. & Engrg., Houghton, MI 49931 USA

As a driven process, mechanical alloying synthesizes a mixture of equilibrium and non-equilibrium phases at a different temporal sequence. In many instances, the relative phase fractions are apparently invariant after long milling times. The situation is termed dynamic equilibrium. Some recent works, however, show evidence of cyclical phase transformations taking place during mechanical alloying. Cyclical phase transformations resemble dynamic equilibrium in the sense that two phases are simultaneously present, but the phase fractions vary. A brief thermodynamic and kinetic account is first discussed to establish the criteria for cyclical transformations. A two-dimensional molecular dynamic work is then followed to demonstrate cyclical phase transitions between an equilibrium and a non-equilibrium phase during mechanical alloying. Model binary nanocrystals, made of Lennard-Jones atoms, are studied to display cyclical transitions between an equilibrium rhombus and a non-equilibrium square phase under periodic, small-shear loading conditions.

11:00 AM Invited
Elastic Inhomogeneities and Microstructural Evolution: *Perry H. Leo*[1]; [1]University of Minnesota, Aeros. Engrg., 107 Akerman Hall, 110 Union St. SE, Minneapolis, MN 55408 USA

The importance of elastic effects on microstructural evolution during diffusional phase transformations has been well documented in the past ten years. In particular, Professor Khachaturyan and his coworkers have made remarkable advances in the theory and simulation of elastic effects on particle behavior and multiparticle interactions. In this talk, we present some work extending these ideas to show how elastic inhomogeneity in cubic materials affects the elastic interactions. We go beyond the ideas of 'soft' and 'hard' precipitates to consider cases where some elastic constants may be higher in the precipitate than the matrix, while other constants are lower. We document the specific role of the elastic constants on particle interactions and on certain behavior of individual particles, such as particle splitting.

11:25 AM Invited
Spontaneous Patterning of the Chemical Order Field in L12 and L10 Ordered Alloys Subjected to Energetic Irradiation: *Pascal M. Bellon*[1]; Jia Ye[1]; [1]University of Illinois, Matls. Sci. & Engrg., 1304 W. Green St., Urbana, IL 61801 USA

Dense displacement cascades produced by irradiation with energetic particles lead to the formation of disordered zones in chemically ordered alloys. At temperatures below the order-disorder transition, these disordered zones, whose sizes range from a few to several nanometers, are thermally annealed out. Under sustained irradiation, the competition between these two dynamics drives the system into various steady states. Kinetic Monte Carlo simulations and analytical modeling are employed to identify these steady states in binary alloys that form L12 and L10 ordered phases at equilibrium. Besides the expected long range ordered and disordered steady states, a new state is discovered, where the microstructure is comprised of well ordered domains of finite size. A two-stage reordering of the disordered zones is at the origin of the dynamical stabilization of these patterns. The present results indicate that ion-beam processing could be used to synthesize ordered nanocomposites with tunable sizes.

11:50 AM
Evolution of Deformation and Recrystallization Textures in Cold-Rolled CP-Ti - Experiments and MC Simulation: Y. B. Chun[1]; S. L. Semiatin[2]; *S. K. Hwang*[1]; [1]Inha University, Sch. of Matls. Sci. & Engrg., 253 Yong Hyun-Dong, Nam-Gu, Incheon 402-751 S. Korea; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

The development of deformation texture in CP-Ti during cold rolling (10%~90%) and the evolution of the recrystallization texture during subsequent heat treatment were determined experimentally using XRD and EBSD and modeled using Monte-Carlo computer simulations. For a low- to-medium level of deformation (up to 40%), twinning governed the deformation and gave rise to a normal basal texture. For higher levels of deformation (up to 90%), however, the main deformation mechanism was slip, and a bi-modal basal texture with peaks at j1=0', F=35', j2=30' was developed. Recrystallization annealing suppressed the cold-rolling texture and introduced instead two new texture components (j1=15', F=35', j2=35' and j1=0', F=35', j2=0'), whose intensities significantly increased during the grain-coarsening stage of recrystallization and grain growth. From the EBSD analysis, it was found that grains with the major recrystallization-texture components had considerably larger grain sizes than others. A two-dimensional Monte-Carlo simulation was conducted to trace the evolution process of the major recrystallization-texture components. The fact that the intensification of the recrystallization texture occurred during the grain-coarsening stage, found by EBSD-mapping method, was also confirmed by the simulation. The present results suggest that the heterogeneous grain-size distribution during the recrystallization process was responsible for the evolution characteristics of the recrystallization texture in CP-Ti.

The Langdon Symposium: Flow and Forming of Crystalline Materials: Grain Boundary Properties and Severe Plastic Deformation

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; P. B. Berbon, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA; Atul H. Chokshi, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India; Z. Horita, Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-8581 Japan; Sai V. Raj, NASA Glenn Research Center, Materials Division, Cleveland, OH 44135 USA; K. Xia, University of Melbourne, Department of Mechanical and Manufacturing Engineering, Victoria 3010 Australia

Tuesday AM Room: 3024
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: T. G. Nieh, University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996-2200 USA; Sergey Dobatkin, Russian Academy of Sciences, A.A. Baikov Inst. of Metall. & Matls. Sci., Moscow 119991 Russia; Sean R. Agnew, University of Virginia, Matls. Sci. & Engrg., Charlottesville, VA 22904 USA; Zenji Horita, Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan

8:30 AM
Fatigue Behavior of Ultra-Fine Grain Copper: Kai Zhang[1]; Adam Chenoweth[1]; Giancarlo Izzi[1]; Abhishek Lahoti[1]; *Julia R. Weertman*[1]; [1]Northwestern University, Matls. Sci. & Engrg. Dept., 2220 Campus Dr., Evanston, IL 60208 USA

Fatigue experiments have been carried out on ultra-fine grain copper prepared by cryogenic rolling. Samples are tested in the pull-pull mode. Results in the form of S-N curves will be presented, along with characterization studies of changes in the surface features and internal structures caused by the cyclic deformation. This research is supported by US DOE grant DE-FG02-02ER 46002.

8:45 AM

Microstructures and Mechanical Properties After Processing by High-Pressure Torsion: *Zenji Horita*[1]; Genki Sakai[1]; Kaoru Kishikawa[1]; Yoshimi Hisatsune[1]; Terence G. Langdon[2]; [1]Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

In this study, severe plastic strain was imposed through the process of high-pressure torsion (HPT). An Al-3%Mg-0.2%Sc alloy and pure Cu were subjected to HPT to examine the effect of applied pressure, number of revolutions and position of the sample on the microstructures and the mechanical properties. The results show the hardness increases with increasing applied pressure or with increasing numbers of revolutions but these increases tend to become saturated and the saturation is more easily attained in the outer area than in the central area. Microstructural observations revealed that the outer part consists of fine grains having high-angle grain boundaries with a grain size of ~0.17 μm for the Al-3%Mg-0.2%Sc alloy. This alloy exhibited superplastic ductilities of ~500% when tensile specimens were taken at points away from the central area.

9:00 AM

Significantly Increased Strength and Rate Sensitivity Due to Grain Refinement or Twinning at the Nanoscale: *Ming Dao*[1]; Lei Lu[2]; Ruth Schwaiger[3]; Subra Suresh[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 8-139, Cambridge, MA 02139 USA; [2]Chinese Academy of Sciences, Shenyang Natl. Lab. for Matls. Sci., Inst. of Metal Rsch., Shenyang 110016 China; [3]Forschungszentrum Karlsruhe, Inst. for Matls. Rsch. II, Karlsruhe 76133 Germany

Indentation and tensile experiments performed on electrodeposited nanocrystalline Ni showed significantly increased rate sensitivity in addition to marked improvement in strength comparing with experiments on ultrafine crystalline Ni. A physically motivated model of Grain Boundary Affected Zone (GBAZ) is proposed to explain the observed experimental phenomenon. The GBAZ model simply assumes that, duo to the presence of a grain boundary, within a distance on the order of 10 lattice parameters, the material is plastically softer than the grain interior material. A simple unit cell finite element model integrating the GBAZ concept is able to capture the correct experimental trends. Taking the assumption that the twin boundaries of nano-sized twins have the similar effect as grain boundaries of nanocryalline materials, the concept of Twin-Boundary Affected Zone (TBAZ) can be integrated into a polycrystalline model to study the significantly increased plastic strength and rate sensitivity in pure Cu with nano-sized twins. Within the proposed physically motivated crystal plasticity framework, the orientation and size dependent plastic behavior parallel (plastically softer) and perpendicular (plastically harder) to the twin boundaries is specifically modeled. Parametric studies showed that the proposed model can correctly capture the significantly increased plastic strength and rate dependence versus a decreasing nanoscale twin thickness. Possible influences on the ductility due to the presence of many nano-sized twins are also discussed.

9:15 AM

Deformation Mechanisms of Nanostructured Materials: *Yuntian Ted Zhu*[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA

Nanostructured materials deform via mechanisms not accessible to their coarse-grained counterparts. Partial dislocation emission from grain boundaries, stacking faults and deformation twinning may occur in metals such as Al, which does not deform by twinning in its coarse-grained state. In this presentation I'll discuss several deformation mechanisms in nanomaterials as well as their formation conditions.

9:30 AM

Influence of Stacking Fault Energy on Deformation Twinning in Nanocrystalline Cu and Cu Alloys: *Yonghao Zhao*[1]; Xiaozhou Liao[1]; Yuntian Ted Zhu[1]; Zenji Horita[2]; Terence G. Langdon[3]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]Kyushu University, Dept. Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan; [3]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Deformation twinning has been observed in nanocrystalline copper and aluminium although these two face-centered-cubic (fcc) metals usually do not deform by twinning in their coarse-grained state. It has been found that partial dislocation emission from the boundaries of nano-grains is responsible for the formation of deformation twins and this twinning mechanism is different from those operating in coarse-grained materials. The stacking fault energy is known to affect the twinning in coarse-grained materials but its effect on twinning in nanocrystalline materials has not been studied. This paper presents a systematic study of the influence of stacking fault energy on deformation twinning in nanocrystalline copper and copper alloys processed by high pressure torsion.

9:45 AM

Deformation Twinning in Nanocrystalline Stainless Steel Within Adiabatic Shear Bands: *Qing Xue*[1]; Xiaozhou Liao[1]; Yuntian Theodore Zhu[1]; George T. Gray[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., G755, Los Alamos, NM 87545 USA

Transmission electron microscopy was used to investigate the nanostructure and the deformation mechanism of adiabatic shear bands in a cold-rolled stainless steel deformed during dynamic forced shear tests, in which the localized deformation occurred under high strain rates and locally high temperature. The development of adiabatic shear localization resulted in continuous refinement of the original coarse grains to nanocrystalline grains. High-resolution transmission microscopy examination showed extremely high density of nanotwins within the nanocrystalline grains at the centers of shear bands, indicating that twining played a significant role in the localized deformation when the grain sizes were at the nano range. The twinning mechanism in nano-scale stainless steel will be discussed.

10:00 AM

Design of Microstructures of Difficult-to-Work Alloys Utilizing Deformation Twinning During Severe Plastic Deformation: *Ibrahim Karaman*[1]; [1]Texas A&M University, Dept. of Mechl. Engrg., MS 3123, Coll. Sta., TX 77843 USA

The works of Prof. Langdon on severe plastic deformation (SPD) of metal alloys have helped understanding of the mechanisms of the SPD stages and often inspired the author in his studies on the SPD of difficult-to-work alloys. This talk will summarize our recent work on severe plastic deformation processing of several structural and smart materials using Equal Channel Angular Extrusion (ECAE). The common characteristic of these studies was the utilization of deformation twinning to modify the microstructure to obtain submicron and nanograins with low CSL boundaries. We will present the common features we have observed in Ti-6Al-4V, AISI 316L stainless steel and NiTi shape memory alloys and challenges and opportunities for twinning induced grain boundary engineering. The unique observation was that significant deformation twinning activity was observed for the first time in Ti-6Al-4V and 316L stainless steel at temperatures as high as 800°C (0.65 Tm!). Possible mechanisms of twin nucleation in these unusual cases will be discussed. ECAE of NiTi led to the observation of highly organized, twin-related nanograins in the high temperature phase which enhance cyclic stability and fatigue resistance of this alloy. Deformation twinning in a B2 intermetallic is an additional mechanism that improves the ductility of these materials. The formation of well-organized twin-related nanograins via severe plastic deformation opens a new opportunity for twinning induced grain boundary engineering in Ti-6Al-4V, austenitic steels and B2 NiTi intermetallics.

10:15 AM

Multiscale Investigations of Grain Refinement Using Severe Plastic Deformation: *Igor V. Alexandrov*[1]; Ruslan Z. Valiev[1]; [1]Ufa State Aviation Technical University, 12 K. Marx, Ufa 450000 Russia

The current work represents the results of combined computer modeling and experiments conducted by Ufa team on different scale (macro-, meso- and micro-) levels. These investigations aimed to study the equal channel angular (ECA) pressing process, reveal the peculiarities and mechanisms of the crystallographic texture development and microstructure refinement. The homogeneity of the obtained texture and microstructure in pure SPD metals depending on the processing parameters has been studied as well. The obtained results help to optimize ECA processing of the bulk nanostructured ingots and to predict regimes of the development of homogeneous structure and texture in pure metals with the different crystal lattice such as FCC copper and HPC titanium. The work was carried out within the framework of project CRDF 10505 Model-driven manufacturing of nanocrystalline structures (project coordinator Dr. I.J. Beyerlein) in cooperation with scientists from LANL (Los Alamos), VNIIEF (Sarov), PTI (St. Petersburg).

10:30 AM Break

10:45 AM
Plasticity and Strength of Nanocrystalline Ni and Ni-W Alloys: *T. G. Nieh*[1]; Jeffrey Wadsworth[2]; [1]University of Tennessee, Matls. Sci. & Engrg., Dougherty Engrg. Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Bethel Valley Rd., No. 1, Oak Ridge, TN 37831 USA

In this paper, both experiments and simulation results from nanocrystalline Ni with a grain size less than 20 nm are presented. The effect of grain size, the type of grain boundary, and alloying on the strength and plasticity will be discussed. Microstructure and impurity distribution in the nano-alloys both before and after deformation are examined using techniques such as atom probe microscopy (APM), electron energy loss spectroscopy (EELs) with a nano-sized probe, and high-resolution TEM with high angle angular dark field (HAADF) imaging capability (i.e. Z-contrast). Fracture process in nanocrystalline alloys will be addressed. MD simulation to show tension-compression strength asymmetry in nanocrystalline Ni will also be presented.

11:00 AM
On Applying Nano-Grained Superplastic Materials to General Residential Seismic Dampers: *Kenji Higashi*[1]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1, Gakuen-cho, Sakai, Osaka 599-8531 Japan

Recently the superplastic damping device, which has been using Zn-22Al alloys with an ultra-fine (nano order) grain size, has been put into practical use for a high-rise building. For the purpose of the improvement and mass-production of the damping devices by nano-grained superplastic materials, the investigation about the mechanical properties in the extruded alloys and the capability of superplastic forming was carried out. The extruded alloys, produced by optimum treatments, exhibited lower stress and higher elongation in comparison with the rolled alloys, i.e., the superplasticity at higher strain rate occurred. As the results of FVM analysis, it was verified that the formability of the rectangular samples was inferior to the rod samples because of the high effective strain at the corner. The tensile properties after the forging deteriorated due to the grain growth during the forging, but satisfied the mechanical properties needed to utilize as a damper in practice.

11:15 AM
Modelling Grain Boundary Strengthening in Ultra-Fine Grained Aluminium Alloys: *Erik Nes*[1]; Bjørn Holmedal[1]; Knut Marthinsen[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., Alfred Getzv. 2, Trondheim N-7491 Norway

By careful annealing of heavily deformed metals ultra-fine grained materials can be obtained. This phenomenon has been known for long and utilised in the production of special aluminium sheet qualities. It has received new interest with the emergence of the equal channel angular pressing (ECAP) technique, where pioneering works have been performed by T. Langdon and co-workers. In the present work the mechanical properties of aluminium alloys with grain sizes in the range from less than a micron (ultra-fine) to hundreds of microns have been modelled within the framework of the multi-parameter microstructural work hardening model developed by Nes and co-workers. The effect of grain size on the flow-stress and work hardening, including a deviation from the Hall-Petch grain size dependency for ultra-fine grain sizes, is well accounted for by the model. A mechanism is suggested for the sharp yield point and associated Lüder-band elongation relevant for ultra-fine grained AlMg-alloys.

11:30 AM
Nano- and Submicrocrystalline Steels by Severe Plastic Deformation: *Sergey Dobatkin*[1]; [1]Russian Academy of Sciences , A.A. Baikov Inst. of Metall. & Matls. Sci., Leninsky prospekt, 49, Moscow 119991 Russia

The aim of this paper is to consider the features of structure evolution during SPD of steels, thermal stability of the UFG structure processed and its influence on mechanical properties. The investigation have been carried out mainly on low and high carbon steels as well as on austenitic and ferritic stainless steels after SPD by torsion under high pressure and equal channel angular (ECA) pressing. Structure formation dependencies on temperature deformation conditions, strain degree, chemical composition, initial state and pressure are considered. The role of phase transformations for additional grain refinement, namely, martensitic transformation, precipitation and dissolution of carbide particles during SPD and others, is underlined. Mechanical properties of several steels made by various ways of SPD such us multiple all-round forging, multi-axis deformation, accumulative roll-bonding, alternating bending and ECA pressing, are compared in relation to their structure.

11:45 AM
Superplastic Flow in Materials Processed by ECAP: *N. Balasubramanian*[1]; Terence G. Langdon[2]; [1]R V College of Engineering, All India Council of Techl. Educ., Mysore Rd., Bangalore 560 059 India; [2]University of Southern California, Aeros. & Mechl. Engrg. & Matls. Sci., 3650 McClintock Ave., Rm. OHE430G, Los Angeles, CA 90089-1453 USA

Equal-channel angular pressing (ECAP), when carried out optimally, produces submicron grain size, equiaxed grains and a significant fraction of high angle boundaries - conditions for high strain rate and/or low temperature superplasticity. Experimental results on superplastic flow after ECAP processing of various alloys will be reviewed. The stress and grain size exponents in the usual strain rate equation,the activation energy and the role of internal stress will be analyzed with a view to identifying the rate-controlling mechanisms.

12:00 PM
A Look into an Aluminum Nano-Composite Material for Advanced Aerospace Fastener Technology: *Patrick B. Berbon*[1]; Steven G. Keener[2]; [1]Rockwell Scientific, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA; [2]The Boeing Company, 2401 E. Wardlow Rd., MC C078-0533, Long Beach, CA 90807-5309 USA

Every aircraft produced today contains hundreds of thousands of fastened joints. These joints and the fasteners that connect them are perhaps the most common source of failure in aircraft structure. Therefore, it is imperative that advancements in fastener materials and designs be given the utmost consideration and attention to achieve increased joint performance and integrity. This paper presents the results of development efforts relating to an advanced processing technique, namely cryogenic milling, and its effect upon material microstructures and mechanical properties. These metallic nano-composites are found appropriate and important for potential fastener applications. By employing this nano-composite material processing technique in the production of aerospace fasteners, the fasteners' performance characteristics are enhanced and the manufacturing costs are reduced, by elimination of processing steps, e.g., thermal treatment, and/or reduction in unacceptable fastener installations.

12:15 PM
The Influence of Boundary Structure on the Mechanical Property in a Commercial Purity Aluminum: *Pei-Ling Sun*[1]; Ellen Kathleen Cerreta[1]; George T. Gray III[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

A commercial purity aluminum AA1050 was subjected to equal channel angular extrusion (ECAE) route A, the billet is not rotated, and C, rotated 180º, between extrusion passes respectively, to a von Mises strain of 8.4. The resulting microstructures have similar grain sizes but relatively different grain boundary structures. The microstructure processed by route A has ~70% high angle boundaries (HABs) while the route C microstructure has ~38% HABs. It was found that boundary structure plays an important role in the mechanical properties of the material because these boundaries act as a source and sink of dislocations. Mechanical tests conducted on these two microstructures at intermediate and dynamic strain rates at both 77 and 298K are compared with the results of material tested at quasi-static strain rates.

The Role of Technology in the Global Primary Aluminum Industry Today and in the Future

Sponsored by: Light Metals Division
Program Organizer: Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday AM Room: 2000
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

8:30 AM
Presentation 1: *Cynthia Carroll*[1]; [1]President and CEO of Alcan Primary Metal Group, Canada

8:55 AM
Presentation 2: *Truls Gautesen*[1]; [1]President of Hydro Aluminium Primary Metal, Norway

9:15 AM
Presentation 3: *Wayne Hale*[1]; [1]Executive Vice President of Alumina and Aluminium, Sual, Russia

9:35 AM Break

9:45 AM
Presentation 4: *Xiangmin Liu*[1]; [1]Vice President, Aluminum Company of China Limited (Chalco), Beijing, China

10:05 AM
Presentation 5: *Valery Matvienko*[1]; [1]Managing Director, Aluminium Division, Rusal, Russia

10:25 AM
Presentation 6: *Bernt Reitan*[1]; [1]Vice President of Alcoa Primary Products, USA

10:45 AM Break

11:00 AM Panel Discussion
Panel Moderator: James W. Evans, University of California, Dept. of Matls. Sci. & Mineral Engrg., Berkeley, CA 94720 USA

TUESDAY PM

6th Global Innovations Symposium: Trends in Materials and Manufacturing Technologies for Transportation Industries: Sheet Metal Processing

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), MPMD-Powder Materials Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee, MPMD-Surface Engineering Committee, MPMD/EPD-Process Modeling Analysis & Control Committee
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John E. Carsley, General Motors Corp, Warren, MI USA; Hamish L. Fraser, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210-1179 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Tuesday PM Room: 2009
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Mahmoud Y. Demeri, FormSys Inc., Northville, MI 48167-3427 USA; Amit K. Ghosh, University of Michigan, Dept. Matls. Sci. Engrg., Ann Arbor, MI 48109 USA

2:00 PM Invited
Improved and Affordable Hot Forming Technologies: *Amit K. Ghosh*[1]; [1]University of Michigan, Matls. Sci. & Engrg., 2300 Hayward St., Ann Arbor, MI 48109 USA

The need to use lightweight materials in aerospace and automotive applications have constantly created the need to innovate new processing methods to deal with rising manufacturing costs. The use of complex aluminum and magnesium alloys to replace steel, without the loss of design flexibility, has spawned a series of warm and hot forming techniques and new methods of material preparation. In this paper, some latest developments in this area will be reviewed, that help to take advantage of higher forming rate, lower forming temperature and use of complex constitutive behavior of materials inherent in such processing operations. (Work supported by National Science Foundation, DMR Grant.)

2:25 PM
Parametric Analysis for Warm Forming of Aluminum Blanks Using 2D FEA and DOE - Effects of Temperature Distribution, Friction and Speed: Peng Chen[1]; *Muammer Koç*[1]; [1]University of Michigan, S. M. Wu Mfg. Rsch. Ctr. & Dept. of Mechl. Engrg., Coll. of Engrg., Ann Arbor, MI USA

The effects of temperature distribution, speed, holding time and friction on warm forming performance are investigated for 5083-O (Al-Mg) sheet metal blanks. Combined isothermal/non-isothermal FEA with DOE tool is used to predict appropriate warm forming temperature conditions for deep drawing and two-dimensional stamping cases. In the ranges investigated (temperature: 25-250°C; speed: 2.5-5 mm/s; holding time: 1-3 sec; friction coefficient: 0.06-0.2), the formability of Al-5083 alloy is found to be greatly dependent on the temperature of the die and punch. To achieve increased degrees of forming, different temperature levels should be assigned to the corner and body of the die and punch in conjunction with a slow forming speed and combination of die-blank low friction and punch-blank high friction.

2:45 PM
The Effect of Lubrication on QPF Formability: *Paul E. Krajewski*[1]; [1]General Motors, R&D Ctr., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090 USA

Lubrication plays an important role in the ability to manufacture components by quick plastic forming (QPF). This occurs both through facilitating metal flow during deformation but also enabling part release from the die. The present paper provides two examples of using lubrication to prevent necking and thereby decrease the time required to make QPF components. The first example shows how the addition of milk of magnesia to boron nitride can modify friction and be used to reduce necking over sharp entry radii. The second example describes how tailored application of lubrication at entry radii can produce the same effect.

3:05 PM
Effect of Copper Additions on Superplastic Behavior of 5083 Aluminum Alloy: *Sooho Kim*[1]; Ravi Verma[1]; [1]General Motors, R&D Ctr., Matls. & Processes Lab., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA

Four 5083 aluminum alloys with varying copper and manganese concentrations were thermomechanically processed to a thickness of 2 mm. All rolled alloys exhibited highly refined grain structures with average grain sizes below 10 μm. Alloy A3, with the highest copper content (0.78%), showed the highest tensile elongation and lowest flow stress under most test conditions. This alloy exhibited peak strain-rate sensitivity at a strain-rate of 1x10-2 s-1, which is an order of magnitude higher than typical peak strain-rate for a base 5083 aluminum alloy, suggesting potential for faster forming with the copper-modified alloy. In biaxial pan forming tests, only the two copper-containing alloys (A2 and A3) produced fully formed pans. Of the two, A3 showed the least die-entry thinning. TEM characterization revealed that A3 had finer size, higher population density, and more uniform distribution of intermetallic particles than A2. It is believed that the finer particle size distribution of A3 is more effective in pinning grain-boundaries, and in turn is responsible for higher superplastic properties.

3:25 PM
Development of High Performance Alloy Sheets by Strip Casting: *Nack J. Kim*[1]; Sunghak Lee[1]; Sung S. Park[1]; Jung G. Lee[1]; [1]POSTECH, Ctr. for Advd. Aeros. Matls., San 31, Hyojadong, Pohang 790-784 Korea

Recently, strip casting has received a large attention due to its several advantages over conventional solidification processes such as continuous casting and direct chill casting. Strip casting combines solidification and hot rolling into one operation, thereby eliminating or reducing the hot rolling needed for conventionally produced ingots. Strip casting also offers much reduced cooling rates over conventional continuous casting or direct chill casting, resulting in the improvement of microstructural features. Such characteristics of strip casting are ideal for the development of high performance alloy sheets. The present paper discusses our efforts on developing the high performance alloys such as Mg and bulk metallic glass (BMG) alloys by strip casting. The impetus for the present research comes from the fact that there are virtually no wrought Mg and BMG alloys with respectable mechanical properties. The development of these alloys, particularly in sheet form, would greatly expand their applications in transportation industries. Microstructure and mechanical properties of strip cast alloys will be discussed with particular emphasis on the solidification behavior during strip casting.

3:45 PM Break

4:00 PM
Low-Cost Aluminum Tubes for Hydroforming Applications: *Alan A. Luo*[1]; Anil K. Sachdev[1]; [1]General Motors Research & Development Center, Matls. & Processes Lab., 30500 Mound Rd., MC 480-106-212, Warren, MI 48090 USA

Low-cost aluminum tubes made from twin-belt continuous casting sheet 5754-CC were evaluated for hydroforming applications, in comparison with extruded 6063 seamless tubes and seam-welded 5754-DC tubes (made from direct-chill cast aluminum sheet). The results show that the low-cost 5754-CC tubes offer acceptable dimensional tolerances and formability upon bending and hydroforming, as well as good mechanical properties after hydroforming. The microstructural evolution of the three materials during bending and hydroforming were closely investigated.

4:20 PM
Damage Tolerance and Durability of Glass Fiber Reinforced Aluminum Laminates for Aircraft Structures: *Guocai Wu*[1]; Jenn-

Ming Yang[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., Los Angeles, CA 90095 USA

Glass fiber reinforced aluminum laminate (GLARE) is a new class of fiber metal laminates (FML) consisting of alternating layers of thin aluminum sheets and unidirectional or biaxial reinforced adhesive prepregs of high strength glass fibers. Due to its outstanding fatigue resistance, high specific static properties, excellent impact resistance, good flame resistance and corrosion properties, it offers the aircraft structural designer a damage-tolerant, light-weight and cost-effective solution for advanced transport structural applications. More recently, GLARE has been selected for the upper fuselage skin structures of Airbus A380. However, the full potential of GLARE as aircraft primary structures has not been fully explored yet. More research and testing are necessary to generate adequate data to facilitate greater utilization of GLARE. One of the important structural integrity issues and concerns is the damage tolerance and durability for airworthiness of GLARE for aircraft structures. This paper presents the investigation results of damage tolerance and durability assessments of newly developed GLARE with cross-plied S-2 glass prepregs. An abroad impact testing program was performed, and the post-impact residual strength and fatigue behavior were extensively investigated to evaluate the damage tolerance of GLARE compared to the monolithic aluminum alloys. The crack propagation, damage progression and detection under static and dynamic loading after impact were investigated with microscopy, X-ray radiography, and by chemically removing outer aluminum layers.

4:40 PM
The Effect of Welding Parameters on the Microstructure and Microhardness of Resistance Spot Welded Galvannealed Steel Sheets: *Cherqueta Romeca Claiborn*[1]; Viola L. Acoff[1]; [1]University of Alabama, Metallurgl. Engrg., 129 Bevill Bldg., 126 7th Ave., Box 870202, Tuscaloosa, AL 35487-0202 USA

Resistance spot welding is used extensively in the automotive industry for joining low carbon steel components in high-volume and high rate production. However, spot welding often results in inconsistent weld quality. This study examines the effect of welding parameters on the microstructure and microhardness of resistance spot welded galvannealed steel. The specimens were welded at various percent currents while holding all other parameters constant. Light microscopy, microhardness testing, nanoindentation measurements, scanning electron microscopy, and x-ray diffraction were used to characterize the welded samples. Microhardness measurements taken across the weld nugget of each sample showed an increase in the microhardness values as the percent current increased. The purpose of this study is to investigate the cause of the observed increase in microhardness.

5:00 PM
Microstructural Evolution During Hemming: *John E. Carsley*[1]; [1]General Motors Corp., R&D, MC 480-106-212, 30500 Mound Rd., Warren, MI 48090 USA

Aluminum sheet is typically more difficult to hem than steel due to edge cracking. The difference has been attributed to the susceptibility of aluminum to strain localization during the hemming process. Recently developed techniques such as roller hemming, which have been used by automakers to assemble aluminum and steel components, have been shown to improve hemming of aluminum. The present study investigates microstructural evolution during both conventional and roller hemming to understand the effect of strain path on fracture during hemming/bending operations.

Alumina and Bauxite: Influences of Alumina on Smelter Performance

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Dag Olsen, Hydro Aluminium AS, Porsgrunn 3907 Norway; Travis Galloway, Century Aluminum, Hawesville, KY 42348 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday PM Room: 2005
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Tony Bagshaw, AMIRA International Ltd., W. Perth, Western Australia 6872 Australia

2:00 PM
SGA Requirements in Coming Years: *Stephen J. Lindsay*[1]; [1]Alcoa, Inc., Primary Metals Div., 300 N. Hall Rd., MS S-01, Alcoa, TN 37701-2516 USA

Refineries report various physical and chemical properties of Smelting Grade Alumina, SGA, on Certificate of Analysis data sheets. Without strong understanding of customer needs such posted properties can fall short of the true needs of smelters and downstream customers. An example is smelting efforts to improve excess fluoride control in pot bath, but with no specification or target for the variability of Na2O content. Downstream customers for aluminum conductor products require excellent electrical conductivity, but this does not always translate itself into alumina shipping limits for properties such as Cr2O3, MnO or V2O5. The author presents these and other examples for consideration during the joint sessions between Alumina & Bauxite and Aluminum Reduction Technology.

2:25 PM
Effects of the Vessel Loading and Unloading on Quality Variations (Particle Size) of Alumina: *Carl Behrens*[1]; [1]Hydro Aluminium Primary Metals, Alumina, Bauxite & Energy, PO Box 2560, Porsgrunn N-3907 Norway

Hydro Aluminium's smelter system is frequently using alumina from 10-15 different sources. In the feed back from the smelters, some sources are characterized as more variable than others. Also some individual shipments from otherwise well reputed qualities show occasionally variable behaviour when processed at the smelter. The sources characterized as variable, manifest them selves in higher pot instabilities such as settings for process control systems (transport systems, pot controls etc.) as well as inferior performance. The variability in smelter performance is most likely attributed to variations in particle size distribution (PSD), but an open mind is kept on this aspect. Segregation processes taking place in the silo systems used along the logistic chain in general generate the variations in PSD. Hydro Aluminium has initiated a number of studies focusing on how quality variations pass through the logistic chain from the alumina refinery to the individual pots in the pot room. The work presented in the present paper focus on the segregation processes taking place during the loading and unloading of alumina vessels. The variability is expressed as the standard deviation of a number of samples collected evenly over the entire shipment. The system is characterized as segregating when the standard deviation increases from the input to the output. In the opposite case, when the standard deviation is suppressed through the system, it is characterized as homogenizing. The present work demonstrates how the loading and un-loading operations of alumina vessels contribute to a reduction of the quality variations by a factor of 2-4 (ratio: Sin:Sout). Consequently the maritime transport system contributes to a homogenisation of the quality.

2:50 PM
Alumina Phase Distribution, Structural Hydroxyl and Performance of Smelter Grade Aluminas in the Reduction Cell: *James B. Metson*[1]; Margaret Hyland[2]; Tania Grouts0[1]; [1]University of Auckland, Light Metals Rsch. Ctr., PB 92019, Auckland New Zealand; [2]University of Auckland, Cheml. & Matls. Engrg., PB 92019, Auckland New Zealand

Recent developments in the phase analysis of smelter grade aluminas have shown that aluminas with nominally similar specifications differ significantly in their phase compositions, particularly in the relative amounts of the transition phases. These differences will affect alumina performance in the smelting cell, for example the potential for the alumina to generate HF. The transition phases are the likely source of residual structural hydroxyl, which recent experimental studies have linked to HF generation in the cell. It is of interest to understand the role of alumina structure, and especially the distribution of residual structural hydroxyl, which is strongly influenced by calcination conditions. In laboratory prepared gamma alumina the residual hydroxyl is thought to be located on the exterior of the alumina structure and at grain boundaries, but for smelter grade aluminas, the very rapid calcination conditions must influence this -OH distribution. Structural analysis studies on a range of SGAs suggest different capacities for HF generation which are not closely tied to the industry specifications for these materials.

3:15 PM
Adsorption/Entrainment of Fluoride in Smelting Grade Alumina: Surface Chemical Speciation and Adsorption Mechanism: *Neal R. Dando*[1]; [1]Alcoa, 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

Smelting grade alumina (SGA) is employed to scrub vapor-phase HF from the exhaust gases of aluminum smelters using either fluidized bed or injection scrubbers. As aluminum plants continue to increase production rates, the efficiency of the fluoride recovery process comes under elevated scrutiny, owing to the considerable cost of fume treatment centers. This elevates the need for an improved understanding of

the factors impacting fluoride evolution and recovery. Comparative characterizations of the short-range bonding and chemical speciation of lab and plant reacted smelting grade aluminas were employed to refine our understanding of the mechanism of fluoride adsorption on SGA surfaces. We have been able to identify and measure the relative populations of at least 6 different amorphous-state adsorbed and entrained fluorides in plant-reacted ore. The relative concentrations of these species reflect dry-scrubber operating conditions and pot-tending practices.

3:40 PM Break

3:55 PM
Exposures of Concern for Developing Occupational Asthma During Production of Primary Aluminium: *Y. Thomassen*[1]; N. P. Skaugset[1]; D. Ellingsen[1]; L. Jordbekken[1]; H. Notø[1]; [1]National Institute of Occupational Health, PO Box 8149, DEP., NO-0033 Oslo Norway

The quality of workroom atmospheres in potrooms have traditionally been monitored by measuring workers exposures to "total" dust and gases including particulate fluorides, hydrogen fluoride, sulphur dioxide and coal tar pitch volatiles (e.g. PAH's). Bronchial hyperresponsiveness (potroom asthma) is despite exposures below international occupational exposure limits (OEL's) for the contaminants mentioned above, still frequently occurring among workers. During recent years a high number of suspected potroom asthma and asthma like symptom cases have been reported by the Norwegian aluminium industry to the Labour Inspection Authority. Recently, a dose-response relationship has been suggested for fluoride exposures, but whether fluorides are the causative agent, co-agent or simply markers for causative agent(s) for potroom asthma, remains to be determined. To better understand the complex workroom composition in aluminium potrooms and the temporal variability in worker's exposure, new air monitoring strategies with the use of aerosol samplers for the health related aerosol fractions are required and has been included in an ongoing project (HAPPA) for the Nordic aluminium industry . Aerosol mass, total and water soluble fluorides, and beryllium in the respirable/thoracic/inhalable fractions in addition to HF and SO2 are measured in 10 different potrooms representing 6 smelters and Søderberg and Prebake-technologies. The use of personal direct reading instruments for SO2 and aerosols is used to obtain information about exposure variability. These exposure indicators may allow more complete description of workroom air contamination situation in Al-potroom. Preliminary results from this monitoring project will be presented.

4:20 PM
Ultrafine Particles at Workplaces of a Primary Aluminium Smelter: *Y. Thomassen*[1]; W. Koch[2]; D. Ellingsen[1]; N. P. Skaugset[1]; W. Dunkhorst[2]; L. Jordbekken[1]; P. A. Drabløs[3]; [1]National Institute of Occupational Health, PO Box 8149 DEP, N-0033 Oslo Norway; [2]Fraunhofer Institute of Toxicology and Experimental Medicine, Nikolai- Fuchs-Str. 1, D-30625 Hannover Germany; [3]Karmøy Plant Norsk Hydro, Håvik Norway

This study was aimed at measuring number concentration, number size distribution, particle morphology and ultrafine particles directly at the source during the process of changing anodes, and at locations representing typical mean exposure situations to aged smoke in plant site of an Al-smelter. The average "background" total number concentration is 20000 [particles/cm³] in Prebake area with concentration peaks an order of magnitude higher related to anode change operations. Søderberg technology produces more pollution with average concentrations of the order of 80000 [particles/cm³]. Time averaged size distribution exhibits a bimodal structure with a peak at or below 10 nm and a second peak at larger size (50-100 nm). Airborne particles show a variety of morphological structures including long thin fibers, chain like agglomerates and different forms of crystallites. Only a small fraction of the particles associated with the 10 nm peak of the number size distribution could be found on TEM-pictures indicating that these particles might be volatile. This study shows existence of elevated number concentrations of nanoparticles at workplaces in the primary aluminium industry. Main emission source of this particle size fraction in Prebake is open bath during anode changing. Nanoparticles were measured directly at the source but could also be identified as episodes of high number concentrations in the general "background" air in the Prebake production hall.

4:45 PM
Origins and Effects of Potroom Dust: *Margaret M. Hyland*[1]; Mark P. Taylor[1]; [1]University of Auckland, Light Metals Rsch. Ctr., PB 92019, Auckland New Zealand

Dust is defined as the fine particulate material that escapes confinement and causes a nuisance or a hazard in the workplace. This paper examines the origins of fine particulates and their impact on process stability and efficiency. This impact is complicated by the fact that it may not be observed on every cell in the line, because of the material's tendency to segregate in handling systems and is further confounded because once a disturbance has been set up in a cell, process conditions work against the containment of particulate and gaseous emissions. Recent work has also demonstrated that the fine fraction of crushed anode cover can have a dramatic effect on potline process stability and dust release. A study is now underway to investigate the origins of potroom dust, and the factors which control its generation and release into the environment. The role of alumina, both directly and through the disruption of reduction cells in the potline is of particular interest. Progress in understanding the origin of dust in the potrooms is reviewed, in light of these investigations.

5:10 PM
Alumina Dissolution and Sludge Formation Revisited: *Rudolf Keller*[1]; [1]EMEC Consultants, 4221 Roundtop Rd., Export, PA 15632 USA

Alumina added to the bath of an aluminum reduction cell does not immediately sink into and through the bath, because of its low bulk density (aside from temporarily freezing some bath). Bath penetrates into the bulk alumina, and in this process, g-alumina transforms into a-alumina and agglomerates are formed. Such agglomerate pieces may sink through the bath to the metal-bath interface. Provided their size and density are sufficient, accumulations of agglomerates may sink through the metal to form sludge at the carbon-metal interface. There can be conditions, however, where they remain at the bath-metal interface. Possible impacts of such accumulations are discussed along with the current distribution in the presence of bottom sludge and the formation of aluminum below the sludge.

5:35 PM
The Model of Dissolving and Heating of Alumina During its Supplying Through PF in the Program "Virtual Cell": *Alexander Berezin*[1]; [1]RUSAL Engineering & Techonology Center, Krasnoyarsk 660011 Russia

PFS (Point Feeding System) Control algorithms, taking into account parameters of bath alumina dissolving are necessary for increasing of cell efficiency, in particular for reducing of anode effects and cell output increasing. Modeling of dissolving process dynamics and alumina heating during its intake through the PFS is necessary for the definition of these algorithms. This work represents mutual solution of the following goals: -Modeling of the process of alumina dissolving on the basis of experimental data taking into account different qualities of alumina. -Modeling of thermal particles interaction and alumina agglomerates with the bath. -Modeling of time and alumina agglomerates settlement quantity at the interface bath-metal. The models are realised in the program "Visualised cell". Test calculations are fulfilled. The results of technical data alteration (cell voltage, bath temperature, overheating, alumina concentration, settlement quantity etc.) are presented during the work of PF.

Aluminum Reduction Technology: Cell Development & Operations - Part 2

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tor Bjarne Pedersen, Elkem Aluminium ANS, Farsund 4551 Norway; Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday PM Room: 2003
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Claude H. Vanvoren, Alcan, Primary Metal Grp., Voreppe 38341 France

2:00 PM
A Case Study on Life Cycle Analysis of Copper Anode Bar: *Jyoti Mukhopadhyay*[1]; Y. V. Ramana[2]; Rajnish Kumar[2]; [1]Jawaharlal Nehru Aluminium Research Development and Design Centre, Amravati Rd., Wadi, Nagpur, Maharashtra 440 023 India; [2]Hindalco Industries Limited, R&D, PO Renukoot, Renukoot, Uttar Pradesh 231 217 India

During aluminium production, frequent failure of copper anode bar was observed. The average life cycle of the bar was found to reduce from 50 cycles to 20 cycles during a defined time span. While reduction, it was observed that both stiffener and the bar were continuously exposed to flue gases. Failures in the vicinity just above the MS stiffener take place due to gradual reduction in cross-sectional area of the

Cu-bar as a result of oxidation, pitting and crack formation. Microstructural analysis also reveals that the presence of pits/voids are also responsible for such failure. In fact, the failure was attributed mostly due to the intergranular mode. In order to prevent such failures, a few important remedial measures were undertaken. Among them, stringent quality control of the copper metal before and after the fabrication of bar as well as also increasing the height of the stiffener could well be explored.

2:25 PM

Application of Aluminum-Copper Bonded Sheet in Aluminum Reduction Cells: *Kayron F. Lalonde*[1]; Mark D. Ohlswager[2]; [1]Alcoa, Inc., Primary Metals, PO Box 472, Rockdale, TX 78664 USA; [2]Elkem Aluminium, Hoffsveien 65B, PO Box 5211 Majorstuen, Oslo No-0303 Norway

Use of aluminum-copper bonded sheets on the anode riser joint connection of prebake aluminum reduction cells has eliminated the occurrence of high voltage drops across these joints. The sheets were installed between the solid aluminum leaves of the anode buswork and the copper tabs of the anode riser-flexibles bolted to the moveable anode bus. Prior to bimetal sheet installation, half of the riser joints would increase in voltage over time, as much as 350 millivolts, requiring expensive disconnecting, cleaning and remaking of the joint connections. Since the bimetal sheet installation began two years ago, all new joints have remained at normal voltage, resulting in a significant reduction of the voltage drop external to the cell.

2:50 PM

The Impact of Anode Cover Control and Anode Assembly Design on Reduction Cell Performance - Part 2: *Evan W. Andrews*[1]; Mark P. Taylor[2]; Greg L. Johnson[1]; Ian Coad[1]; Geoff P. Brookes[1]; [1]Boyne Smelters, Boyne Island, Queensland Australia; [2]University of Auckland, The Light Metals Rsch. Ctr., New Zealand

Reduction line amperages have been increasing around the world in recent years as companies seek to increase the return from existing assets. The key aspects of anode cover quality and anode hanger design on the cell top heat balance were exemplified in Part 1 of this work, published in TMS Light Metals 2004. The anode assembly design criterion, which is now most critical for the stability of the cell is the dissipation of sufficient heat from the assembly and the cover to maintain component temperature and overall cell heat balance. This paper completes last year's work, with presentation of an operational anode cover control loop that has permitted tighter control of the heat balance in cells. The importance of the anode assembly design on the cell operating heat balance window and reduction of anode problems is also considered, especially for smelters approaching their upper amperage and heat balance limits. Plant data is presented to demonstrate and support the practical aspects of the work.

3:15 PM

Fluoride Evolution/Emission from Aluminum Smelting Pots: Impact of Ore Feeding and Cover Practices: *Neal R. Dando*[1]; Robert Tang[2]; [1]Alcoa, 100 Techl. Dr., Alcoa Ctr., PA 15668 USA; [2]Alcoa, Smelting Div., Eastalco Works, Frederick, MD 21703 USA

Dry scrubbers use smelting grade alumina (SGA) to capture vapor-phase fluoride from the exhaust gases of aluminum smelters. The reacted SGA from the scrubbers is then fed back to the pots to recover the lost fluoride. A range of smelter-owned factors such as pot chemistry, operating practice, pot-tending practice, and ore feeding dramatically affect the dynamics of this recovery process. This presentation will discuss real-time measures of the impact of ore feeding practices and tending practices on fluoride emission and evolution from aluminum smelting pots. The dynamics of fluoride release from reacted SGA directly fed to the pots was studied by varying the rate and dynamics of the ore feed cycle. Of particular interest is the relative magnitude of pot-tending practices on emission and evolution.

3:40 PM Break

3:55 PM

Anode Cover Material and Bath Control: *Siegfried Wilkening*[1]; Pierre Reny[2]; Brian Murphy[2]; [1]Hydro Aluminium T & P, PO Box 2468, Bonn 53014 Germany; [2]Aluminery Alouette Inc., Case postale 1650, Sept Iles, Quebec G4R5M9 Canada

The various types of recycled bath material and anode cover material are defined in clear terms. The basic properties of primary anode cover material are described and discussed as well as their testing and control in the laboratory. Further important aspects are dealt with, such as the handling, crushing, grinding, classification and storage of recycled bath materials, followed by the preparation and control of a suitable and consistent anode cover material in modern potlines. Examples of the equipment generally used in bath treatment centres, or which may be employed in the future, are given.

4:20 PM

CFD Modeling of the Fjardaal Smelter Potroom Ventilation: *Jon Berkoe*[1]; Philip Diwakar[1]; Lucy Martin[1]; Bob Baxter[1]; Mark Read[1]; Patrick Grover[2]; Don Ziegler[2]; [1]Bechtel M&M, N. America, 1500, rue Université, Bureau 910, Montréal, Québec H3A 3S7 Canada; [2]Alcoa, 6603 W. Broad St., Richmond, VA 23230 USA

The Fjarðaál Smelter potline buildings are designed to achieve ventilation of heat and fugitive contaminants using a system based on natural air circulation. The design of the potline buildings is based on Alcoa's Aluminerie Deschambault smelter facility, located in Deschambault Quebec, Canada. The Fjarðaál Smelter Project potline buildings are longer, with reduced spacing between buildings and are situated on a sloping site adjacent to a fjord located in eastern Iceland. The Fjarðaál Project is faced with additional unique factors like local terrain, high wind speed, and multiple approach wind directions - that make the predictability of the ventilation system performance more complex. To help guide the ventilation design, computational fluid dynamics (CFD) modeling was employed. The CFD analysis utilized state-of-the-art capabilities to capture in detail the velocities, temperatures, pressures, and pollutant concentrations inside and outside the buildings. The model was validated against smoke tests conducted at the Deschambault smelter. The model demonstrated that the ventilation system performance is relatively unaffected by the terrain and winds, implying that the claustra wall design is very effective. Pressure gradients can cause locally non-uniform flow patterns in the potroom but these effects are manageable within the system's performance constraints.

4:45 PM

Egyptalum Experience in Operating End to End Prebaked Cells with Over 200 kA: M. M. Ali[1]; M. El-Ghonimy[1]; F. M. Ahmed[1]; Z. A. Bassiony[1]; *S. M. El-Raghy*[2]; [1]Aluminium Company of Egypt (Egyptalum), Nage-Hammadi Egypt; [2]Cairo University, Faculty of Engrg., Cairo Egypt

Egyptalum believes in continuing development. Improvements in Soderberg cells have resulted in increase of current efficiency from project design (84%) to today's level of more than 88%. A major milestone in development was the conversion of Soderberg cells into prebaked cells. This achievement has been reported in JOM in May 2003. It has gone through three phases, namely: familiarization, mathematical models and actual design, fabrication and operation. As expected, early stages of operation of the home-made 200 kA prebaked cells faced some difficulties. Overcoming these difficulties was followed by converting Soderberg line #5 into prebaked cells. Another goal was to increase the operating current. This development was achieved through 6 steps: 203 in the year 1997 up to 210 in the year of 2003. In each step, unsteady operation needed careful changes in operating parameters. Thus, many factors have been changed to match the new situation. Cell stability was one of these factors to be checked each time. Today at Egyptalum, 210 kA prebaked home-made cells are operating at a steady state showing satisfactory norms. Current efficiency reached 95%; energy consumption is 13870 k w hr/ton Al; also carbon consumption has come below 410 kg/ton Al.

Applications and Fundamentals of High Aspect Ratio Nanomaterials: Applications of Carbon-Based and Inorganic Nanostructures

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Nanomaterials Committee
Program Organizers: Jud Ready, Georgia Tech Research Institute - EOEML, Atlanta, GA 30332-0826 USA; Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; Lourdes G. Salamanca-Riba, University of Maryland, Materials Science and Engineering Department, College Park, MD 20742-2115 USA; Nagarajan Valanoor, Forschungzentrum Juelich, IFF and Institute for Electronic Materials, Juelich, Germany D52425

Tuesday PM Room: 3018
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Apparao M. Rao, Clemson University, Physics, Clemson, SC 29634-0978 USA; Nagarajan Valanoor, Forschungzentrum Juelich, Juelich D52425 Germany; Seung H. Kang, Agere Systems, Allentown, PA 18109 USA

2:00 PM Opening Remarks

2:05 PM Invited
Novel One Dimensional Nanostructures: *Meyya Meyyappan*[1]; [1]NASA, Ctr. for Nanotech. - Ames Rsch. Ctr., MS 229-3, Moffett Field, CA 94035 USA

Carbon nanotubes (CNTs) exhibit a combination of remarkable mechanical properties and unique electronic properties and thus offer significant potential for a wide range of applications. In this talk, our recent results on growing very well-aligned, individual, freestanding nanotubes by plasma CVD and development of bio and chemical sensors will be described. It is possible now to grow vertically oriented, well-aligned nanowires of semiconducting materials such as silicon, germanium and high temperature oxides. These inorganic nanowires exhibit very interesting electronic and optical properties compared to their two dimensional thin film cousins and are being investigated for device, laser and sensor applications. Growth of ZnO, InO and other nanowires, characterization and development of vertical transistors will be discussed.

2:35 PM Invited
Rational Design and Fabrication of High-Quality Inorganic Semiconductor Nanostructures: *L. Bock*[1]; [1]Nanosys, Inc., 2625 Hanover St., Palo Alto, CA 94304 USA

Nanosys synthesizes inorganic semiconductor nanostructures in the form of nanorods, nanowires and more complex shapes, such as cones, teardrops and tetrapods. Using computer modeling and synthetic methods, we rationally design inorganic semiconductor nanostructures with desired composition, size, shape, crystal structure, doping and surface chemistry characteristics, and successfully fabricate these materials every time. In addition to controlling the composition and structure of these nanomaterials, we can even change the composition as they are grown, forming nano heterostructures containing nano-sized heterojunctions. These junctions can be made atomically sharp, and defect-free; allowing the production of complex, high-performance electronics integrated directly within a single nanostructure. In contrast to traditional microelectronics, elements such as p-n diodes, p-i-n diodes, LEDs, bipolar transistors, etc. can be constructed chemically from the bottom up as the nanostructures are grown.

3:05 PM Invited
Recent Progress in Carbon Nanotube Field Emission Display: *Jong Min Kim*[1]; In Taek Han[1]; YongWan Jin[1]; Jun Hee Choi[1]; Jung Hee Lee[1]; Jae Eun Jung[1]; Young Joon Park[1]; Deuk Seok Chung[1]; Sang Hyun Park[1]; Hang Woo Lee[1]; [1]Samsung Advanced Institute of Technology, Matls. Lab, Keyheung NongSeoRi San 14-1, YongIn, KyoungKi 449-712 Korea

Field Emission Display (FED) having carbon nanotube (CNT) emitters has been fabricated for several years in Samsung. The phase shift from metal electron emitter to CNT has given us great success not only in the business field but also in the academic field. In this presentation, the parameters to enhance field emission property of CNT will be discussed. The study and precise control of CNTs in the form of paste and CVD grown thin films gave us much informations to use CNTs as a better electron emitter. Four components in CNT paste are CNT, frit, filler, and vehicle. The pretreatment of CNT, using proper frit and vehicle, and modifying frit resulted 10 times larger emission current and half value of operating bias. The patterning process of CNT paste has been developed and the results of hole filling process will be addressed. The CNTs are also directly grown on the FED structure by CVD method. The emission properties of CVD grown multiwalled CNT are similar with those of physically synthesized single wall CNTs. Finally our recent large screen demonstration image of FED will be shown.

3:35 PM Break

4:00 PM Invited
High Performance Nanotube/Fiber Field Emitters for Microwave Applications: *K. B.K. Teo*[1]; E. Minoux[2]; O. Groening[3]; L. Gangloff[2]; J.-P. Schnell[2]; D. Dieumegard[2]; F. Peauger[2]; R. G. Lacerda[1]; P. Legagneux[2]; D. G. Hasko[4]; G. A.J. Amaratunga[1]; W. I. Milne[1]; [1]Cambridge University, Dept. of Engrg., Cambridge UK; [2]Thales Research and Technology, France; [3]Federal Laboratories for Materials Testing and Research, EMPA Thun, Switzerland; [4]University of Cambridge, Microelect. Rsch. Ctr., UK

Multiwalled carbon nanotubes/fibers (CN) are pursued here as field emission electron sources because of their whisker-like shape, high aspect ratio, high conductivity, thermal stability and resistance to electromigration. Despite these favourable properties, it is often difficult to integrate this material into devices because of the lack of control in the synthesis of this material. We report the use of plasma enhanced chemical vapour deposition to provide ultimate control of the CN structure such as their alignment, individual position, diameter, length and shape. The CN produced from this process exhibit excellent structural uniformity, with typical standard deviation in the diameter and height of 4.1% and 6.3% respectively. Using such well controlled structures, we show it is possible to predict the electrical characteristics of the CN emitters as an individual electron source or as an array. Thus, it is now possible to build designer cathodes with predictable characteristics, and also to determine optimised arrangements for these cathodes. We demonstrate high current densities of 1A/cm^2, under DC and 1.5GHz direct modulation, from CN cathodes. These CN cold cathodes offer considerable weight and size savings over conventional hot cathode cathodes used in microwave applications (eg. SATCOM, radar).

4:30 PM Invited
Electron Filtering by Multiply-Connected Carbon Nanotubes and Field Emission of Double-Wall Carbon Nanotubes: *Jisoon Ihm*[1]; [1]Seoul National University, Sch. of Physics, Seoul Korea

We have investigated the electron transport in multiply-connected metallic carbon nanotubes within the Landauer-Buttiker formalism. Quasibound states are coupled to the incident Π* states and give rise to energy levels of different widths depending on the coupling strength. In particular, donor-like states originated from heptagonal rings are found to give a very narrow level. Interference between broad and narrow levels produces Fano-type resonant backscattering as well as resonant tunneling. Over a significantly wide energy range, almost perfect suppression of the conduction of Π* electrons occurs, which may be regarded as filtering of particular electrons. We have also performed first-principles pseudopotential calculations for the field emission from carbon nanotubes by solving the time-dependent Schroedinger equations under high applied voltage. Both capped and open-ended geometries are considered. Carbon atoms bridging two walls are assumed to exist in the open-ended double-wall tubes. The outer wall screens the external field very effectively in general. Double-wall nanotubes with at least one metallic wall are shown to produce more stable emission current than single-wall nanotubes. Implications to the performance of the actual field emission display developed in the industry are discussed.

5:00 PM
The Effect of Structural Change and Ni Doping on Hydrogen Storage Properties of Carbon Nanotubes: *Jai-Young Lee*[1]; Hyun-Seok Kim[1]; Kyu-Sung Han[1]; Min-Sang Song[1]; Min-Sik Park[1]; [1]Korea Advanced Institute of Science and Technology, Matls. Sci. & Engrg., 373-1 Guseong-dong, Yuseong-gu, Daejeon 305-701 Korea

Hydrogen storage properties in carbon nanotubes(CNTs) were investigated from the view points of not only physical hydrogen molecules adsorption in nano-hole but also chemical hydrogen adsorption on graphite surface. CNTs with closed or open capped were studied through hydrogen thermal desorption technique equipped with gas chromatograph. The precise analysis on thermal desorption spectra on CNTs with closed structure showed that hydrogen gas was released at three major temperature ranges such as 100-230K, 290-350K, and 600-625K, where the evolved hydrogen amount were about 1.65wt%, about 0.64wt%, and about 0.03wt%, respectively. However, in case of

open CNTs, the evolution peak around 290-350K was highly developed (about 1.9wt%). And Ni-doping(dispersion) effect on hydrogen storage properties were investigated comparatively. The metal nanoparticles were homogeneously dispersed using incipient wetness impregnation method. Hydrogen desorption spectra of MWNTs with Ni nanoparticles showed that about 2.8wt% of hydrogen was released in the range of 340-520K.

Arsenic Metallurgy: Fundamentals & Applications: Removal of Arsenic and its Precipitation from Process Streams II

Sponsored by: Extraction & Processing Division, EPD-Copper, Nickel, Cobalt Committee, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, LMD/EPD-Recycling Committee
Program Organizers: Ramana G. Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; V. Ram Ramachandran, Scottsdale, AZ 85262-1352 USA

Tuesday PM Room: 2014
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Shijie Wang, Phelps Dodge Refining Corp, El Paso, TX 79915 USA; Larry G. Twidwell, Montana Tech of University of Montana, Sch. of Mines & Engrg., Butte, MT 59701 USA

2:30 PM
Biological Removal of Arsenic from Tailings Pond Water at Canadian Mine: *Tina Maniatis*[1]; Tim Pickett[1]; [1]Applied Biosciences, PO 520518, 265 Crossroads Sq., Salt Lake City, UT 84152 USA

Applied Biosciences has developed a biological technology for removal of arsenic, nitrate, selenium, and other metals from mining and industrial waste waters. The ABMet® technology was implemented at a closed mine in Canada for removing arsenic from tailings pond water. The system included six bioreactors that began treating water in the spring of 2004. Design criteria incorporated a maximum flow of 567 L/min (150 gallons per minute) and temperatures ranging from 10°C to 15°C. Influent arsenic concentrations range from 0.5 mg/L to 1 mg/L. The ABMet® technology consistently removes arsenic to below detection limits (0.02 mg/L). Data from the bench scale testing, and the full scale system will be presented, as well as regulatory requirements site specific and challenges.

2:55 PM
Arsenic Removal from Contaminated Waters: *Jack Adams*[1]; Ximena Diaz[1]; Jan D. Miller[1]; Terrence Chatwin[2]; [1]University of Utah, Dept. Metallurgl. Engrg., 1335 S. 1460 E., Salt Lake City, UT 84112-0114 USA; [2]University of Utah, Utah Engrg. Experiment Sta., 1495 East 100 S., Salt Lake City, UT 84112 USA

Arsenic is a contaminant at 781 of 1,430 sites identified on the National Priorities List and in mining and mineral processing wastewaters, smelter wastes, and sites for manufacture of semi-conductors, petroleum products, wood preservatives, animal feed additives, and herbicides. Arsenic affects ~4,100 municipal water systems nationwide and is difficult to treat to 10 ppb levels. Adsorptive media can remove up to 99% of arsenic from drinking water at costs for POU/POE applications of $0.20/0.02/gal. Full-scale microbial arsenic removal/stabilization treatment costs of $0.10/1,000 gal have been demonstrated for mining and ground waters to 2ppb levels. Processes using magnetically activated carbon and bacteria/biopolymers; separately and combined (BIOMAC) have been demonstrated to treat high levels of Arsenic III and V to 2 ppb under wide water chemistry ranges. Additional BIOMAC benefits include other heavy metal removal, such as lead, copper, zinc, fluoride, selenium, and improvement in taste and odor.

3:20 PM
Arsenic Removal by Electrocoagulation Technology in the Comarca Lagunera Mexico: *Jose R. Parga*[1]; David L. Cocke[2]; Jesus L. Valenzuela[3]; Ventura Valverde[1]; Jewel A. Gomes[2]; Mehmet Kesmez[2] Hector A. Moreno-Casillas[2]; [1]Instituto Tecnologico de Saltillo, Matls. Sci., V. Carranza 2400, Saltillo, Coahuila 25280 Mexico; [2]Lamar University, Gill Chair of Chmst. & Cheml. Engrg., PO Box 10022, Beaumont, TX 77710 USA; [3]University of Sonora, Chmst., Zona Centro, Hermosillo Sonora, Sonora Mexico

In some parts of the Comarca Lagunera with population of about 2.5 million people, situated in the central part of northern México, chronic arsenic poisoning is endemic and severe adverse effects attributed to Arsenic exposure have been reported. There are several methods available for removal of arsenic from well water in large conventional treatment plants, however a very promising electrochemical treatment technique that does not require the addition of chemicals or regeneration is Electrocoagulation (EC). The EC operating conditions are highly dependent on the chemistry of the aqueous medium, conductivity and pH. In this study, Powder X-ray Diffraction, Scanning Electron Microscope, and Transmission Mössbauer Spectroscopy were used to characterize the solid products. Finally the results of this study suggest that the presence of maghemite and magnetite particles can be used to remove arsenic (III) and arsenic (V) in a field pilot plant scale study yielded 99% removal from groundwater.

3:45 PM
Removal of Arsenic from Soda Matte Process Solutions-An Overview: *Shijie Wang*[1]; [1]Phelps Dodge Refining Corp, 850 Hawkins Blvd., El Paso, TX 79915 USA

Soda Mattes are normally generated by treating the rough copper dross with sodium to produce a high grade copper matte which is actually a mixture of sodium sulfide, lead metal, and lead and copper sulfides. Since about 60% of the arsenic is solubilized during the hydrometallurgical treatment, the solution from the process has to be treated for arsenic removal. In this paper, the treatment scheme is evaluated. A mass balance of the process is presented. The characteristics of the process control and the efficiency of arsenic removal are discussed.

Automotive Alloys 2005: Session I

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: Subodh K. Das, Secat, Inc., Coldstream Research Campus, Lexington, KY 40511 USA

Tuesday PM Room: 2006
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Subodh K. Das, Secat Inc., Lexington, KY 40511 USA; Zhong Li, Commonwealth Aluminum, Lexington, KY 40511 USA

2:00 PM
Application of 5182 for Automotive Parts: *Gyan Jha*[1]; W. Yin[2]; [1]Arco Aluminum, 9960 Corp. Campus Ste. 3000, Louisville, KY 40223 USA; [2]SECAT, 1505 Bull Lea Rd., Lexington, KY 40511 USA

Aluminum alloys have been increasingly used in the automotive industry due to their excellent properties,high strength and corrosion resistance. The formability of these alloys is critical for automotive applications. The Forming Limit Diagram (FLD) allows for an opportunity to determine process limitations in sheet metal forming and is used as an input to aid in the analysis of the stamping characteristices of sheet metal materials. This paper will investigate the application of 5182 sheet for Automotive applications by analyzing the of FEA part simulations that have utilized the FLD for 5182.

2:25 PM
Deformation-Induced Surface Roughening in 6022-T4 Al Sheets: *Y. S. Choi*[1]; H. R. Piehler[2]; A. D. Rollett[2]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Carnegie Mellon University, Dept. of Matls. Sci. & Engrg., Pittsburgh, PA 15213 USA

The surface roughening behavior of 6022-T4 Al sheets deformed in plane-strain tension was thoroughly studied using multi-scale approaches. Various experimental techniques and analytical tools were used to characterize surface roughening and its evolution. The results showed the strong anisotropy in development of the characteristic roughening patterns, and their interrelations among different scales of the observation windows. The roughening patterns undesirable from an engineering aspect were identified. The through-thickness roughening behavior was also investigated, and the results showed a symmetry in roughening pattern between top and bottom surfaces, depending on the pulling direction, the size of the observation window and the level of the strain. Various efforts were made to clarify causes of surface roughening, particularly focusing on the correlation between the orientation texture and the surface roughness, and some new and useful results will be presented.

2:50 PM
Microstructure and Property Inhomogeneity of Cast Aluminum Wheels: *Robert Shang*[1]; Naiyi Li[2]; William J. Altenhof[1]; Henry Hu[1]; [1]University of Windsor, Mechl., Auto. & Matls. Engrg., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Ford Motor Com-

pany, Rsch. & Advd. Engrg., 2101 Village Rd., Dearborn, MI 48124 USA

The automotive industry has been attracted to the application of aluminum in road wheels, as aluminum wheels with reduced unsprung weight offer better vehicle handling and drivability. In an effort to better understand engineering functionality and performance of aluminum wheels, a study on structure inhomogeneity of cast A356 aluminum wheels has been carried out. Samples for mechanical properties and microstructure analysis were sectioned from various locations of the aluminum wheels. The microstructures have been investigated by means of optical metallography and scanning electron microscopy (SEM). Eutectic phase and intermetallic compounds are identified using energy dispersive X-ray analysis (EDS). The microstructure analysis reveals the dependency of the dendrite arm spacing (DAS) on the section thickness of the sampling regions. Tensile properties were obtained from specimens extracted from the hub, spoke and rim of the wheels. The results indicate that the tensile strength, and in particular, elongation for the sampling locations vary considerably. The property inhomogeneity is primarily attributed to inhomogeneous microstructure in the wheels resulting from the nonuniform cooling occurred during solidification.

3:15 PM

Microstructure Evolution During the Annealing of Cold Rolled AA6111: *Johnson Go*[1]; Matthias Militzer[1]; Warren J. Poole[1]; Mary A. Wells[1]; [1]University of British Columbia, Ctr. for Metallurgl. Process Engrg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

The heat-treatable Al-Mg-Si-Cu alloy AA6111 is one of the main aluminum choices for automobile sheet skin due to its excellent combination of paint bake hardening response and high formability. In the commercial processing for this alloy, fine and closely spaced precipitates that developed during hot rolling and subsequent coiling processes can retard the evolution of recrystallized microstructure by suppressing the movement of high angle boundaries. On the other hand, large primary Fe-containing constituent particles can promote recrystallization via particle-stimulated-nucleation (PSN). In an effort to clarify the interactions between these microstructural processes, a series of isothermal annealing experiments have been conducted on cold rolled AA6111 with systematically varied precipitation states. The evolution of microstructure, both in terms of the recrystallized grain size distribution and spatial distribution of second phases are characterized using a variety of experimental techniques including EBSD grain mapping, electron channelling contrast in SEM, optical microscopy and resistivity measurements. The results indicate that the recrystallization kinetics are severely retarded irrespective of prior aging conditions.

3:40 PM

Particle Cracking Damage Nucleation in Wrought Aluminum Alloys: *Y. Mao*[1]; Joel Harris[1]; A. M. Gokhale[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA

Damage nucleation in numerous ductile Al-alloys involves cracking of brittle inclusions; subsequent void growth around the cracked inclusions and the void coalescence leads to the global fracture. Consequently, it is of interest to quantify particle/inclusion cracking in wrought Al-alloys. In the past, such quantitative microstructure characterization involved manual detection of the cracked particles using optical or scanning electron microscopy, which is quite laborious and inefficient. We present a novel digital image analysis technique for automatic detection of cracked particles and cracks and subsequent measurements of their geometric attributes such as number density, and size, shape, and orientation distributions. The technique is presented through its applications to characterization of cracking of Fe-rich intermetallic inclusions in 7075, 6061, and 5086 Al-alloys.

4:05 PM

Use of EBSD to Quantify the Microstructural Damage in Aluminum Alloys Under Monotonic Loading: *Joseph A. Querin*[1]; J. A. Schneider[1]; Mark F. Horstemeyer[1]; [1]Mississippi State University, Dept. of Mechl. Engrg., Mississippi State, MS 39762 USA

A high degree of success has been achieved in the use of damage modeling for predicting the durability and reliability of cast components. Cast components typically have a high void volume fraction due to porosity. Under the stress triaxiality conditions present in thick walls, the porosity voids grow and coalease. Implementation of this damage modeling technique for predicting durability and reliability in rolled sheet components presents new challenges. Rather than damage accumulation originating at porosity voids, damage accumulation originates within shear bands in deformed aluminum sheet metal. The initial texture and evolving texture in sheet metal affects the shear band formation and is an important factor in material failure. How this texture evolves will be affected by grain size and grain boundary orientations. Electron backscattered diffraction EBSD is useful for characterizing texture, but it may also be used to characterize shear banding and damage progression. Results from our experiments will illustrate how the microstructural changes in monotonically loaded tensile specimens can provide the damage evolution data necessary for predictive modeling of aluminum sheet metal components.

4:30 PM

Microstructure, Texture and Mechanical Properties of Continuous Cast AA5083 and 5182 Aluminum Alloys: *Tony Zhai*[1]; Jin Li[2]; Xiyu Wen[3]; [1]University of Kentucky, Dept. of Cheml. & Matls. Engrg., 177 Anderson Hall, Lexington, KY 40506 USA; [2]Beijing Jiaotong University, Dept. of Civil & Environmental Engrg., Beijing China; [3]University of Kentucky, Ctr. for Al Tech., 1505 Bull Lea Rd., Lexington, KY 40511 USA

The recrystallization textures of cold-rolled continuous cast AA 5083 and 5182 aluminum alloys with and without prior heat treatment were investigated by X-ray diffraction. The results showed that the prior heat treatment strongly affected the recrystallization texture of CC AA 5083 aluminum alloy. The recrystallization texture of CC AA 5083 aluminum alloy without prior heat treatment was characterized by a major strong {113}<110> component and a minor R component, while the recrystallization texture of the alloy with prior heat treatment consisted of a weak R component. The tensile properties and their anisotropy of the 5083 and 5182 alloys were also studied.

4:55 PM

Development of Twin Roll Cast AA 6016 for Automotive Applications: *Murat Dundar*[1]; *Ozgul Keles*[1]; Necmi Dogan[1]; Bilal Kerti[1]; Gerhard Anger[2]; [1]Assan Aluminum, R&D, E5 Karayolu 32. km. Tuzla, Istanbul 81700 Turkey; [2]AMAG Rolling GmbH, PO Box 32, Ranshofen A-5282 Austria

Aluminum alloys employed for inner and outer panel applications of automotive industry necessitate alternative solutions to implement some improvements on mechanical properties and formability as well as a cost efficient production. Continuous interest to twin roll casting technology, in recent years, originates from cost effective nature of the casting technique and some unique features of the materials cast in comparison to conventional casting. Very high solidification rate at the roll gap introduces unique micro structural features that will further contribute mechanical properties and formability performance, with the application of appropriate downstream processing route. In present study, detailed micro structural characterization studies were conducted on the as-cast samples of twin roll cast AA6016. Casting parameters were optimized to tailor micro structural features at the surface and through the thickness. Further on results for specific automotive applications as car body sheet and structural sheet will be discussed.

5:20 PM

Processing of AA6082 Profiles for Automotive Applications: *Yucel Birol*[1]; Osman Cakir[1]; Tanya Aycan Baser[1]; Fahri Alageyik[1]; Erdogan Bengu[2]; [1]Marmara Research Center, Gebze, Kocaeli 41470 Turkey; [2]Asas Aluminum, Akyazi, Adapazari Turkey

Among several alloys within the Al-Mg-Si system, AA6082 alloy is regarded as a higher strength alloy and is used for sections requiring tensile strengths exceeding 300 MPa in the automotive industries. This alloy needs to be processed in an optimum way in order to meet the everincreasing market demand for improved performance particularly with regard to their strength. Factors such as alloy chemistry, microstructure or the extrusion process parameters have a big impact on the strength of the final product. For high strength extruded profiles, processing must assure either fine recrystallized grains or a uniform unrecrystallized structure as intermediate partially recrystalized structures with coarse grains particularly near the profile surface, are detrimental to strength, toughness, formability and weldability of these products. The present work was carried out to investigate the effect of Mn content, the quench rate, the solution temperature, the quenching practice and the storage time before artificial ageing on the final properties of 6082 hollow cylindirical profiles for antivibrational elements used in automotive industry where high strength and dimensional stability is critical.

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Properties of Al-Si-Mg Automobile Body Sheet by Twin Roll Casting: *Hideyuki Uto*[1]; Hiroki Esaki[1]; Yoshio Watanabe[1]; Tadashi Minoda[1]; Kaoru Ueda[1]; Kazuhisa Shibue[1]; [1]Sumitomo Light Metal Ind., Ltd., R&D Ctr., 1-12 Chitose 3, Minato-ku, Nagoya, Aichi Prefecture 4558670 Japan

Properties of Al-1.0%Si-0.4%Mg alloy sheets for automobile body by twin roll casting (TRC sheets) were investigated. Additionally me-

chanical, forming and corrosion properties of the Al-Si-Mg alloy containing ferrous impurity from 0.15 to 1% were estimated in order to develop the material recycling technology for body panels. After cold rolling and heat treatment the mechanical properties of TRC sheets are almost the same as that of the sheets by DC casting (DC sheets). At deep-drawing test the forming height of TRC sheets is almost the same. However, both the forming height at stretching test and the bendability for hemming of TRC sheets is lower than that of DC sheets. Moreover, the surface quality of TRC final products was evaluated. As a result it is clarified ripple mark affects filiform corrosion sensitivity.

Beta Titanium Alloys of the 00's: Composites and Processing

Sponsored by: Structural Materials Division, SMD-Titanium Committee

Program Organizers: Rod R. Boyer, Boeing Company, Metall./6-20J1, Seattle, WA 98124-2207 USA; Robert F. Denkenberger, Ladish Co., Inc., Cudahy, WI 53110-8902 USA; John C. Fanning, TIMET, Henderson, NV 89009 USA; Henry J. Rack, Clemson University, School of Materials Science & Engineering, Clemson, SC 29634-0921 USA

Tuesday PM — Room: Salon 10/11
February 15, 2005 — Location: San Francisco Marriott

Session Chairs: John Fanning, TIMET, Henderson, NV 89009 USA; James G. Ferrero, The Perryman Company, Houston, PA 15342 USA

2:00 PM
Mechanical Properties of Laser Deposited Beta Titanium Alloys with TiB Reinforcements: *Davion Hill*[1]; Rajarshi Banerjee[1]; Daniel Huber[1]; Peter C. Collins[1]; Jaimie Tiley[2]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Dayton, OH USA

In recent years there has been considerable interest in b Ti alloys due to their wide applicability in a number of different areas, for example, in aircraft structural components. In order to increase the stiffness and strength of these alloys, reinforcements such as transition metal borides are considered promising. Therefore, there is substantial interest in the development of metal-matrix composites consisting of borides dispersed in a b Ti matrix. Laser engineered net-shaping (LENS®) is a directed laser deposition process which uses a powder feedstock and appears to be a promising technology for the processing of these metal-matrix composites. Two types of TiB reinforced composites have been studied, one based on the alloy Ti-5Al-5V-5Mo-3Cr-0.5Fe (TIMETAL 5553) and the other based on the alloy TIMETAL 21S with TiB reinforcements. Using a feedstock consisting of a blend of pre-alloyed TIMETAL 5553 (or TIMETAL 21S) and elemental boron powders, these composites have been deposited in a single step via the LENS® process. These as-deposited composites exhibit a refined homogeneous distribution of TiB precipitates within the alloy matrix, a consequence of the rapid solidification rates inherent to the LENS® process. The microstructure of the LENS® deposited composites has been investigated in detail using SEM and TEM based techniques. The room temperature tensile properties and wear resistance of these composites is currently being investigated and will be presented in this paper.

2:25 PM
Processing, Microstructure, and Properties of Beta Ti Alloys Modified with Boron: *Sesh Tamirisa*[1]; Radhakrishna B. Bhat[2]; Jay Tiley[3]; Dan B. Miracle[3]; [1]Ohio University/AFRL, AFRL/MLLMD, 2230 Tenth St., Ste 1, Wright-Patterson AFB, OH 45433-7817 USA; [2]UES Inc., Matls. Procg., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [3]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., Ste. 1, Wright-Patterson AFB, OH 45433-7817 USA

The development of next generation beta Ti alloys is expected to involve very attractive combinations of strength-toughness-fatigue resistance at large cross sections, improved and affordable processibility, and higher elevated temperature capability. This paper describes the development of beta Ti alloys modified with small boron additions to achieve the above goals. Two important aerospace alloys, Ti-15Mo-2.6Nb-3Al-0.2Si (TIMETAL21S) and Ti-5Al-5V-5Mo-3Cr (Ti-5553) with various boron levels are considered. The objective of this study is to establish the influence of boron addition on the microstructural evolution and mechanical properties. Ingots of 70 mm diameter and 500 mm length are cast using induction skull melting. Detailed microstructural characterization and tensile property evaluation are conducted. Effect of boron addition on the microstructural stability and properties in the as-cast condition will be presented. The implications of boron addition on the microstructural evolution and affordability of subsequent processing will be discussed.

2:50 PM
Effects of Carbon on the Creep Behavior of Burn-Resistant â-Phase-Based Ti-35V-15Cr-xC Alloys: *Fusheng Sun*[1]; E. J. Lavernia[1]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA

There is a great deal of interest in developing burn-resistant titanium alloys for aerospace applications. The objective of this research is to study the effects of carbon on the creep resistance in burn-resistant beta-phase-based titanium alloys. The creep behavior of the Ti-35V-15Cr and Ti-35V-15Cr-0.2C alloys at 500-580°C within a stress range of 200-300MPa was studied. The deformation behavior was characterized using a JEM-2010 transmission electron microscope (TEM). The experimental results showed that the creep resistance of Ti-35V-15Cr-0.2C is considerably improved by incorporation of Ti2C particulates into the matrix. The creep deformation of the Ti-35V-15Cr-xC (x=0, 0.2%) alloys was dominated by a dislocation-controlled creep-process. The improved creep resistance of the Ti-35V-15Cr-0.2C is attributed to both solid solution strengthening of carbon and dispersion strengthening of Ti2C particles by inhibiting dislocation motion in the matrix.

3:15 PM
New Class of Heat-Resistant Titanium Alloys Produced by MEM Technology: *Yaroslav Yurievich Kompan*[1]; Valentin Nikolaevich Moiseev[2]; [1]E.O. Paton Electric Welding Institute, Dept. of Magnetic Hydrodynamics of Electroslag Processes, 11, Bozhenko Str., Kyiv 03680 Ukraine; [2]All Russian Institute of Aircraft Materials, 17, Radio Str., Moscow 107005 Russia

Presentation covers the first steps towards creation of new class of heat-resistant alloys and investigates the new class of alloys based on alpha- and beta-solid solutions with addition of intermetallic second phase particles. These alloys are characterized by the intermetallic dispersion strengthening and/or a "rigid frame" of eutectoid forming phase in a soft matrix. These alloys have been achieved by the method of magnetically controlled electroslag melting (MEM) which assures purified, refined heterogeneous (in terms of the eutectoid-forming phase) cast structure. The MEM technology allows to produce round ingots of 300 mm plus diameter and rectangular ingots of similar mass. Average strength of the alloys at room temperature and at UTS=1400-1600MPa and operating temperatures are estimated 700-800°C respectively.

3:40 PM Break

3:55 PM
Effect of Deformation Conditions on Grain Size and Microstructure Homogeneity of Beta-Rich Titanium Alloys: *G. A. Salishchev*[1]; R. M. Galeyev[1]; O. R. Valiakhmetov[1]; M. F.X. Gigliotti[2]; B. P. Bewlay[2]; C. U. Hardwicke[2]; [1]Institute for Metals Superplasticity Problems, 39 Khalturin Str., Ufa 450001 Russia; [2]GE Research, Niskayuna, NY USA

The control of grain size, shape, and microstructural homogeneity is of great importance for secondary processing and to achieve optimal mechanical properties in a final forging. We will present and discuss the development of processing windows in both beta and alpha/beta fields for forming homogeneous microstructures with grain sizes down to sub-microcrystalline. Alloys studied include stable beta, metastable beta and high-beta alpha/beta alloys. The effects of deformation temperature, strain and strain rate, initial microstructures and alloy-content on final grain size and microstructural homogeneity will be reported. In one example of forming a homogeneous microstructure in a beta-rich alloy, isothermal multiple-step forging was used to produce billets with low ultrasonic noise. Heat treatment response, and room and elevated temperature mechanical properties of these billets will be discussed.

4:20 PM
Anisotropy of Mechanical Properties in High-Strength Titanium Beta-Alloys: *Orest M. Ivasishin*[1]; Pavlo E. Markovsky[1]; Sergiy A. Kotrechko[1]; Vadim I. Bondarchuk[1]; Stephen P. Fox[2]; [1]Institute for Metal Physics, 36 Vernadsky St., Kiev 03142 Ukraine; [2]TIMET Henderson Laboratory, Henderson, NV USA

Thermomechanical processing of beta alloys substantially influences their microstructure and crystallographic texture, thus resulting

in anisotropy of mechanical properties. Usually, final high-strength conditions in beta alloys are formed by solution treatment followed by aging (STA), in which alloy is solution treated below beta-transus temperature. This means that high strength material exhibits anisotropy undesired for the many applications. It was shown in the present paper, taking TIMETAL-LCB beta alloy as an example, that application of special heat treatment consisting of solid solutioning by rapid continuous heating into single phase beta field and subsequent aging allows to reduce an influence of primary thermomechanical processing and attain more isotropic structural condition as compared to conventionally heat treated material. Tensile mechanical properties were determined in rolling and transverse directions. In the last case a unique testing technique was developed. Obtained results are discussed in terms of high-strength material resistance to brittle fracture. Since nucleation cracks open up in distinct crystallographic planes, their orientational distribution was determined what allowed to predict influence of texture on anisotropy of mechanical properties.

4:45 PM
Comparative Study of the Mechanical Properties of High-Strength Beta-Titanium Alloys: *Orest M. Ivasishin*[1]; Yuriy V. Matviychuk[1]; Pavlo E. Markovsky[1]; S. L. Semiatin[2]; [1]Institute for Metal Physics, 36 Vernadsky St., Kiev 03142 Ukraine; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Wright-Patterson AFB, OH 45433-7817 USA

The mechanical properties of three commercial beta-titanium alloys (TIMETAL-LCB, Ti-15-3, and VT22) were compared in the STA-condition following thermomechanical processing comprising beta-solid solutioning, cold deformation (CD), continuous rapid heating, and final aging. The peak temperature during the rapid heating step was chosen to obtain either a polygonized or recrystallized condition and thereby to control the distribution of residual deformation defects, which, in turn, influenced the dispersion and distribution of alpha-phase precipitates in the final microstructure. It was established that a good balance of high strength (in excess of 1600 MPa) and reasonable ductility could be obtained if a fine-grained microstructure with a beta-grain size of ~10 mm was formed by recrystallization. The development of such a fine grain size enabled a reduction in the aging temperature and thus increased the strength while maintaining ductility within the desired limit. In this respect, the properties could be varied to a great extent by varying the rate of heating to the aging temperature. On the other hand, aging of the polygonized condition also led to a strength level of approximately 1600 MPa but with generally lower ductility than in the fine-grained recrystallized condition. Another distinctive feature of the polygonized condition was that the sensitivity of properties to the rate of heating to the aging temperature was not very strong.

5:10 PM
Rapid Beta Solution Treated Timetal® LCB Alloy Mechanical Properties and Texture: *Allan J. Hutt*[1]; Orest M. Ivasishin[2]; [1]Perryman Company, 213 Vandale Dr., Houston, PA 15342 USA; [2]National Academy of Sciences, Inst. for Metal Physics, 36, Vernadsky str., Kiev 252142 Ukraine

Timetal® LCB Alloy has demonstrated a good balance of strength, ductility, and fatigue properties for automotive spring applications in the alpha-beta solution treated and aged condition. Mechanical properties and texture were evaluated in the rapid beta solution treated and aged condition. Conventional beta treatments yield material with low ductility. However, if the beta grain size can be kept below 10 microns by using controlled heating rates, beta solution treatment can improve strength and ductility from alpha-beta solution treated material.

Biological Materials Science and Engineering: Biological Materials/Bio-Medical Applications II

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Society for Biomaterials, Surfaces in Biomaterials Foundation, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), EMPMD/SMD-Biomaterials Committee
Program Organizers: Marc Andre Meyers, University of California, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Tuesday PM — Room: 3009
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: Robert O. Ritchie, University of California, Matls. Sci. & Engrg., Berkley, CA 94720-1760 USA; Paul Calvert, University of Massachusetts, Dept. of Textile Scis., N. Dartmouth, MA 02747 USA

2:00 PM Invited
Biomimetic Bonelike Composites and Novel Bioactive Glass Coatings: *Antoni P. Tomsia*[1]; [1]Lawrence Berkeley Laboratory, Matls. Scis. Div., One Cyclotron Rd., MS 62R0203, Berkeley, CA 94720 USA

This presentation focuses on the design and synthesis of artificial bone-like composite materials and novel bioactive glass coatings for metallic implants using natural bone as a guide. Hydrogel polymers that possess anionic groups suitably positioned for nucleating biominerals, and therefore mimic the natural function of the collagen-phosphoprotein matrix in bone, were designed to direct template-driven biomineralization in a microscopically controlled fashion. Different mineralization techniques were developed to enable the formation of hydrogel-calcium phosphate composites with either porous microstructures suitable for potential tissue penetration or excellent polymer-mineral interfacial affinity that is desirable for bone mimics with enhanced biocompatibility and interfacial mechanical properties. Novel osteophilic glass coatings with thermal expansion properties matching with those of their metallic bone implant substrates such as Ti or Co/Cr, and therefore preventing interfacial cracking during the enameling process, were also developed.

2:30 PM Invited
Effect of Bone Density on the Damping Coefficients of Dental Implants: An In Vitro Method: Lindsey R. VanSchoiack[1]; Jean C. Wu[3]; *James C. Earthman*[1]; [1]University of California, Biomed. Engrg., 916 Engrg. Tower, Irvine, CA 92697 USA; [3]Newport Coast Oral Facial Institute, 360 San Miguel Dr., Ste. 204, Newport Beach, CA 92660-7828 USA

For normal healthy teeth the percussive energy generated by mastication is attenuated by the periodontal ligament at the healthy bone-natural tooth interface, this is not the case when dental implants are present. Previous studies have looked at the mechanical damping in dental implants, however the studies have never examined the effect of bone density at the time of implant placement in conjunction with quantitative measures of mechanical energy dissipation. Utilizing a series of artificial foam bone models which vary in density and structure, we have tested the mechanical energy dissipation of several different dental implant makes and models as a function of simulated bone density. Our hypothesis is that the Periometer, a percussion probe system designed to measure local damping capacity, can assess the quality of the underlying support structure. We also hypothesize that the Periometer will be able to differentiate between implant model geometries within the same support structures.

3:00 PM Invited
Titanium Alloys for Biological Applications: *H. J. Rack*[1]; [1]Clemson University, Sch. of Matls. Sci. & Engrg., 213 Olin Hall, Clemson, SC 29634-0971 USA

Titanium alloys, because of their excellent mechanical, physical and biological performance are finding ever-increasing application in biomedical devices. This presentation will review and illustrate the history of titanium alloy use for medical devices, their current status, future opportunities and obstacles for expanded application. Illustrations will be given for commercial purity titanium, alpha-beta and metastable beta alloys, intermetallic compounds and metal matrix composites.

3:30 PM Break

3:45 PM
Surface Treatments of Ti Dental Implant: *Carlos Nelson Elias*[1]; Rodrigo Prioli[2]; Guilhermo Solorzano[2]; Ricardo Bathomarco[2]; [1]Military Institute of Engineering, Pr Gen. Tiburcio 80, Rio de Janeiro Brazil; [2]Pontifícia Universidade Católica, Caixa Postal 38071, Rio de Janeiro, RJ Brazil

The surface of commercial unalloyed titanium used in dental implants was analyzed by atomic force microscopy. The morphology, roughness and surface area of the samples submitted to anodizing, shot-peening, acid etching and a combination of them, were compared. The results show that surface treatments strongly influence the dental implant physical and chemical properties. An analysis of the length dependence of the implant surface roughness shows that, for scan sizes larger than 50mm, the average surface roughness is independent of the scanning length and that all the surface treatments lead to surfaces with similar roughness in the range of 435 nm ± 49 nm. It is shown that the implant surface energy is sensitive to the titanium surface area. As the area increases there is a decrease in the surface contact angle.

4:05 PM
Surface Characteristic of Metallic Biomaterials and Their Effect on Interaction with Osteoblast Cells: L. Bren[1]; *J. Drelich*[1]; L. English[1]; J. Fogarty[1]; N. Istephanous[2]; R. Policoro[1]; A. Zsidi[1]; [1]Michigan Tech, Matls. Sci. & Engrg., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]Medtronic USA, Inc., Matls. & Bioscies. Ctr., 710 Medtronic Pkwy., Minneapolis, MN 55432 USA

The application of bone screws was examined with the goal of learning which surface properties of 316L stainless steel and Ti-6Al-4V metal alloy elicit the desired osteoblast cell response. Mice osteoblast cells were cultured on seven different surface treatments and material combinations and the resulting cell proliferation, differentiation and morphologies were studied. The research results indicate on the positive effects of nano-scaled roughness, random surface topography, and reduced surface potential on improved cell attachment and differentiation. It was also found that the alkaline phosphatase activity increased with increasing surface tension and increasing electron-acceptor surface tension parameter of the implant material. These results suggest that the formation of surface hydroxyl groups with acidic character of metallic biomaterial gives rise to enhanced differentiation of osteoblast cells.

4:25 PM
Comparison of the Fatigue and Tensile Behavior and Biocompatibility of Ti-17Al-33Nb(at.%) and Ti-22Al-28Nb(at.%) with Ti-6Al-4V(wt.%): *C. J. Cowen*[1]; M. Niinomi[2]; T. Akahori[2]; L. M. Flick[3]; K. A. Rider[3]; C. J. Boehlert[1]; [1]Michigan State University, Cheml. Engrg. & Matls. Sci., Engrg. Bldg., E. Lansing, MI 48824-1226 USA; [2]Toyohashi University of Technology, Production Sys. Engrg., Toyahashi 441-8580 Japan; [3]Alfred University, Alfred, NY 14802 USA

In this work the fatigue (R=0.1, 10Hz, max. stress between 60-95% of the UTS) and tensile properties of Ti-17Al-33Nb(at.%) and Ti-22Al-28Nb(at.%) were compared to those for Ti-6Al-4V(wt.%), which is commonly used for biomedical implant applications. The S-N behavior illustrated that both the as-processed and heat-treated Ti-Al-Nb alloys exhibited fatigue lives equal to or greater than those for Ti-6Al-4V. The RT tensile strength and elastic modulus of the Ti-Al-Nb alloys were also comparable. Biocompatibility experiments, measuring the resorption of mouse calvarial tissue in response to the Ti-Al-Nb particles, indicated that no significant reaction occurs between the particles and living cells, resulting in a midline sagittal suture area comparable to that of untreated mice. These experiments demonstrated that there is no difference in the biological response to either Ti-17Al-33Nb or Ti-22Al-28Nb. The results of this study suggest Ti-Al-Nb alloys have potential for biomedical implant applications. This work was partially supported by the National Science Foundation (DMR 0134789).

4:45 PM
Design of an Optimum Acetabular Cup Prosthesis: *Kamran Tabeshfar*[1]; [1]Bournemouth University, Design Grp., 12 Christchurch Rd., Bournemouth BH13NA UK

With the current trend of performing Total Hip Replacements (THR) on younger patients, incurring an increasing number of revisions, prolonging the life of a THR is of paramount importance and one of the main goals of research in the field. Younger patients not only require increased longevity from their prosthesis but also increased performance so as to be able to undertake more strenuous everyday activities, such as sports. Historically the main objective of THR was to relieve pain and increase quality of life in the elderly; generally, these were not intended for young active patients. Previous research has proposed that the acetabular cup design has far more impact on long-term survival of the THR than the femoral component. Optimising the acetabular cup prosthesis produces a highly complex problem where many of the individual design factors have massive impact on the system. A main aim is to develop a material or combination of materials to optimise the stress distribution in the system without sacrificing the service life of the THR. Of the 800,000 hip replacements carried out annually, many of the current acetabular cups have some form of polyethylene bearing surface, but polyethylene wear debris is seen as a major contributing factor to bone resorbtion and hence prosthesis loosening. Changes in stress values, even caused by initial primary fixation during the operation, can result in stresses being transferred in an unrealistic manner. The effect is that the bone grows to differing thickness and strengths (remodelling). For optimisation of the acetabular cup, the properties of the natural hip must be retained by minimising both remodelling and bone resorbtion. This paper describes research and development related to a novel composite acetabular cup prostheses with a ceramic on ceramic bearing surface that should last longer and perform better, thus reducing the necessity for costly and debilitating revisions later on in life. In addition, the use of the proposed novel anatomically orientated mechanical testing methods gave fast, cheap results and could also allow further research into fatigue failure of alumina bearing couples in THR.

5:05 PM
Fabrication of Novel TiZr Alloy Foams for Biomedical Application: *Cui'e Wen*[1]; Peter Damian Hodgson[1]; Yasuo Yamada[2]; [1]Deakin University, Sch. of Engrg. & Tech., Pigdons Rd., Geelong, VIC 3217 Australia; [2]National Institute of Advanced Industrial Science and Technology, Matls. Rsch. Inst. for Sustainable Dvlp., 2266-98 Anagahora, Simoshidami, Moriyama-ku, Nagoya, Aichi 463-8560 Japan

Bone injuries and failures often require the inception of implant biomaterials. Research in this area is receiving increased attention globally. In particular, porous metals are attractive due to its unique physical, mechanical, and new bone tissue ingrowth properties. In the present study, TiZr alloy powders were prepared by a mechanical alloying process. Novel TiZr alloy foams with porosities of about 80% were fabricated by a powder metallurgical process using the MA powders. The TiZr alloy foams display an interconnected porous structure resembling bone and the pore size ranges from 200 μm to 500 μm. The TiZr alloy foams exhibit an appropriate strength to withstand physiologic loading and a low elastic modulus very close to bone.

5:25 PM
A Novel Combinatorial Approach to the Development of Beta Titanium Alloys for Orthopaedic Implants: *Soumya Nag*[1]; Rajarshi Banerjee[1]; John Stechschulte[2]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Cornell University, Matls. Sci. & Engrg., Ithaca, NY USA

Orthopeadic alloys for implant applications typically require a combination of different properties such as excellent biocompatibility with no adverse tissue reactions, excellent corrosion resistance in the body fluid where it will be used, high mechanical strength and fatigue resistance, low modulus, and good wear resistance. Since the beta phase in Ti alloys exhibits a significantly lower modulus than the alpha phase, and the beta alloys also satisfy most of the other requirements for an ideal orthopaedic alloy, there is a thrust towards the development lower modulus beta-Ti alloys which retain a single beta phase microstructure on rapidly cooling from high temperatures. While a number of biocompatible beta-Ti alloys have been reported in recent literature, there is still a tremendous scope for improvement in terms of alloy design via optimization of alloy composition and thermo-mechanical treatments. A novel combinatorial approach has been developed for rapid assessment of the microstructure and properties of such alloys and consequently aid in the development of new orthopaedic alloys as well as in the maturation of existing ones. This approach is based on the use of directed laser deposition to rapidly process compositionally graded alloys, administer appropriate heat-treatments to these alloys, characterize and quantify their microstructures, assess their mechanical properties, and finally build a database relating the composition-microstructure-property. This database has been used to train and test fuzzy-logic models to predict property-microtructure-composition relationships in these alloy systems. These results will be discussed in this presentation.

Bulk Metallic Glasses: Shear Banding and Deformation

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA

Tuesday PM Room: 3006
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Michael Atzmon, University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109 USA; Mo Li, Georgia Institute of Technology, Matls. Sci. & Engrg., Atlanta, GA 30332 USA

2:00 PM
Annealing of Shear Bands — A Nanoindentation Study of Plasticity in a Cold-Rolled Al-Based Metallic Glass: Wenhui Jiang[2]; *Michael Atzmon*[1]; [1]University of Michigan, Dept. of NERS & MSE, Cooley Bldg., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA; [2]University of Michigan, Dept. of NERS, Cooley Bldg., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA

Few studies have addressed the effect of existing shear bands on the deformation behavior of metallic glass. Serrated flow has recently been observed in nanoindentation studies of metallic glasses, and linked to shear-band formation. Using instrumented nanoindentation, we have investigated the plastic flow behavior of a cold-rolled, amorphous, $Al_{90}Fe_5Gd_5$ ribbon at various loading rates and compared it with that of undeformed, as-spun ribbon. Cold rolling to a total thickness reduction of 45.5% was performed in a large number of small steps so as to prevent sample heating. While the as-spun ribbon exhibited serrated flow and pileups around the indents, the rolled amorphous ribbon demonstrated a smooth load-displacement curve and no pileup. Furthermore, annealing of the cold-rolled ribbon below the crystallization temperature restored the serrated flow and pileups. The role of shear-band initiation in the deformation behavior will be discussed. Support provided by the National Science Foundation under Grant DMR-0314214.

2:20 PM
Experimental Observations of Shear Banding in Bulk Metallic Glasses: *J. J. Lewandowski*[1]; N. A. Stelmashenko[2]; A. L. Greer[2]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., Cleveland, OH 44106-7204 USA; [2]University of Cambridge, Dept. Matls. Sci. & Metall., Cambridge CB2 3Q UK

Structural applications of bulk metallic glasses (BMGs) are limited by the inhomogeneous nature of their plastic deformation, leading to interest in the mechanisms of shear banding. The temperature rise associated with shear-band operation has been investigated for BMGs based on Cu, Hf or Zr, and for in-situ composites of beta phase in Zr-based BMG, by coating them with thin films of low-melting-point metals. Temperature increases have been detected in all cases by morphological changes taken to arise from melting of the coating. The increases are at least 200 K, and within the band are much higher. As indicated by melting of the coating, here is heating not only associated with final rupture, but also around subsidiary bands, extending 1 to 10 micrometres on either side of the band. No similar observations were noted on identical experiments conducted on an oxide glass or a high strength aluminum alloy. The local heating estimated from these observations on the BMGs will be compared with published observations made by different techniques and with calculations based on models of the flow mechanism.

2:40 PM
In Situ Visualization of Shear-Band Evolution During Tensile Testing of a Bulk Metallic Glass: *Mark L. Morrison*[1]; Bing Yang[1]; Peter K. Liaw[1]; C. T. Liu[2]; Raymond A. Buchanan[1]; Cecil A. Carmichael[2]; Ramon V. Leon[3]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty, Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA; [3]University of Tennessee, Dept. of Stats., Ops & Mgmt. Scis., 337 Stokely Mgmt. Ctr., Knoxville, TN 37996-0532 USA

For the first time, the evolution of multiple shear bands has been visualized in a bulk metallic glass (BMG) with infrared (IR) thermography. Surprisingly, up to 58 shear bands were observed to initiate, propagate, and arrest during a single tensile test of a $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10.0}Ti_{5.0}$ (atomic percent) BMG alloy, commonly known as Vitreloy 105. By utilizing this unique visualization technique, the length, width, location, sequence, temperature evolution, and velocity of individual shear bands were quantified. Furthermore, correlations among the variables also were investigated. Detailed statistical analyses of these parameters were conducted in order to gain insight into the process of the inhomogeneous deformation in BMG materials. The authors are grateful to the National Science Foundations, Integrative Graduate Education and Research Training (IGERT) program under grant number DGE-9989548; the Combined Research and Curriculum Development (CRCD) Training program under ZZC-9527527 and EEC-0203415 with Dr. L. Clesceri, Dr. W. Jennings, Dr. L. Goldberg, and Ms. M. Poats as the contract monitors; and the Division of Materials Science and Engineering, Department of Energy under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory (ORNL) operated by UT-Battelle, LLC.

3:00 PM
Modeling Shear Band Propagation in Bulk Metallic Glasses: *Brian J. Edwards*[1]; Kathleen Feigl[2]; Peter K. Liaw[3]; Mark Morrison[3]; Bing Yang[3]; Ray A. Buchanan[3]; [1]University of Tennessee, Cheml. Engrg., Knoxville, TN 37996 USA; [2]Michigan Tech. University, Math. Scis., Houghton, MI 49931 USA; [3]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA

Recent observations have indicated that shear bands originate and propagate in bulk metallic glasses (BMGs) under tensile loading once plastic deformation has begun. These shear bands propagate and dissipate on the order of milliseconds, as witnessed with high-speed and high-sensitivity infrared thermography. In this presentation, we present results of a study aimed at understanding the onset, propagation, and eventual dissipation of these shear bands under tensile loading in BMGs. Initial results are discussed, based on the application of a non-equilibrium thermodynamics approach to this problem, which results in a system of equations that couples the applied stress distribution within the sample with temperature and an additional vector field associated with the free volume in BMGs. This set of equations describes the shear-band formation, propagation, and dissipation within the BMGs, and gives hints concerning the origination of shear bands, their speeds of propagation, their width and length, their direction of propagation, and the magnitude of any permanent plastic deformation that occurs across them.

3:20 PM
Mesoscopic Theory and Modeling of Shear Localization in Metallic Glass: *Mo Li*[1]; Guang-Ping Zheng[2]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332 USA; [2]Hong Kong University, Dept. of Mechl. Engrg., Hong Kong China

A mesoscopic theory for shear localization in metallic glass is proposed. Based on experimental observations and atomistic modeling results, we establish the relation between local free volume density and applied stress. This phenomenological theory predicts shear localization, shear instability and shear zone propagation. A phase-field modeling is applied based on this theory.

3:40 PM Break

4:00 PM
Shear Localization in Metallic Glass: An Atomistic Study: *Mo Li*[1]; QiKai Li[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332 USA

Shear localization in binary metallic glass ZrNi, CuZr and PdSi are investigated using extensive molecular dynamics simulations. It is found that shear localization is a result of local instability of deformation triggered by local concentration of stress. Contrary to long-held view that shear localization is adiabatic in its origin, our work suggest that local heating and fluid-like behavior are simply a consequence of the shear instability, not the cause. Excess free volume is found in the shear zone. The speed of the shear zone propagation is estimated. Effects of various processing factors on shear zone formation are also investigated.

4:20 PM
Shear Localization and Percolation of Quasicrystalline Structure in a Simulated Model Metallic Glass: *Yunfeng Shi*[1]; Michael L. Falk[1]; [1]University of Michigan, Matls. Sci. & Engrg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

Molecular dynamics simulations of uniaxial tension in a two-dimensional model of a metallic glass exhibit varying degrees of shear localization depending upon the process by which each of the materials was produced. The samples that were quenched most gradually show the largest degree of localization. In addition higher strain rates lead to

increased localization in the most rapidly quenched samples, while the more gradually quenched samples show the reverse strain rate dependence. This transition in localization and strain rate dependence coincides with a structural transition in the material. Gradually quenched samples have a higher percentage of atoms in quasi-crystal-like local environments. The transition in the mechanical properties coincides with the percolation of this backbone of quasi-crystal-like material. Shear localization occurs in regions where material is altered from this more stable structure to a fully amorphous structure under the effect of plastic deformation.

4:40 PM
Micromechanics and Macroscopic Mechanical Behavior of In Situ Formed Metallic Glass Matrix Composites: *Ryan T. Ott*[1]; Frederic Sansoz[2]; Jean-Francois Molinari[3]; Jon Almer[4]; Todd C. Hufnagel[1]; [1]Johns Hopkins University, Matls. Sci. & Engrg., 102 MD Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]University of Vermont, Dept. of Mechl. Engrg., Burlington, VT 05405 USA; [3]Johns Hopkins University, Dept. of Mechl. Engrg., Baltimore, MD 21218 USA; [4]Argonne National Laboratory, Advd. Photon Source, Argonne, IL 60439 USA

We have examined the mechanical behavior of in situ formed composites consisting of crystalline Ta particles in a Zr-based amorphous matrix. We evaluate yield criteria for the composite based on yield stresses in tension v. compression, as well as shear band angles. We also examine the micromechanics of deformation of the composite alloys using in situ high-energy x-ray scattering. Yielding of the Ta-rich particles creates a misfit strain in the surrounding glass matrix; this creates a stress concentration that leads to initiation of shear bands. As a result, the glass yields locally around the Ta-rich particles prior to global yielding of the amorphous matrix. Combining the results from the uniaxial compression tests, the in situ strain measurements, and finite element models of deformation, we discuss the relationship between the micromechanics and the macroscopic mechanical behavior of the composite alloys.

5:00 PM
Evolution of Multiple Shear Band Patterns Beneath an Indentation in Bulk Metallic Glasses Using a Bonded Interface Technique: *Ghatu Subhash*[1]; Hongwen Zhang[1]; Laszlo J. Kecskes[2]; Robert J. Dowding[2]; [1]Michigan Technological University, ME-EM Dept., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]Army Research Laboratory, AMSRD-ARL-WM-MB, Aberdeen Proving Ground, Aberdeen, MD 21005 USA

Although shear band formation has been known to be the dominant deformation mechanism in bulk metallic glasses (BMGs), many indentation studies have revealed only a few shear bands that cannot account for the large ductility observed in BMGs. Therefore a bonded interface technique has been developed to observe the shear bands beneath the indentation. Vickers indentations were performed along this interface. At small indentation loads (<50g) numerous semicircular shear bands surrounding the indentation were observed. The spacing between these shear bands decreases with increasing distance from the indentation. At moderate loads (100-300g), largely spaced secondary shear bands emanate from each of the deformed surfaces of the indentation. At higher loads (>300g), a third set of shear bands radiate from the tip of the indentation and cut across the first two sets. The evolution of shear band patterns and the effects of indenter orientation with respect to the interface will be presented.

Cast Shop Technology: Melt Treatment: Degassing and Filtration

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Gerd Ulrich Gruen, Hydro Aluminium AS, Bonn 53117 Germany; Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Tuesday PM Room: 2001
February 15, 2005 Location: Moscone West Convention Center

Session Chair: John Courtenay, MQP Ltd, Casthouse Tech., Solihull, W. Midlands B93 9EW UK

2:00 PM
An Analysis of Impeller Performance Based on an Air-Water Model: *J. J.J. Chen*[1]; M. Nilmani[2]; [1]University of Auckland, Cheml. & Matls. Engrg., PB 92019, Auckland New Zealand; [2]NCS Associates (Australia) Pty Ltd., 101/180, Flinders Ln., Melbourne Australia

While there are many designs of degassing impellers available in the market, it had been reported that the majority of these impellers gave similar degassing efficiency under supplier recommended operating conditions based on tests conducted in full scale water model experiments. This paper shows that the degassing performance of these impellers can be described by a mass transfer equation which considers the power input based on a consideration of the Power number versus Reynolds number relationship in the absence of gas injection and under fully turbulent conditions, and the actual power input in the gassed condition. For one of the impellers used, the Power number versus Reynolds relationship has been measured. For the other impellers, the Power number at large Reynolds number was assumed. The oxygen desorption data for all four commercial impellers were found to be well-represented by one single equation.

2:25 PM
Ultrasonic Degassing of Molten Aluminum Under Reduced Pressure: *Hanbing Xu*[1]; Xiaogang Jian[1]; Thomas T. Meek[1]; Qingyou Han[2]; [1]University of Tennessee, Matls. Scis. & Engrg., 434 Dougherty Hall, Knoxville, TN 37996 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., Matls. Procg. Grp., Oak Ridge, TN 37831-6083 USA

Ultrasonic degassing, an environmentally clean and cheap technique, is an efficient way of degassing in a static volume melt. However, there is a steady-state hydrogen concentration below which the effect of ultrasonic vibration will be absent. The limit of ultrasonic degassing will occur within a few minutes of ultrasonic vibration, regardless of the initial hydrogen concentration in the melt. Vacuum degassing, a practical technique used in Europe, also has been tested a beneficial and clean method in producing high quality products. Combination of these two techniques will help to lower the limit in ultrasonic degassing and promote the efficiency in vacuum degassing. An experimental device which combines the vacuum degassing and ultrasonic degassing has been built in Oak Ridge National Laboratory recently. Parametric studies have been carried out to investigate the efficacy of the ultrasonic degassing of molten Aluminum alloy under reduced pressure. This article reports the experimental results and discusses the mechanism of degassing in molten Aluminum under the influence of vacuum and ultrasonic vibrations.

2:50 PM
Characterization of Droplets Produced by Bubbles Bursting: *Autumn Fjeld*[1]; James W. Evans[1]; [1]University of California, Matls. Sci. & Engrg., 210 Hearst Mining Bldg., MS 1760, Berkely, CA 94720 USA

A limitation on the metal throughput of a gas fluxing unit is posed by splashing and spraying of metal droplets as the gas throughput is increased to keep pace. In an investigation at Berkeley funded by DOE (OIT DE-FC07-01ID14192) and Alcoa, droplets ejected as a result of bubble rupture at the free surface of a molten metal are examined via high speed digital photography and image tracking software. Experiments are carried out in a custom built, glass walled vessel designed for observation of splashing and spraying at the surface of a low melting temperature alloy. A controlled bubble release system permits variation in bubble size, release of single or multiple bubbles, or a continuous stream of bubbles. The effects of melt depth in the vessel, oxide formation at the melt surface, and the presence of a second liquid on droplet behaviour are also examined. Droplet numbers, velocities, trajectories and size distributions are determined for the aforementioned conditions with image analysis software.

3:15 PM
Upstream Fluid Flow Particle Removal: *Laurens Katgerman*[1]; Jan Zuidema[2]; [1]Delft University, NIMR, Rotterdamseweg 137, Delft 2628AL The Netherlands; [2]Corus RD&T, P.O.Box 10000, IJmuiden 1970 CA The Netherlands

The occurrence of oxides films and inclusions can give major problems during aluminium casting and processing. The level of oxides and inclusions in standard DC casting practices is controlled with the use of in-line filter boxes and degassing units. However, during the transfer from these metal treatment systems to the casting station pick up of inclusions and oxides can occur. This can be either caused by the varying efficiency of the metal treatment system and/or uncontrolled fluctuations in the metal flow. The aim of this paper is to assess quantitatively some of the upstream flow modifiers and their effect on melt cleanliness. The first method is placing baffles in the launder system. The location of these baffles is an important parameter in modifying the fluid flow behaviour. Both the controlled filling of the mould as well as entrapment of inclusions can be achieved. The second method is modifying the flow pattern in such a way that separation of

fluid from the particles occurs. This is done by employing a cyclone. Results of numerical simulations of fluid flow with discrete particles of different mass and size distribution are given as well as experimental results obtained from a water model.

3:40 PM Break

3:50 PM
XC Filter - A New Hybrid Filter for Improved Filtration Efficiency: *Stephen Instone*[1]; Mark Badowski[1]; Wolfgang Schneider[1]; [1]Hydro Aluminium Deutschland, CC Casting, Alloys & Recycling, Georg-von-Boeselager-Str. 21, Bonn, NRW 53117 Germany

The quality of the aluminium rolling ingot is increasingly important to fulfil requirements of the various aluminium rolling and subsequent downstream finishing process chains. The presence of non-metallic inclusions is particularly important for products such as aluminium foil and lithographic sheet. Inclusions in the size range 10-40μm can pass through current in-line filters and lead to limitations in the processing capabilities of the ingot. A new design for a filter unit, named the XC filter, for the treatment of liquid aluminium has been tested and found to give superior filtration efficiency particularly in the important size range mentioned above. The XC Filter, combines elements of ceramic foam filtration (CFF) and deep bed filtration (DBF) to overcome limitations of the established technologies. The performance of the XC Filter, measured during industrial casting trials using LiMCA, is presented. Comparison is made to performance data of CFF filters obtained under similar conditions.

4:15 PM
AlF_3 as an Aluminium Filter Medium: *Harald Görner*[1]; Martin Syvertsen[2]; Eivind J. Øvrelid[2]; Thorvald Abel Engh[1]; [1]NTNU, Matls. Tech., Alfred Getz Vei 2, Trondheim N-7491 Norway; [2]SINTEF Materials and Chemistry, Metall., Alfred Getz Vei 2, Trondheim N-7465 Norway

An AlF_3 "active" filter is studied for removal of dissolved impurities (alkali, Mg, Sr, H) in addition to suspended particles in Al melts. Industrially, AlF_3 is injected as a refining powder. However, then the yield for removal of Na is low. A bed filter containing AlF_3 as a filter media should improve the kinetics by increasing both contact time and area. A lab setup has been built consisting of a AlF_3 filter and an Al_2O_3 dummy. This makes it possible to quantify the fraction of sodium removal due to the "active" filter media. Pure Al melts with Na additions were filtered. Disc samples were taken to determine the Na concentration at the filter inlet and the two outlets. Removal is from 55% to 73% for the conventional filter and from 80% to 91% for the active filter. The data are employed to derive a kinetic model for such filters.

4:40 PM
An Ultrasonic Sensor for the Continuous, On-Line Monitoring of the Cleanliness of Liquid Aluminum: M. Kurban[2]; P. H. Mountford[2]; *Ian D. Sommerville*[1]; N. D.G. Mountford[2]; [1]University of Toronto, Dept. of Matls. Sci. & Engrg., Toronto, Ontario M5S 3E4 Canada; [2]Metal Vision Manufacturing, Toronto, Ontario Canada

Over the past twenty years or so, an ultrasonic sensor has been developed which is capable of providing continuous, on-line monitoring of the inclusion content of liquid aluminum, where these inclusions can be either non-metallic or intermetallic. By inserting air cooled guide rods into the liquid metal, ultrasound can be passed into the liquid and the return signals processed to provide three pieces of information: a measurement of the average particle size of inclusions counted, a histogram display of the size distribution of the largest particles counted and an attenuation resulting from the scattering of the ultrasound by the particles suspended in the melt. This scattering is caused by all the particles present, but mainly by the very large number of particles which are too small to count because they are too small to cause discrete reflections. This technique has been used to detect particles in the size range 15-100 μm, and in principle is capable of detecting sizes up to 400 μm, which is regarded as the lower threshold for visual detection by the naked eye. Thus, it is capable of monitoring the acceptability of metal for a wide range of applications from the very high quality required in continuously cast metal to the less demanding requirements typical of foundry and die casting operations. The technique is virtually non-obtrusive, and is capable of monitoring significant proportions of the total metal volume. In the case of metal flowing in a launder, the proportion monitored is a linear function of the flowrate, and could amount to as much as 20 or even 25% of the total metal volume.

5:05 PM
Cleanliness of Aluminum and Steel: A Comparison of Assessment Methods: T. A. Utigard[1]; *Ian D. Sommerville*[1]; [1]University of Toronto, Dept. of Matls. Sci. & Engrg., Toronto, Ontario M5S 3E4 Canada

The quality and fitness for service of metals is determined in large part by casting them at the appropriate temperature, with the correct chemical composition and sufficiently clean that the inclusion content does not seriously compromise their performance in the intended application. In comparison with the methods available for determination of temperature and chemical composition, those available until recently for the assessment of cleanliness have been very unsophisticated and unsatisfactory. Many different methods have been devised for assessment of the cleanliness of both metals, most of them applicable to the solid metal, which is "after the fact" and allows only very limited opportunity for any required corrective measures to be taken. The fact that so many methods exist is clear evidence that none has been generally accepted as providing an accurate measure of the incidence of inclusions. Because there are so many methods, they can not be discussed in detail, but rather they are briefly reviewed in an attempt to highlight their strengths and weaknesses. Some emphasis is placed on those techniques which can be applied on-line to liquid metals, and which therefore provide a greater opportunity for corrective measures to be taken when an unsatisfactory level of cleanliness is detected.

5:30 PM
Filtration Efficiency and Melt Cleanness Evaluation Using LAIS Sampling at Valesul Aluminio S.A.: *Alexandre Vianna da Silva*[1]; Alberto Maia[1]; Luiz C.B. Martins[2]; Ramon Duque[2]; [1]Valesul Aluminio S.A., Estrada Aterrado do Leme, 1225, Santa Cruz, Rio de Janeiro, RJ 23579-900 Brazil; [2]SELEE Corporation, 700 Shepherd St., Hendersonville, NC 28792 USA

Valesul Aluminio castshop is a premier supplier of extrusion billets to the Brazilian and Overseas market. Billets are cast using an in-line MINT degasser and SELEE filtration system. A designed experiment was conducted to determine the influence on melt quality and filtration efficiency of chlorine content (3% and 0), and ceramic foam filter type (17-inch 20 ppi and 20-inch 30 ppi). Melt cleanliness was measured using a Liquid Aluminum Inclusion Sampler (LAIS), a total of 12 drops were tested. Oxide-type inclusions included spinels and oxides, grain refiner was present in all three sampling locations. Final melt quality was 0.010 mm^2/kg total less grain refiner, being similar for 3% and 0 chlorine. Melt cleanness improvement across both filter types were statistically significant, with filtration efficiencies of 51.9 % for the 17-inch 20 ppi filter and 77.5% for the 20-inch 30 ppi filter.

Characterization of Minerals, Metals and Materials: Characterization of Light Weight Materials - II

Sponsored by: Extraction & Processing Division, EPD-Materials Characterization Committee
Program Organizers: Tzong T. Chen, Natural Resources Canada, CANMET, Ottawa, Ontario K1A 0G1 Canada; Ann M. Hagni, Construction Technology Laboratories, Inc., Microscopy Group, Skokie, IL 60077 USA; J. Y. Hwang, Michigan Technological University, Institute of Materials Processing, Houghton, MI 49931-1295 USA

Tuesday PM Room: 2010
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Arun M. Gokhale, Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30318 USA

2:00 PM
An Investigation of Mechanical Behavior and Damage Evolution of Ti-6Al-4V-Al3Ti Metal Intermetallic Laminate (MIL) Composites: *Tiezheng Li*[1]; Marc A. Meyers[1]; Kenneth S. Vecchio[1]; Engene A. Olevsky[2]; [1]University of California, Mechl. & Aeros. Engrg., 9500 Gilman Dr., UCSD-0411, La Jolla, CA 92093-0411 USA; [2]San Diego State University, Mechl. Engrg., 5500 Campanile Dr., San Diego, CA 92182-1323 USA

The mechanical performance of Ti-6-4-Al3Ti metal-intermetallic laminate (MIL) composites synthesized by a reactive foil sintering technique was evaluated. Crack morphology of untested Ti-6-4-Al3Ti MIL composites has been characterized by optical microscopy. Mechanical tests were performed on Ti-Al3Ti metal-intermetallic laminate (MIL) composites and pure Al3Ti, and the principal mechanisms of damage initiation and accumulation were identified experimentally. The elastic properties and anisotropy of the laminates were calculated and successfully compared with Resonant Ultrasonic Spectroscopy

(RUS) measurements. The effect of residual stress on the fracture toughness of the Ti-6-4-Al3Ti MIL composites was analyzed.

2:25 PM
Stereological Unfolding to Determine the Three-Dimensional Bivariate Size and Shape Distribution of TiB Whiskers in Ti-6Al-4V-2.9B: *Scott Lieberman*[1]; David Mebane[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332 USA

Metal matrix composite (MMC) consisting of TiB whiskers distributed in a Ti-6Al-4V alloy matrix was produced using blended elemental powder metallurgy and compaction of Ti-6Al-4V-2.9B. In such composites an important aspect of microstructure representation is the size and shape distribution of the whiskers. The desired three-dimensional bivariate size and shape distribution of TiB whiskers has been estimated from measurements performed on a two-dimensional metallographic section by using a recently developed stereological unfolding procedure. The distribution confirmed that the orientation of TiB whiskers is random in three dimensions, and that whisker growth is anisotropic with an increase in aspect ratio with increased length.

2:50 PM
Characterization of Microstructural Evolution in AlMgSi(Cu) Alloys Using Calorimetry Methods: *Shahrzad Esmaeili*[1]; Warren J. Poole[2]; David J. Lloyd[3]; [1]University of Waterloo, Dept. of Mechl. Engrg., 200 Univ. Ave. W., Waterloo, Ontario N2L 3G1 Canada; [2]University of British Columbia, Dept. of Metals & Matls. Engrg., 309-6350 Stores Rd., Vancouver, British Columbia V6T 1Z4 Canada; [3]Alcan International Limited, Kingston R&D Ctr., PO Box 8400, Kingston, Ontario K7L 5L9 Canada

Until recently, the evaluation of microstructural evolution during age hardening in the automotive AlMgSi(Cu) alloys was considered a difficult task due to a combination of factors including the type and low concentration of solutes, the complicated precipitation sequence and small size of precipitates. Two analytical methods based on isothermal calorimetry and differential scanning calorimetry have been recently developed which provide practical tools for the characterization of microstructural evolution in the aging regimes where precipitation hardening occurs in these alloys. The present work summarizes the basic procedures, as well as the combined analytical and modeling approach to determine the relative volume fraction and size of precipitates in commercially relevant aging treatments.

3:15 PM
Acoustic Properties of Ni3Si Intermetallic Materials: Shih-Jeh Wu[1]; *Shian-Ching Jason Jang*[2]; Chen-Ming Kuo[1]; Dong-Yih Lin[2]; Chen-Ching Ting[1]; [1]I-Shou University, Dept. of Mechl. Engrg., 1, Sect. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan; [2]I-Shou University, Dept. of Matls. Sci. & Engrg., 1, Sect. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

Intermetallic compounds are defined as a mixture in specific proportion of two metallic elements that form a periodic crystalline structure different from those of the original elements. Ni3Si intermetallic is a popular structural material in aerospace engines and acidulous environment due to its good mechanical integrity and acid resistance at elevated temperatures. The mechanical properties were investigated by ultrasonic method non-invasively from different forming processes at different temperatures. The Young's and shear moduli can be calculated easily from their relationship with longitudinal and shear acoustic speeds. Experimental data also showed the ultrasonic attenuation is quite different from that of ordinary metals or alloys. The much stronger attenuation may be related to the special L12 structure of Ni3Si and indicate the microstructure change at different compounds.

3:40 PM Break

3:50 PM
Study of Long-Term Durability of Aluminum Lithium Alloys: *Hajer Mahmoud Awatta*[1]; Ali Merati[2]; Marko Yanishevsky[2]; Vivier Lefebvre[3]; [1]Carleton University, Dept. of Mechl. & Aeros. Engrg., 1125 Col. By Dr., Ottawa, Ontario K1S 5B6 Canada; [2]National Research Council Canada, Inst. for Aeros. Rsch., 1200 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada; [3]Department of National Defence, NRC/IAR, 1200 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada

Weight reduction of aerospace vehicles is best achieved by decreasing the density of aircraft structural materials. However, the development of Al-Li alloys has faced several technical problems, which include high anisotropy of mechanical strength, low stress corrosion threshold and low ductility. Long-term performance, in particular fatigue and corrosion behaviours, are also concerns that need to be further studied. Al-Li alloys have been used in the new fleets of the search and rescue helicopters to replace most of the conventional aircraft alloys such as 7075-T6 and 2024-T3 due to their low density and high elastic modulus. The present work investigates Al-Li alloy durability issues to provide proactive support to the Canadian Forces.

4:15 PM
Rheological Study of the Foundry 356 Al-Alloy Prepared by a New Innovated SSM Process: *Omid Lashkari*[1]; Reza Ghomashchi[1]; [1]University of Quebec, Applied Scis., DSA, UQAC, 555, Blvd. de l'Univ., Chicoutimi, Quebec G7H 2B1 Canada

SEED (Swirled Enthalpy Equilibrium Device) is established itself as a new technology amongst the SSM processes, which uses swirling as agitator parameter of the molten metal and dendrite breaking operant within mushy zone state. Swirling causes primary particle evolution during solidification. Swirling changes the dendritic microstructure and creates different primary phase size and distribution. In the current research, variation of microstructure due to swirling intensity has been studied by an image analysis system and a simple parallel plate test machine, i.e. rheological test machine. Microstructure and viscosity of the new SSM 356 Al-alloy has been proportioned by the rheological tests.

4:40 PM
Quantitative Linking of Ceramic Matrix Composite Microstructure with Impedance Spectra: *Rosario A. Gerhardt*[1]; David S. Mebane[1]; [1]Georgia Institute of Technology, Matls. Sci. & Engrg., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

The orientation and length-radius distributions of silicon carbide whiskers embedded in alumina are shown to correlate with the composite's resistivity. The resistivity was measured using impedance spectroscopy. Stereological unfolding techniques are applied to SEM images in order to measure the size-orientation distributions. A mathematically defined, stochastic microstructural simulation provides a link between the measured microstructure and percolating volume fraction. Percolating volume fraction, as measured through the simulation, is shown to be proportional to high-frequency features in the impedance spectra for Al2O3-SiCw composites of 10 and 20% SiCw by volume. These results suggest a general method for linking composite microstructure to AC electrical response of many different composite systems.

5:05 PM
Characterization of Heat-Treatable Aluminum Matrix Composite from Recycled Aluminum Cans Alloys Containing Boron as Reinforcers: *Ely X. Colon*[1]; O. Marcelo Suarez[2]; [1]University of Puerto Rico, Mechl. Engrg. Dept., PO Box 9045, Mayagüez, PR 00681-9045 USA; [2]University of Puerto Rico, Gen. Engrg. Dept., PO Box 9044, Mayagüez, PR 00681-9044 USA

The present project proposes the fabrication of a novel series of cast aluminum matrix composites (AMC), using aluminum-boron master alloys and aluminum-magnesium from recycled aluminum cans. This new AMC series do not present several deleterious effects found in currently available cast AMC containing ceramic reinforcements such as SiC, graphite, and Al2O3 particulates. The resulting material has low cost of production and low reactivity between reinforcing particles and matrix. Mg is incorporated as an alloying element to improve ageing characteristics of the composite that boosts hardness in these precipitation-hardened Al-Mg-B composites. The Mg-containing alloy is of the AA 5XXX series provided by clean used beverage cans. The present investigation represents a baseline of research for further investigations that are being conducted on Al-Cu-B, Al-B-Cu-Mg and Al-B-Mg composites. Microhardness measurements, Scanning Electron Microscope (SEM) and X-Ray Diffraction are being used to test and characterize the resulting composites.

Computational Aspects of Mechanical Properties of Materials: Meso-Scale and Continuum Modeling

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)

Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA

Tuesday PM Room: 3012
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Michael J. Mills, Ohio State University, Matls. Sci. & Engrg., Columbus, OH 43210 USA; Nik Chawla, Arizona State University, Dept. of Cheml. & Matls. Engrg., Tempe, AZ 85287 USA

TUESDAY PM

2:00 PM
A Data-Mining Approach for the Design of Optimized Polycrystalline Materials: *Veeraraghavan Sundararaghavan*[2]; Nicholas J. Zabaras[1]; [1]Cornell University, Matls. Process Design & Control Lab., Sibley Sch. of Mechl. & Aeros. Engrg., 188 Frank H. T. Rhodes Hall, Ithaca, NY 14853-3801 USA; [2]Cornell University, Matls. Process Design & Control Lab., Sibley Sch. of Mechl. & Aeros. Engrg., 169 Frank H. T. Rhodes Hall, Ithaca, NY 14853-3801 USA

A data-driven reduced-order optimization procedure is presented for the design of deformation process sequences for controlling texture and texture-dependent properties. The inverse problem of identifying processing stages leading to a desired texture is initially solved using an unsupervised data-mining methodology based on the x-means algorithm. The hierarchical classifier matches the lower-order ODF features in the form of pole density functions of important orientation fibers and associates the desired texture to a class of pre-existing textures within a database. Texture classes in the database are affiliated with processing information, hence, enabling identification of multiple process paths that lead to a desired texture. The process parameters are fine-tuned using a gradient optimization algorithm driven by continuum sensitivity analysis of texture evolution. An adaptive reduced-order model based on proper orthogonal decomposition is employed wherein the texture modes corresponding to the intermediate stages of the design process are adaptively selected from the database. Further, the database continuously improves during the optimization problem through addition of new, unknown data sets, which would be useful during future optimization runs.

2:20 PM
Predicion of Crystallographic Texture Evolution and Anisotropic Stress-Strain Curves in α-Titanium Using a Taylor-Type Crystal Plasticity Model: *Xianping Wu*[1]; Surya R. Kalidindi[1]; Roger D. Doherty[1]; [1]Drexel University, Dept. of Matls. Sci. & Engrg., 3141 Chestnut St., Philadelphia, PA 19104 USA

A Taylor-type polycrystalline model has been developed to simulate the evolution of crystallographic texture during large deformation processing of α-Titanium at room temperature. In addition to slip, deformation twinning has been incorporated as an additional plastic deformation mode in this model. Each activated family of twins in a grain is addressed as a quasi-independent grain that will undergo further deformation and rotation independently, but its volume fraction and orientation are updated at the end of each time step. Slip inside twinning has been allowed in this computation. New hardening functions for both slip and twinning are provided in this model. Good prediction of the overall stress-strain response and texture evolution in a number of different deformation modes were obtained using this newly developed crystal plasticity model.

2:40 PM
3D Characterisation and Modelling of an Aluminium Matrix Composite Using X-Ray Microtomography: *Ian G. Watson*[1]; Peter D. Lee[1]; Richard J. Dashwood[1]; [1]Imperial College London, Dept. of Matls., Prince Consort Rd., S. Kensington, London SW7 2BP UK

The high strength and stiffness of particulate-reinforced aluminium matrix composites is well documented. The size, morphology, orientation and distribution of the reinforcement particles are critical parameters in achieving optimum stiffness, strength and fatigue resistance. Understanding the interaction of the reinforcement particles with the matrix alloy is a dual-scale problem. On the micro-scale, individual reinforcement particles coagulate during slow cooling to form clusters. The local strengthening effect of these clusters depends on their packing density and morphology. On the macro-scale, the three dimensional (3D) distribution of the clusters dictates the overall efficiency of load transfer. Traditionally, two-dimensional (2D) visualisation techniques such as optical and scanning electron microscopy have been used to characterise composite microstructures. However, the extrapolation of 3D morphologies from 2D images is only valid if the material is not textured and the features are equiaxed and of uniform size. In this investigation x-ray microtomography (XRMT) was used to characterise the complex three dimensional morphology and distribution of TiB2 clusters in an aluminium alloy matrix. A finite element model was used to estimate the local stiffening and strengthening effects of an individual cluster. This representative volume element (RVE) was combined with the 3D cluster distribution obtained via XRMT in a macroscale model to determine the elastic-plastic response of the MMC as a whole.

3:00 PM
Simulation of Mechanical Behaviors of Die-Cast Mg Alloy Based on Three-Dimensional Porosity and Finite Element Method: *Soon Gi Lee*[1]; Arun Sreeranganathan[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., NW, Atlanta, GA 30332-0245 USA

Porosity is one of the most important microstructural attributes of die-cast Mg-alloys. Several features of porosity such as the amount, geometry, and locations have dominant influence on the mechanical behavior of die-cast Mg alloys. Therefore, it is of interest to incorporate quantitative description of actual three-dimensional porosity in micro-mechanical analysis of the cast alloys. In this contribution, a montage-based serial sectioning technique has been applied for constructing actual three-dimensional porosity, and the finite element (FE)-based simulations have been performed on the 3D microstructure of a high-pressure die-cast Mg-alloy containing "real" pores to reveal the relationships between the distributions of local stresses and strains and size, orientation, and spatial arrangement of the porosity.

3:20 PM
Rapid Evaluation of Mechanics of Defects in Casting: *Yi Cheung Lok*[1]; Adam Clayton Powell[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Engrg., 77 Mass. Ave., Cambridge, MA 02139 USA

The presence of porosity has always been a major concern in castings. While the detrimental effect of porosity on the mechanical properties of casting is well established, their presence in varying amount throughout the casting can often be tolerated. Numerical simulations on the stress state of a component can provide an insight into the optimal tolerance level and configuration of defects. The simulation is done using the Boundary Element Method (BEM) because it only requires surface meshing and thus allows for geometrical changes without complete mesh regeneration. This enables a rapid solution regeneration for various interior defect configurations because the inverted matrix corresponding to the outer part surface is reusable for subsequent simulations. Result of the simulation might help foundry engineer design castings with lower a rejection rate.

3:40 PM Break

3:50 PM
Variability of Fatigue Life in Two-Phase Alloys: *Kwai S. Chan*[1]; Yi-Der Lee[1]; Michael P. Enright[1]; [1]Southwest Research Institute, Matls. Engrg. Dept., 6220 Culebra Rd., San Antonio, TX 78238 USA

Experimental evidence indicates that a large variability in fatigue life can exist in two-phase alloys at or near the fatigue limit. This variability is generally accompanied by a transition of fatigue crack initiation site from surface grains to internal grains. To better understand these experimental observations, theoretical models have been developed to compute fatigue life variations of two-phase alloys resulting from three possible scenarios: (1) hard and soft phases in the two-phase microstructure, (2) planar slip versus cell-forming grains, and (3) crack initiation versus crack growth. The theoretical results are utilized to elucidate the effects of microstructure, slip morphology, and grain location on fatigue life variability in two-phase alloys, using Ti-6Al-4V as an illustration. Work supported by AFOSR MEANS Program through Contract No. F49620-01-1-0547, Dr. Craig S. Hartley, Program Manager.

4:10 PM
Microstructure-Based Modeling of Crack Growth in Particle Reinforced Metal Matrix Composites: *Adarsh Ayyar*[1]; Jason Williams[2]; Nik Chawla[2]; [1]Arizona State University, Dept. of Mechl. & Aeros. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287 USA; [2]Arizona State University, Dept. of Cheml. & Matls. Engrg., Fulton Sch. of Engrg., Tempe, AZ 85287 USA

Crack growth in SiC particle reinforced Al matrix composites is significantly influenced by the size, distribution, and morphology of the SiC particles. Reinforcement particle clustering also influences the mechanical behavior of metal matrix composites. Hence, to accurately model the crack growth in such a system it is important that the complex microstructure of the particles be taken into account and not simplified by circles or ellipses. In this paper, the effects of particle morphology and distribution (homogenous and clustered) on crack growth have been studied using the finite element method. The degree of particle clustering in aluminum/silicon-carbide composites was quantified by the coefficient of variance in the mean near-neighbor particle spacing, and cluster size distributions using an image analysis technique. The clustering analysis correlated well with the observed microstructures. Two dimensional linear elastic fracture mechanics principles were used to propagate the crack and obtain the local stress intensity values, and to obtain an understanding of the local stress state. Predictions from this analysis correlated well with experimental observations of crack growth in these systems.

4:30 PM
The Effect of Colony Orientation on Deformation Behavior and Slip Transmission During Hot Working of Ti-6Al-4V Single-Colony Samples: *A. A. Salem*[1]; S. L. Semiatin[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

The deformation behavior of individual alpha/beta colonies of Ti-6Al-4V has been established under hot working conditions. Constant strain-rate uniaxial compression tests were conducted on samples cut from single-colony crystals that were grown using a float zone technique. Each sample was oriented for slip along a different prismatic slip system in the alpha-phase. The mechanical behavior exhibited a strong dependence on colony orientation. The apparent anisotropy in the critical resolved shear stress was explained based on the burgers orientation relationship between the alpha (hcp) and beta (bcc) phases of the colonies and hence the orientations of alpha slip directions relative to those in the beta phase.

4:50 PM
Prediction of Creep Behavior of Ti-6Al-4V on the Basis of Time Dependent Models Involving Microstructural Parameters and Mechanical Properties: *Dhriti Bhattacharyya*[1]; Sujoy Kar[1]; John J. Schirra[2]; Michael Savage[2]; Walter W. Milligan[3]; Hamish L. Fraser[1]; Michael J. Mills[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Pratt & Whitney, Matls. & Processes Dvlp., Structural Alloys; [3]Michigan Technological University, Dept. of Matls. Sci. & Engrg., Houghton, MI USA

The creep behavior of titanium alloys is an important factor in the life expectancy of crucial service parts in aircraft engines. Room temp creep exists, and should be incorporated in designs. In this work, an attempt has been made to predict the creep behavior and mechanisms of the Ti alloy Ti-6Al-4V at room temperature. For this purpose, extensive mechanical tests, including tensile and creep tests, have been done on a set of alpha-beta-forged samples with different heat treatment parameters in order to obtain a large range of microstructures. The tensile curves have been fitted with a time-dependent viscoplastic model to obtain parameters like the strain hardening exponent n and the strain rate sensitivity m. These results, combined with measured microstructural parameters such as grain size and volume fraction of primary alpha, have been used to feed an artificial neural network model. This model is subsequently used to predict the creep behavior of Ti-6Al-4V with arbitrary heat treatment schedules and microstructural parameters. These results will also be discussed in light of existing knowledge of room temperature creep mechanisms in the two-phase titanium alloys.

5:10 PM
Strength Predictions in Sintered "Tough Coated Hard Particle" Systems: *Ivi Smid*[1]; Rick E. Toth[2]; [1]Pennsylvania State University, Engrg. Sci. & Mech., 147 Rsch. W., Univ. Park, PA 16802-6809 USA; [2]Allomet Corporation, 509 Hahntown-Wendel Rd., N. Huntingdon, PA 15642 USA

Tough coated hard particles (TCHP) are a new microstructure designed to offer high performance levels in cutting tools, wear components and forming dies. Control of the sintered microstructure is critical to tailor and promote optimal property combinations. If preserved into the final densified microstructure, the coated hard particles will open new, unprecedented performance combinations. The TCHP concept will be introduced, and the theoretical basis for new performance combinations via various powder and coating and matrix phase combinations will be shown. Results of liquid phase sintering experiments will be presented with model TCHP powders, showing the needed balance between liquid content, thermodynamic reactions during heating, time and temperature with respect to densification, grain size and grain shape changes, and preservation of the desired coated grain microstructure. A calibration of strength predictions based on filler-particle/matrix adhesion and particle shape, and results on performance will be presented.

5:30 PM
Modeling of Grinding Dynamics Using Second Order Dynamic System: *Olga Karabelchtchikova*[1]; Simon M. Hsiang[2]; [1]Worcester Polytechnic Institute, Matls. Sci. & Engrg., 100 Inst. Rd., Worcester, MA 01609-2280 USA; [2]Texas Tech University, Industl. Engrg., Box 43061, Lubbock, TX 79409-3061 USA

A second order dynamic system is proposed to model residual stresses (RS) distributions under various heat treatment procedures and grinding dynamics. The study was motivated to (1) predict the magnitude of the RS and tensile peak location, and (2) establish superposition relationship in the RS distribution due to number of grinding passes. Grinding dynamics was represented as a lumped system composed of spring-mass-damper of the grinding machine and damper-stiffness of the workpiece. A nested factorial experiment of 18 conditions with 3 levels of tempering factor, 2 levels of grinding conditions and 3 multipass grinding levels was used for the model development and validation. The proposed model depicts changes of the complex experimental conditions and demonstrates a good estimation of the subsurface RS distribution. With only one parameter involved, the prediction elucidates grinding dynamics and supports a theory of the heat treatment and grinding effects on the material characteristics.

Computational Thermodynamics and Phase Transformations: Atomistic and Ab Initio Methods

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Corbett C. Battaile, Sandia National Laboratories, Materials and Process Modeling Department, Albuquerque, NM 87185-1411 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Tuesday PM — Room: 3005
February 15, 2005 — Location: Moscone West Convention Center

Session Chair: Mark T. Lusk, Colorado School of Mines, Mechl. Engrg. Prog., Golden, CO 80401 USA

2:00 PM Invited
Elucidating Kinetic Pathways for Martensitic Phase Transformations in Crystals: *Emily Ann Carter*[1]; Kyle J. Caspersen[1]; [1]Princeton University, Mechl. & Aeros. Engrg., The Engrg. Quad., Princeton, NJ 08544 USA

A method for characterizing the minimum energy path for solid-solid phase transitions is presented. The technique involves a generalization of the so-called Nudged Elastic Band method for finding the minimum energy path for atomic and molecular reaction paths. Instead of only minimizing the forces on the atoms to find critical points, we also minimize the Cauchy stress on the unit cell of the crystal, in order to find saddle points for the conversion of one phase of material to another. We can also calculate the minimum energy path in the presence of external pressure, so that pressure-induced phase transformations may be explored at the atomistic level. The method can be used with any energy representation of a material, however, we focus here on applications using first principles methods, specifically density functional theory. Applications include, but are not limited to, metallic phase transformations.

2:30 PM
A First Principles/Kinetic Monte Carlo Study of Oxygen Diffusion in YSZ-Based Oxides: *Ramanathan Krishnamurthy*[1]; Young-Gui Yoon[1]; Konstantin Kudin[1]; Roberto Car[1]; David J. Srolovitz[1]; [1]Princeton University, Princeton Inst. for the Sci. & Tech. of Matls. & Dept. of Mechl. & Aeros. Engrg., 70 Prospect Ave., Princeton, NJ 08542 USA

Understanding oxygen diffusion in yttria stabilized zirconia based oxides is of paramount importance for their effective use in

applioations. We present a multi-scale model to study oxygen diffusion in YSZ. We employ density functional theory methods to calculate activation energies for oxygen migration in different local cation environments. These results serve as input to a kinetic Monte Carlo study of oxygen diffusivity as a function of temperature and yttria content. The simulations, in agreement with experiment, show that the oxygen diffusivity attains a maximum value around 10 mol% yttria. The oxygen vacancy concentration, diffusion activation energy and correlation effects all vary differently with yttria content, producing the behavior described above when their collective effects are considered. A simple analysis of dopant-induced correlations supports this explanation. This methodology is extended to study oxygen diffusion in similar oxides. The results are discussed with regard to their implications for several applications.

2:50 PM
Kinetic Monte Carlo Study of a Mechanism for Coalescence of Precipitates: *Zugang Mao*[1]; Georges Martin[2]; *David N. Seidman*[1]; [1]Northwestern University, Matls. Sci. & Engrg., Cook Hall, 2220 Campus Dr., Evanston, IL 60208-3108 USA; [2]French Atomic Energy Commission, Paris France

The decomposition of a Ni-Al-Cr alloy at 873 K is studied by kinetic Monte Carlo (KMC) simulation. In agreement with our three-dimensional atom-probe microscope experiments we find that a significant fraction (30%) of the gamma-prime (L12) precipitates coarsen via coalescence and that the fraction coalesced is a function of aging time. The atomic scale mechanism for our observations is studied by KMC simulation. A key quantity is the monovacancy-solute binding energy, which is shown to control the presence or absence of coalescence. It is demonstrated that the mechanism involves the formation of clusters of Al and Cr solute atoms (dimers, trimers, quadramers, and pentamers) all of which are more mobile than Al or Cr monomers. The diffusive behavior of these higher order clusters is studied and it is shown that they undergo both 2D and 3D motion, and that the 2D diffusivity is greater than the 3D diffusivity.

3:10 PM
Self-Diffusion Mechanisms in Two-Dimensional Metals: Gennady Mikhailovich Poletaev[1]; *Mikhail Dmitrievich Starostenkov*[1]; Julia Vladimirovna Patzeva[1]; [1]Altai State Technical University, Gen. Physics Dept., Lenin st., 46, Barnaul 656038 Russia

The paper is concerned with the research of self-diffusion mechanisms in two-dimensional crystals of Ni, Al, Cu metals by the method of molecular dynamics. The packing of two-dimensional metals in a computer model was corresponded to the plane (111) of FCC lattice. Morse pair potentials and Finnis-Sinclair multipartial potentials were used in the researches. Different self-diffusion mechanisms were studied. Activation energies of the mechanisms, the contribution of every mechanism into diffusion process in the dependence on temperature were calculated. It was found, that vacancy mechanism (the vacancy was formed by Shottki method) and the mechanism of the formation and annihilation of Frenckel pairs made major contribution into self-diffusion in two-dimensional metals. Frenckel pair forms in the crystal in the result of the crossing of thermal crawdion displacements. The other diffusion mechanisms influence on self-diffusion in two-dimensional metals not so gritty.

3:30 PM
Kinetics of Precipitation in Al-Zr-Sc Alloys: From Atomic to Mesoscopic Models: *Emmanuel Clouet*[1]; Maylise Nastar[1]; Christophe Sigli[2]; [1]Service de Recherches de Metallurgie Physique, CEA/Saclay France; [2]Pechiney, Centre de Recherches de Voreppe France

Zr and Sc precipitate in aluminum alloys to form the compounds Al3Zr, Al3Sc, and Al3ZrxSc1-x which for low super-saturations of the solid solution have the L12 structure. The aim of the present study is to model at an atomic scale this kinetics of precipitation and to build a mesoscopic model based on classical nucleation theory so as to extend the field of super-saturations and annealing times that can be simulated. In this purpose, we use some ab-initio calculations and experimental data to fit an Ising model describing thermodynamics of the Al-Zr-Sc system. Kinetics of precipitation are studied using a kinetic Monte Carlo algorithm based on an atom-vacancy exchange mechanism. These simulations show that in ternary Al-Zr-Sc system Zr addition mainly affects nucleation whereas Sc addition influences nucleation as well as growth and coarsening stage. This allows us to understand experimental results showing that a Zr addition to an Al-Sc alloy leads to finer precipitates and increases their density. Structure of precipitates obtained in these simulations, a Sc rich hearth surrounded by Zr rich shelves, agrees with the one observed with 3D atom-probe.[1] A comparison of these kinetics of precipitation for Al-Zr and Al-Sc binary systems with prediction of the classical nucleation theory shows that the nucleation stage can be well reproduced by mesoscopic models as long as the short range order tendency of the system is considered.[2] This result is then used so as to build a mesoscopic model allowing to study nucleation in the ternary Al-Zr-Sc system. [1]B. Forbord, W. Lefebvre, F. Danoix, H. Hallem, and K. Marthinsen. Three dimensional atom probe investigation on the formation of Al3(Sc,Zr) dispersoids in aluminium alloys. Scripta Mater., 51:333 337, 2004. [2]E. Clouet, M. Nastar, and C. Sigli. Nucleation of Al3Zr and Al3Sc in aluminum alloys: from kinetic Monte Carlo simulations to classical theory. Phys. Rev. B, 69:064109, 2004.

3:50 PM Break

4:00 PM Invited
Phonon Transport and Scattering at the Nanoscale: *Patrick Kenneth Schelling*[1]; Simon R. Phillpot[2]; Brian Becker[1]; [1]University of Central Florida, AMPAC, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA; [2]University of Florida, Dept. of Matls. Sci. & Engrg., 162 Rhines Hall, Gainesville, FL 32611 USA

Phonon wave-packet dynamics is a powerful technique for gathering detailed information about phonon dynamics, scattering, and thermal transport. In this talk, I will present recent result for the scattering of phonons at silicon grain-boundaries, silicon nanowires, and carbon nanotubes. I will describe recent conceptual improvements that have allowed us to compute experimentally-measurable quantities in a one-shot simulation, without the need for multiple simulations previously needed to sample the entire Brillouin zone. Finally, I will show how this work can lead to the development of mesoscale models of thermal transport.

4:30 PM Invited
Adjusting the Melting Point of a Model System via Gibbs-Duhem Integration: Application to a Model of Aluminum: *Brian B. Laird*[1]; Jess B. Sturgeon[2]; [1]University of Kansas, Dept. of Chmst., Lawrence, KS 66045 USA; [2]Lawrence Livermore National Laboratory, 7000 E. Ave., Livermore, CA 94550 USA

Model interaction potentials for real materials are generally optimized with respect to experimental properties that are easily evaluated as mechanical averages (e.g., elastic constants (at T=0K), static lattice energies and liquid structure). Agreement with experiment for the non-mechanical properties, such as the melting point, is not guaranteed and such values can deviate significantly from experiment. We present a method for re-parameterizing any model interaction potential of a real material to adjust its melting temperature. This is done without significantly affecting other mechanical properties of the system. This method is an application of Gibbs-Duhem integration [D. Kofke, Mol. Phys.78, 1331 (1993)]. As a test we apply the method to an embedded atom model of aluminum [J. Mei and J.W. Davenport, Phys. Rev. B 46, 21 (1992)] for which the melting temperature for the thermodynamic limit is 826.4 ± 1.3K - somewhat below the experimental value of 933K. After re-parameterization, the melting temperature of the modified potential is found to be 931.5K ± 1.5K.

5:00 PM
The Research of Evolution of Microstructure in Two-Dimensional Crystals Cu3Au and Ni3Al at Phase Transition Order-Disorder: *Mikhail Dmitrievich Starostenkov*[1]; Gennady Mikhailovich Poletaev[1]; Natalia B. Cholodova[1]; Irina A. Demina[2]; [1]Altai State Technical University, Gen. Physics Dept., Lenin st., 46, Barnaul 656038 Russia; [2]East Kazakhstan State University, Ust Kamenogorsk Kazakhstan

The mechanisms of the disordering process of two-dimensional crystals Cu3Au and Ni3Al were studied by the method of molecular dynamics. The order of crystals was corresponded to L12 superstructure. The two-dimensional crystal was presented by the packing of atoms corresponding to the plane (111). The crystal block was subjected to the impulsive heating to the definite temperature. The following diffusion mechanisms of the disordering of alloys were found: crawdion, ring, vacancy and mechanism of Frenckel pairs formation (vacancies and interstitial atoms). Every mechanism began to function from the definite temperature of impulsive heating in the sequence, as it was above mentioned. The temperature of the beginning of disordering process activation was lower for the alloy Cu3Au. Frenckel pairs contributed greatly in the evolution of microstructure at high temperatures. It was shown, that the diffusion coefficient is integral characteristic, depending on all the mechanisms of diffusion reconstruction of the material.

5:20 PM
The Research of Mechanical Stability of Two-Dimensional Metallic Composites: *Mikhail Dmitrievich Starostenkov*[1]; Gennady Mikhailovich Poletaev[1]; G. V. Popova[2]; [1]Altai State Technical Univer-

sity, Gen. Physics Dept., Lenin st., 46, Barnaul 656038 Russia; [2]East Kazakhstan State University, Ust Kamenogorsk Kazakhstan

The stability of interphase boundaries in two-dimensional thin films of composites, consisting of the phases Ni3Al-Al and Ni3Al-Ni is studied in the dependence on deformation of hydrostatic pressure and uniaxial tension. The structure of composition material is given by different variants of packing of Ni3Al intermetallid phases and pure Al or Ni metals. Physical and physics-mechanical properties of the composite are connected with the structure of interphase boundaries. Washing process of interphase boundaries begins with the growth of temperature at the expense of different types of diffusion mechanisms. The temperature of the beginning of the transformation stage of interphase boundaries is connected with the type of metallic layer, given between the phases of Ni3Al intermetallid. Washing process begins at higher temperatures (in comparison with Al layer) in the layer, consisting of pure Ni. The deformation changes the temperature of the beginning of washing process of interphase boundary.

Converter and Fire Refining Practices: Processing Fundamentals

Sponsored by: Extraction & Processing Division, EPD-Pyrometallurgy Committee
Program Organizer: Alistair G. Ross, INCO, Ltd., Canadian Smelting & Copper Business, Copper Cliff, P0M 1N0 ON Canada

Tuesday PM Room: 2016
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Tony Warner, Inco Ltd, Techl. Serv., Mississauga, Ontario L5K 1Z9 Canada; Cameron Harris, H.G. Engineering Ltd., Toronto, ON M6S 3G3 Canada

2:30 PM
Copper Converting Fluxing Practise During Instantaneous High Arsenic Containing Feed Mixture in Harjavalta: *Kim Olof Fagerlund*[1]; Pekka Pyykkö[2]; Pekka A. Taskinen[1]; [1]Outokumpu Research, Kuparitie 10, Box 60, Pori FIN-28101 Finland; [2]Harjavalta Copper, Teollisuuskatu 1, Harjavalta FIN-29200 Finland

Boliden Harjavalta Oy operates a copper smelter in Finland, which is based on flash smelting together with Peirce-Smith converting of copper matte. This paper reviews a case study when a high arsenic feed mixture was successfully eliminated in order to guarantee the anode quality. Based on theoretical and practical knowledge in slag chemistry at Outokumpu Research, the CaO-fluxing practice was introduced. Description of a thermodynamic simulation using MTDATA and implementation of the results into converting practice is given. Results showed that the impurity fluctuations in raw materials could be effectively controlled by efficient utilization of different slag chemistry.

3:00 PM
Controlling the Processing Parameters Affecting the Refractory Requirements for Peirce-Smith Converters and Anode Refining Vessels: *Anthony J. Rigby*[1]; [1]RHI Canada, 4355 Fairview St., Burlington, ON L7L 2A4 Canada

Refractory linings in converters or anode furnaces used to overoxidize the blister copper will suffer rapid deterioration, due to prolonged contact with copper oxide-rich slag. Nitrogen stirring with COP KIN porous plugs and use of SEMTECH OPC in converters and anode refining vessels can considerably reduce both refractory wear and processing times. In addition, careful control of the vessel atmosphere and the use of real-time optical process control minimize both slag volume and contact time with the vessel refractory.

3:30 PM
Metallurgical Considerations for Recycling Siliceous Bearing Materials in the Mitsubishi Continuous Copper Converter: *Fumito Tanaka*[1]; [1]Mitsubishi Materials Corp., Central Rsch. Inst., 1-297 Kitabukuro-cho, Omiya-ku, Saitama 330-8508 Japan

The Mitsubishi Process is the sole pyrometallurgical process for the continuous production of blister copper from copper concentrates. Continuous converting originates from the application of the proven technology of a lime-ferrite slag (CaO-FeOX-Cu2O) on the Converting Furnace (C-furnace). Recent improvements on the C-furnace operation have included increasing the processing of recycled materials and the investigation of alternative fluxing agents. However, some minor oxides adversely affect C-furnace operation, with respect to magnetite behavior of the slag, with the most significant impact having been observed with the silica content of the slag. This paper will quantify the impact of silica on the lime-based slag, and discusses the controls that have been identified to sustain continuous converting operation.

4:00 PM Break

4:15 PM
Minor Element Control by Vacuum Calcination and Recycling of Copper Smelter Dust: *Jin Qiu*[1]; Ralph Harris[1]; [1]McGill University, Mining, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

Experiments were performed to determine the extent of minor element elimination that occurred when mixtures of copper smelter dust and copper concentrate, were calcined under vacuum. Almost complete removal of the As, Sb, Bi and Pb occurred due to the conversion of the minor element compounds of these elements into their highly volatile sulphides by reaction with sulphur from the concentrate. Heat and mass balance calculations of the slag-blow in a copper converter found that ~30 tonnes of the vacuum calcine could be used to produce 100 tonnes of copper as white metal. The use of dirty concentrate as the sulphidizing agent in the vacuum calcination means that the minor element treatment capacity of the smelter complex could be increased by 60%. Also the productivity of the converters may be increased by up to 8% due to the supply of copper values and oxygen in the calcined mixture.

4:45 PM
Control of Magnetite Formation During Slag-Making in Copper P-S Converter: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Mount Isa Mines Limited, Metallurgl. Plants Bldg., Private Mail Bag 6, Mt. Isa, Queensland 4825 Australia

Converter slag chemistry control has played an important role in optimizing converter operations. A thermodynamic model of copper P-S converter has been developed to predict the behavior of magnetite in the converter slag. Predictions of bath temperature, slag and matte compositions, and magnetite content in the slag have been validated by industrial data. The effects of fluxing strategy, returns and skim charges, oxygen enrichment, and temperature on the magnetite formation in the slag have been predicted and discussed. Some improvements of the industrial operations have been presented.

5:15 PM
Physicochemical Study of Reduction of Nickel Concentrate Calcine With Products of Vapor Oxidation of Fuel Oil and Solid Carbon: *A. V. Tarasov*[1]; *V. M. Paretsky*[1]; V. A. Bryukvin[1]; [1]State Research Center of Russian Federation, 13, Acad. Korolyov St., 129515, Moscow Russia

To carry out a physicochemical substantiation of a transition to "soft" modes of reduction of calcine obtained by desulfurizing roasting of nickel concentrate produced as a result of copper-nickel converter matte separation in a nickel anode production circuit, an analysis has been conducted of the macrokinetic behavior of actual calcine samples in the presence of gaseous H2-CO mixtures and solid reductant additives. Within a temperature range of 900C to 1100C the main regularities typical of the calcine reduction process with individual gases (H2, CO) also take place when using a gaseous mixture. Under the conditions of low-temperature (900C to 1000C) nickel oxide reduction with products of fuel oil conversion in the presence of coke, the role of the latter is insignificant from the technological viewpoint. As a result of the present investigations, we have obtained analytical expressions of macrokinetic parameters of the reaction interaction within the [NiO] calcine-H2-CO-C-CO2-H2O-O2-N2 system.

Extractive Metallurgy: Copper

Sponsored by: Extraction & Processing Division, EPD-Aqueous Processing Committee, EPD-Pyrometallurgy Committee, EPD-Waste Treatment & Minimization Committee
Program Organizers: Thomas P. Battle, DuPont Titanium Technologies, Wilmington, DE 19880-0352 USA; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA; Courtney A. Young, Montana Tech of the University of Montana, Metallurgical Engineering, Butte, MT 59701 USA

Tuesday PM Room: 2018
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Florian Kongoli, Flogen Technologies Inc., Matls. Tech. Dept., Montreal, Quebec H3S 2CS Canada; Arthur Morris, Thermart, San Diego, CA 92128 USA

TUESDAY PM

2:30 PM
Copper Distribution Between Reverb Furnace Smelting Products: *Natasha Mitevska*[1]; [1]RTB BOR, Copper Institute, Dept. of Metall., Zeleni bulevar 35, BOR 19210 Serbia

The results of industrial investigations of copper distribution between matte and slag, and in slag melt along the slag depth and length of the reverberatory furnace No.1 in RTB BOR, Copper Smelter and Refinery (Serbia) in 2004 are presented in this paper. Copper distribution coefficient between copper matte and slag phase is calculated, too. On the base of results obtained, the computer program for simulation of copper distribution in both melt phases in furnace is developed.

2:55 PM
Thermal Conductivity of Copper Flash Smelting Flue Dust: *Elli Vilhelmiina Nurminen*[1]; Laura Stykki[1]; Kim O. Fagerlund[2]; Pekka A. Taskinen[2]; [1]Helsinki University of Technology, Matls. Procg. & Powder Metall., PO Box 6200, Espoo 02015 Finland; [2]Outokumpu Research, PO Box 60, Pori 28101 Finland

Aggregations and build-ups in metallurgical heat recovery boilers decrease the efficiency of the heat transfer and may cause process shut downs. Transient plane source method (Hot Disk) has been applied to measure the thermal conductivities of the Copper Flash Smelting flue dusts. Thermal conductivities of copper, iron and zinc sulphates, and process flue dusts were measured. The results indicate that all the components are effective thermal insulators, having a thermal conductivity of less than 1 W/mK. Decreasing dust layer porosity has an increasing effect on thermal conductivity. Insulating layers on the boiler walls decrease greatly the heat transfer efficiency of the boiler. The gathered information can be used in the CFD-modelling of the process, and in process control.

3:20 PM
Kinetics of Copper Slag Reduction with Natural Gas: *Gabriel Riveros*[1]; Andrzej Warczok[1]; Roberto Parada[2]; [1]Universidad de Chile, Ingenieria de Minas, Av.Tupper 2069, Santiago Casilla 2777 Chile; [2]Anglo American Chile, Minera Sur Andes, Fundición Chagres, Chagres Chile

Smelting of copper concentrate in a flash smelter produces high grade matte and a slag containing from 2 to 3% Cu and 10-12% Fe3O4. The slag is processed in Teniente slag cleaning furnace by reduction with injected bunker oil, followed by slag sedimentation. The use of natural gas as a reductant is of great interest due to its price, easier management and benefits related to the decrease of negative environmental impact. The results of crucible scale simulation of slag reduction with injected air/natural gas mix showed as a major factor affecting the rate of magnetite reduction the air/natural gas ratio. The dependence of reaction rate on temperature is weaker in comparison to magnetite reduction from slag with solid carbon and carbon monoxide. Activation energy 100 kJ/mol and first order reaction regarding to magnetite content in the slag point out diffusional control in the gas towards gas/slag interface. Reaction thermal instability indicates the temperature of bubble/slag interface as a major factor determining the kinetic of magnetite reduction.

3:45 PM
Copper Flash Smelting Mass Balance Calculations Using Genetic Algorithms: *Victor Manuel Sanchez-Corrales*[1]; Jose Adolfo Valera-Gonzalez[1]; Pedro Flores-Perez[2]; Manuel Perez-Tello[1]; [1]Universidad de Sonora, Ingeniería Química y Metalurgia, Blvd. Luis Encinas y Rosales, Colonia Centro, Hermosillo, Sonora 83000 Mexico; [2]Universidad de Sonora, Dept. de Matematicas, Blvd. Luis Encinas y Rosales, Colonia Centro, Hermosillo, Sonora 83000 Mexico

In this paper, mass balance calculations using genetic algorithms for copper smelting in an Outokumpu flash furnace are presented. Based on the elemental composition of the copper concentrates being fed to the reactor, the mineralogical composition of the concentrate mixture is adjusted by means of genetic algorithms. The macroscopic mass balance equations for the species entering and leaving the furnace are solved and the compositions and flow rates of matte, slag, and the off-gas stream are computed. Good agreement between theoretical predictions and plant data was obtained in terms of matte and slag flow rates, matte grade, and copper, iron, magnetite, and silica contents in the slag. Predictions of the present method showed to be superior to those of a conventional one in which the mineralogical composition of the feed is not adjusted. Future applications of the present formulation are discussed.

4:10 PM Break

4:25 PM
Separation of Copper and Cobalt from Nickel Sulphate Solution by Organophosphorus Mixied Extractant: Chen Song[1]; Luo Yuan Hui[1]; Wang Li Jun[1]; Zhang Li[1]; Wang Rui Zhong[1]; [1]General Research Institute for Non-Ferrous Metals, Minl. Resources, Metall. & Matls., No.2 XinJie Kou Wai Da Jie St., Beijing 100088 China

During separation process of copper and cobalt from nickel sulphate solution, the effects of constitutions and compositions of mixted extractant D2EHPA and PC88-A or Cyanex272, has been studied. In one step extraction cycle, the following results are obtained:[Ni]/[Co]>4000 and [Ni]/[Cu]>40000 in nickel sulphate solution, [Co]/[Ni]>1000 and [Co]/[Cu]>30000 in cobalt chlorinate solution, [Cu]/[Ni]>10000 and [Cu]/[Co]>200 in copper sulphate solution. It was elucidated that the above-mentioned synergistic mixtures can effectively separate cobalt and copper from nickel sulphate acid solution and achieve a greater synergistic effect.

4:50 PM
Electrolysis and Reduction of Copper Slag: *Andrzej Warczok*[1]; Gabriel Riveros[1]; Martin Artigas[1]; [1]Universidad de Chile, Ingenieria de Minas, Av.Tupper 2069, Santiago Casilla 2777 Chile

Fayalite slags from smelting of copper concentrate contain from 2 to 25% of copper and from 8 to 25% of magnetite depending on the smelting process. Pyrometallurgical slag cleaning based on its reduction and sedimentation is carried out in an electric furnace or various furnaces with injection of carboneous reductant. Since common acceptance of ionic structure of liquid slags the possibilities of utilization of electrochemical phenomena in copper recovery are the point of interest. Slag electrolysis combined with chemical reduction together with electrokinetics phenomena may accelerate copper removal during slag cleaning. Results of crucible scale tests of slag electrolysis with inert and graphite electrodes showed relatively high current efficiency of copper and significant acceleration of copper removal from the slag.

Friction Stir Welding and Processing III: Process/Applications

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Lienert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Tuesday PM Room: Nob Hill C/D
February 15, 2005 Location: San Francisco Marriott

Session Chair: Julie A. Christodoulou, Office of Naval Research, Arlington, VA 22217-5660 USA

2:00 PM Keynote
Friction Stir Welding - A Brief Review and Perspective for the Future: *Tracy W. Nelson*[1]; [1]Brigham Young University, Mechl. Engrg., 435 CTB, Provo, UT 84663 USA

Friction stir welding (FSW), a tremendous development in mechanical working of metals, is a proven technology for joining aluminum and other lower temperature metals. Application in aluminum and copper are growing while FSW in steels and other higher temperature

materials are nearing implementation status due to the advent of new tool materials and process understanding. In addition, Friction Stir Processing (FSP) has been gaining interest over the past few years in a variety of commercial applications. With such promising benefits, the technology of FSW&P has consumed much of the welding R&D community in just over a decade. Despite the progress in process development and technology application, the drive to implementation has left behind the research necessary for understanding essential process fundamentals. Fundamental research, in parallel with technology development, must be undertaken in order to achieve broad application of FSW&P. A researcher's perspective regarding what research and development activities are needed to evolve the technology for future applications will be given.

2:30 PM Invited
Process Dynamics of Friction Stir Welding (FSW) of 2024 Al: *J. A. Baumann*[1]; R. J. Lederich[1]; W. C. Starnes[1]; [1]The Boeing Company, PO Box 516, MC: S245-1003, St. Louis, MO 63166-0516 USA

For many FSW applications, a relatively simple delineation of the appropriate weld operating window in terms of travel rate and spindle speeds ("feeds and speeds") can be sufficient for successfully making suitable welded components. In some envisioned applications, however, such as closing-out structural components by welding skins to substructure, that substructure must react or carry the loads generated by the FSW process. In this scenario, a fuller understanding needs to be developed between the relationships of tool features, travel rate, rotation speeds and required or resultant process loads and torques, in order to properly design the substructure to withstand these forces during the welding operation, or, conversely, to tailor the FSW process to a specific structural architecture. We have explored these relationships in the lap joining of 2024 Al sheets, with top-sheet thickness ranging from 3.2 mm (0.125") thick to 9.5 mm (0.375"), using both standard, one-piece (solid shoulder and pin) FSW tools and two-piece, Retractable Pin Tools (RPT). This work has enabled us to evaluate and separate the contributions to process loads and torques arising from the pin, turning within the aluminum, and that of the shoulder, engaged with the surface of the material.

2:50 PM
Features and Configurations of FSW Equipment: *Donald J. Holman*[1]; [1]Nova-Tech Engineering, 6808 220th St. SW, Ste. 200, Mountlake Terrace, WA 98043-2187 USA

The FSW process has researched for several years. Most of the research has been dedicated to optimizing welding parameters to facilitate improved properties compared to conventional welding techniques. Extensive publications and presentations have been made regarding the material science and application benefits of friction stir welding. As the process becomes better defined, the criteria for FSW equipment design is becoming evident, yet many people are un-familiar of what constitutes a FSW machine. This presentation will outline the major systems of a FSW machine, and how they are unique compared to conventional milling machines.

3:10 PM
Friction Stir Welding of Castings: *Richard Johnson*[1]; Philip L. Threadgill[2]; William J. Kyffin[1]; [1]TWI Technology Centre (Yorkshire) Ltd, PO Box 3314, Sheffield S13 9WZ UK; [2]TWI Ltd, Friction & Forge Processes Tech. Grp., Granta Park, Great Abington, Cambridge, Cambridgeshire CB1 6AL UK

There has been considerable interest in the application of friction stir welding processes to castings in many materials, as a method of joining to other materials (cast or wrought), for repair of local defects, and for re-processing, (where the microstructure can be converted to a forged microstructure without a significant change in shape). This paper will summarise recent progress made in friction stir welding/processing of a range of aluminium alloys, copper alloys and steels, and will give examples of where these techniques could be used commercially. The paper will include process information and microstructural assessments of the welded areas.

3:30 PM Break

3:50 PM Invited
Unraveling the Material Processing Conditions for Optimizing the FSW Process: *Judy A. Schneider*[1]; Arthur C. Nunes[2]; [1]Mississippi State University, Mechl. Engrg. Dept., PO Box ME, 210 Carpenter Engrg. Bldg., Mississippi State, MS 39762 USA; [2]NASA-Marshall Space Flight Center, Matls., Processes & Mfg. of Metallic Matls., ED33, Huntsville, AL 35812 USA

In friction stir welding (FSW), a rotating threaded pin tool is inserted into a weld seam and literally stirs the edges of the seam together. This environmentally friendly, solid-state technique has been successfully used in the joining of materials that are difficult to fusion weld. To determine optimal processing parameters for producing a defect free weld, a better understanding of the resulting metal deformation flow path and velocity is required. In this study the metal flow fields are marked by the use of thin (0.001 tungsten) wires embedded in the weld seam at various locations. X-ray radiographs record the position and segmentation of the wire and are used to elucidate the flow field. Microstructures observed in a FSW cross-section in an aluminum alloy are related to their respective strain-strain rate-temperature histories along their respective flow trajectories. Two kinds of trajectories, each subjecting the weld metal to a distinct thermomechanical process and imparting a distinct microstructure, can be differentiated within the weld structure.

4:10 PM
Determining Optimum Friction Stir Weld Process Variables to Minimize Abnormal Grain Growth in Al-2195: *Srikanth Labhala*[1]; Stanley M. Howard[1]; William J. Arbegast[2]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Engrg., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakotra School of Mines and Technology, Advd. Matls. Procg. Ctr., Rapid City, SD 57701 USA

The objective of this work was to investigate the inhibition of abnormal grain growth (AGG) in friction stir welded (FSW) Al-2195. The weld process parameters of weld speed, rotation speed, and pin tool geometry were varied. Additionally, cryogenic quenching with liquid nitrogen during FSW was applied in an attempt to inhibit AGG. Liquid nitrogen was directed closely behind the pin tool on the retreating side of the weld. The test matrix contains 48 six-inch welds and includes the use of three different pin tools. Post FSW AGG was initiated by heating and holding the samples at 950°F for five minutes. The results are analyzed using plots of the extrusion ratio of the weld nugget with respect to grain size. The relationship between the AGG and the extrusion ratio of welds is summarized. Optimum processes parameters and weld conditions to inhibit AGG are suggested.

4:30 PM
FSW of Aluminum Using a Tapered and Scrolled Shoulder Tool: *Kevin J. Colligan*[1]; [1]Concurrent Technologies Corporation, Harvest, AL USA

Since FSW was originally invented by The Welding Institute advancements in welding tool design have allowed the process to be operated at higher travel speeds, in thicker sections, and with more reliable quality. A new feature has been developed, referred to as the tapered shoulder, which allows FSW to be operated more easily and with more simple equipment. The tapered shoulder consists of a scrolled shoulder that is not generally flat but is tapered outward from the outside diameter to the pin. This scrolled cone can be embedded beneath the surface of the plate to any depth, with the width of the crown of the weld being variable depending on the depth of penetration. This patented tool design has implications for conventional welds, for welds in material with complex curvature and in pipe, and for self-reacting welds using tools that have a fixed geometry. This paper describes the welding tool design and its use in different welding applications.

4:50 PM
Surface Friction Welding – A New Process for Butt Welding of Thin Metal Sheets: *Sung-Joon Kim*[1]; Heung Nam Han[1]; Chang Gil Lee[1]; Sang-Sik Kim[2]; [1]Korea Institute of Machinery & Materials, 66 Sangnam, Changwon 641-010 Korea; [2]Gyeongsang National University, Jinju 660-701 Korea

A novel process for butt welding of metal sheets thinner than 2.0 mm was invented and patented by the authors. The process was named as surface friction welding (SFW) which utilizes friction heat and plastic deformation like friction stir welding (FSW). The SFW was successfully applied to butt welding of 6061Al sheets of 1.5 mm thick and Mg alloy sheet of 1.0 mm thick. This paper will introduce the principle of SFW, the difference between FSW and SFW, the effect of welding parameters, and the microstructure and mechanical properties of welded sheets.

5:10 PM
Comparison of Self-Reacting and Standard Fixed Pin Friction Stir Welding for Fabrication of Aluminum Box Beams: *Alex Paul Toskey*[1]; William J. Arbegast[1]; Anil K. Patnaik[1]; Casey A. Allen[1]; [1]South Dakota School of Mines and Technology, Advd. Matls. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD 57701 USA

Work has been done to investigate friction stir welding as a cost effective alternative to extruded hollow aluminum box beams. The proposed design is to extrude two C-Channels and join them along the length by FSW. Both self-reacting and standard fixed pin tools have

been evaluated as methods of joining. The effects of different pin tool geometries will be presented as well as tooling setups for each type. Metallurgy and mechanical assessment of resulting welds will be compared along with process forces and thermal data. In the end, tooling limitations and issues with long self reacting pin tool use in closed sections resulted in the standard fixed pin tool being chosen for the final design fabrication approach.

5:30 PM
Experimental Measurements of Longitudinal Load Distributions on Friction Stir Weld Pin Tools: *Aaron L. Stahl*[1]; Carl D. Sorensen[1]; [1]Brigham Young University, Mechl. Engrg., 435 CTB, Provo, UT 84602 USA

An understanding of the forces acting on the pin of FSW tools is critical to appropriate design, especially in materials with limited toughness like PCBN. This paper describes a study to measure the longitudinal force distribution on a friction stir weld pin tool. Total longitudinal forces were recorded on a dynamometer while welding 6061 aluminum with pins that varied in length and diameter. A model was developed that characterizes pin force as a function of pin length and diameter. As the pin length approaches zero, the longitudinal force reaches an asymptote, which is apparently the longitudinal force due to the shoulder. The force due to the pin increases with pin length, but varies insignificantly with pin diameter. The force distribution on the pin appears to increase linearly with distance from the shoulder. Unexpected force variation was found at large pin lengths, a result which has yet to be explained.

TUESDAY PM

Frontiers in Solidification Science: Crystal-Melt Interfaces: Fundamental Properties and Related Behavior

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Solidification Committee
Program Organizers: Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; James R. Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Tuesday PM Room: 2020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Mike I. Baskes, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Martin E. Glicksman, Rensselaer Polytechnic Institute, Troy, NY 12180 USA

2:00 PM Invited
Atomistic Simulations of Solid-Liquid Interface Mobility in FCC and BCC Metals: *J. J. Hoyt*[1]; M. D. Asta[2]; D. Y. Sun[3]; A. Karma[4]; [1]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185 USA; [2]Northwestern University, Dept. of Matls Sci. & Engrg., Evanston, IL 60208 USA; [3]East China Normal University, Physics Dept., Shanghai China; [4]Northeastern University, Physics Dept., Boston, MA 02115 USA

The crystal-melt interface mobility plays an important role in controlling the morphology and growth kinetics of dendrites. The mobility is typically characterized by the kinetic coefficient, μ, defined as the constant of proportionality between the interface undercooling and the solidification velocity. Using molecular dynamics simulations and interatomic potentials of the embedded atom form, we have computed the kinetic coefficient in several FCC and BCC pure metals. With the exception of BCC metals growing along [100], the crystallization rates and their dependence on crystallographic growth direction are well described by a density functional theory based model due to Mikheev and Chernov. The discrepancy in the BCC case stems from an anomalously large solid-liquid interface width observed for the [100] orientation. In addition, we have computed the kinetic coefficient in a binary alloy, namely the B2 ordered NiAl system. For NiAl a low value of μ and a pronounced asymmetry between solidification and melting rates is observed.

2:35 PM Invited
Crystal-Melt Interfacial Free Energies in Metals: Role of Crystal Structure: *Mark D. Asta*[1]; Jeffrey J. Hoyt[2]; Deyan Sun[3]; [1]Northwestern University, Dept. of Matls. Sci. & Engrg., Evanston, IL 60208 USA; [2]Sandia National Laboratories, Albuquerque, NM USA; [3]East China Normal University, Dept. of Physics, Shanghai China

The magnitudes and crystalline anisotropies of crystal-melt interfacial free energy have been calculated for a variety of metal and alloy systems, based upon molecular-dynamics simulations employing embedded-atom potentials. Results for fcc Ag, Al, Au, Cu, Fe, Ni and Pb are compared with recent calculations for bcc Fe, Mo, V and hcp Mg. Calculated interfacial free energies are found to be well described by Turnbull's empirical scaling relation, with Turnbull coefficients that are roughly 30 percent lower for the bcc metals than for fcc and hcp. A trend towards lower anisotropies for bcc relative to fcc metals is observed. Results for the ordered bcc-based B2 NiAl compound show Turnbull coefficients and anisotropies consistent with those for the elemental bcc metals. Overall these results suggest that the underlying crystal lattice structure plays a primary role in governing crystal-melt interfacial free energies in metals.

3:10 PM Invited
Calculation of Crystal-Melt Interfacial Free Energies by Atomistic Simulation: Dependence Upon Crystal Structure and Interatomic Forces: *Brian B. Laird*[1]; Ruslan L. Davidchack[2]; [1]University of Kansas, Chmst., 1251 Wescoe Hall Dr., Lawrence, KS 66045 USA; [2]University of Leicester, Math., Univ. Rd., Leicester LE1 7RH UK

We review our recent work on the direct calculation, via MD computer simulation, of the interfacial free energy, γ, of the crystal-melt interface for a number of model systems. The value of γ as a function of crystal orientation is determined using a thermodynamic integration technique employing moving cleaving walls [Phys. Rev. Lett., 85, 4751 (2000)]. The calculation is sufficiently accurate to at least partially resolve the small anisotropy in γ, which is important input for continuum simulation of dendritic growth. We report values of γ for the hard-sphere and Lennard-Jones systems, as well as recent results on the series of repulsive inverse-power potentials. For the inverse sixth and inverse-eighth power systems, we determine γ for both fcc and bcc crystal structures.For these systems, the bcc-melt γ is lower that that of fcc by about 30%, in qualitative agreement with recent simulations on iron by Asta, Hoyt and Karma.

3:45 PM Break

3:55 PM Invited
Modeling Interaction of Dendritic Interfaces with Biological Cells: Anthony Chang[1]; *Jonathan A. Dantzig*[1]; [1]University of Illinois, Mechl. & Industl. Engrg., MC-244, 1206 W. Green St., Urbana, IL 61801 USA

Cryopreservation involves solidification of a water-based solution and incorporation of biological cells. A successful protocol ensures that the cells are not damaged during freezing by any of several possible factors, including excessive solute concentration, intracellular ice formation and mechanical damage. The solidifying interface is almost always dendritic. In this work, we present models for directional solidification (DS) of aqueous solutions containing cells. We use a level set method to model the solidification, and include direct calculation of the interaction forces between the cells and the moving interface. We examine the role of particle size, and location with repsect to the evolving interface structure, on capture and pushing ahead of the interface.

4:30 PM
In-Situ Synchrotron Microtomography Study of Morphology Evolution and Microporosity Formation in Solidifying AlSiCu-Alloys: Dominique Bernard[2]; Marco Di Michiel[3]; *Øyvind Nielsen*[1]; Luc Salvo[4]; [1]SINTEF, Matls. & Chmst., PB 124 Blindern, Oslo 0314 Norway; [2]ICMCB-CNRS, 33608 Pessac, Cedex France; [3]ESRF, Polygone Scientifique Louis Néel, 6 rue Jules Horowitz, Grenoble 38000 France; [4]GPM2-INPG-CNRS, Saint Martin d'Heres 38402 France

Direct observation of the microstructure evolution in solidifying metal alloys is still a great challenge in solidification science, which is manifested by the extensive literature on quenching experiments and transparent analogues. Although recent progress has been made in time resolved, high resolution x-ray imaging of thin metal samples during solidification (2D), the extension to microtomography (3D) has not yet been made, due to limitations in e.g., the x-ray beam intensity and characteristics, and the data acquisition and transfer speed. In the present work, recent results are shown from a study aiming at time resolved, high resolution 3D x-ray-imaging using microtomography at the European Synchrotron Radiation Facility (ESRF). Cylindrical samples (D=1.2 mm, H=1.2 mm) of Al-9 wt.% Si-3.5 wt.% Cu alloys were mounted in the microtomography setup at ESRF Beamline ID15A, melted completely by the use of a small furnace, and then cooled at a constant rate of 0.1°C/s. During solidification, about 40 tomograms were acquired for each sample. The spatial resolution is 2.8 μm and the data acquisition time for each tomogram (400 images) is 13 s. Due to

the segregation of copper into the liquid phase, solid/liquid absorption contrast is achieved and the 3D evolution of the solid/liquid interface was observed and quantified. Moreover, the nucleation and growth of gas and shrinkage porosity were observed.

4:50 PM

Containerless Solidification of Undercooled Eutectic Melts: *Mingjun Li*[1]; Yasutomo Arai[1]; Jianding Yu[1]; Takehiko Ishikawa[1]; Shinichi Yoda[1]; Kosuke Nagashio[2]; Kazuhiko Kuribayashi[2]; [1]Japan Aerospace Exploration Agency, Inst. of Space & Astronautical Sci., Tsukuba Space Ctr., 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505 Japan; [2]Japan Aerospace Exploration Agency, Inst. of Space & Astronautical Sci., Sagamihara Campus, 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan

We employed an electromagnetic levitator to investigate Ni-Sn, Ni-Si, Co-Sn, Co-Sb, and Co-Ge eutectics at various undercoolings. Microstructural observation revealed that individual eutectic colonies distribute not only at sample surface, but also throughout the volume of an entire sample. Similar microstructural characteristics were found in binary Al2O3-ZrO2, Fe2O3-La2O3, Yb2O3-Al2O3, Al2O3-MgO, Yb2O3-SiO2, Sm2O3-SiO2, and Y2O3-SiO2 oxide eutectics solidified on an aero-acoustic levitator. The latest microstructure on Al2O3-ZrO2-Y2O3 eutectics consisted of independent eutectic colonies, indicating that massive or copious nucleation occurs in free solidification of these poly-domain systems, either binary metallic or binary and ternary oxide eutectics. The eutectic growth model is modified to incorporate kinetic undercooling for competing solid solution and compound; collision-limited growth generates a highly mobile interface compared with that of diffusion-limited growth and thus the solid solution overgrows the compound to yield anomalous eutectic. This model can interpret most published data on the formation of anomalous eutectics.

5:10 PM

Microstructure Evolution on Shock Melt and Refreeze in Bismuth: *Bryan W. Reed*[1]; Jeff Colvin[1]; Alan Jankowski[1]; Mukul Kumar[1]; Dennis Paisley[2]; Damian Swift[2]; Tom Tierney[2]; [1]Lawrence Livermore National Laboratory, Chmst. & Matls. Sci., PO Box 808, L-370, Livermore, CA 94551 USA; [2]Los Alamos National Laboratory, Plasma Physics, P-24, MS E526, PO Box 1663, Los Alamos, NM 87545 USA

Because solid bismuth is less dense than the liquid, pressurized liquid bismuth should begin to freeze immediately upon release of the pressure, yielding extremely fast initial undercooling rates (~10^{10} K/s upon initial release of a shock wave, according to a single-phase model). Using the technique of tamped ablation (Colvin et al., Phys. Plasmas 10/7, 2940, 2003), we used long-pulse lasers to shock-melt preheated Bi in order to induce a rapid melt/refreeze cycle. The recovered material has a mix of microstructures including fine dendrites indicating rapid resolidification. We interpret the shocked microstructures in light of theoretical predictions, real-time shock diagnostics (resistivity and free-surface velocity), and controlled variations of the pre-shock microstructure. The results carry implications for the study of solid-liquid phase changes on very short time scales. This work was performed under the auspices of the U.S. Department of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

Frontiers in Solidification Science: Poster Session

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Solidification Committee

Program Organizers: Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; James R. Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Tuesday, 5:30-7:30pm Room: 2020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Shan Liu, Ames Laboratory, Ames, IA 50011 USA; Ralph Napolitano, Iowa State University, Ames, IA 50011 USA; James Morris, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

Convective Effects on Thermosolutal Free Dendritic Growth: *Juan C. Ramirez*[1]; Christoph Beckermann[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., MST-8, PO Box 1663, MS G755, Los Alamos, NM 87545 USA; [2]University of Iowa, Dept. of Mechl. & Industl. Engrg., 2412 Seamans Ctr., Iowa City, IA 52242-1527 USA

Two-dimensional phase field simulations are performed to examine the effects of melt convection on the dendrite tip operating state of dilute binary alloys. The model employed reproduces the usual sharp interface conditions in the limit of a thin, diffuse interface region. Detailed knowledge of thermal, solutal and velocity fields around the dendrite tip from simulations provide great insight into the understanding of convective effects on dendritic growth. Depending on the relative importance of thermal and solutal effects, three regimes are considered: purely thermal, purely solutal and thermosolutal solidification. The simulation results are compared to theoretical models.

Mechanisms of Primary Silicon Growth in Near-Eutectic Al-Si Alloys: *Ralph E. Napolitano*[1]; Choonho Jung[1]; [1]Iowa State University, Matls. Sci. & Engrg., Ames Lab., Matls. & Engrg. Physics, Ames, IA 50011 USA

Morphological evolution and selection of angular primary silicon is investigated in near-eutectic Al-Si alloys. Angular silicon "dendritic" arrays are grown directionally in a Bridgman furnace at velocities in the regime of 10-3 m/sec. Serial milling and x-ray techniques are combined with backscattered electron diffraction analysis to examine the early-stage selection dynamics and primary array evolution through various tip-splitting and branching mechanisms. The detailed structure of the twinned bicrystal dendritic cores and the role of twinning in the mechanisms of branching and spacing adjustment are discussed. Compositions from 10 wt% Si to 14 wt% Si are investigated, and the transition from primary Si growth to coupled eutectic growth is examined.

Convectional Effect on Morphology and Segregation of Directionally Solidified Ductile Irons: *Wen Shiung Chang*[1]; Tien Shou Lei[1]; [1]National Taiwan University of Science and Technology, Dept. of Mechl. Engrg., #43, Sect. 4, Keelung Rd., Taipei 106 Taiwan

Convection effect plays an important role in the final microstructures and chemical segregations during the solidification of castings. In this paper the effect of fluid convection on morphology and segregation of directionally solidified ductile irons will be presented. A vertical cylindrical mold cavity set-up which consists by two furan molds sandwiched with a water chilled copper mold in the middle of the furans was used for studying that provides three distinct solidifying zones: an upward, a bidirectional and a downward. In the downward portion, it was observed that a number of larger nodular graphite near the chilled mold which solidified first, but some smaller nodular graphite existed at the bottom which was last solidified. These phenomena are different from that of casting which solidified in upward direction. The mechanism of elements redistribution such as molybdenum and chromium, analyzed by EPMA, in two founded alloy-rich carbides will also be discussed.

The Effect of Seed Dendrite Arm Spacing on Single Crystal Ni-Base Superalloys: *Elyssa Renee Cutler*[1]; Gerhard E. Fuchs[1]; [1]PO Box 116400, Gainesville, FL 32611 USA

Recent cellular automaton-finite difference (CA-FD) models have predicted a range of stable dendrite arm spacings for single crystal binary castings according to thermal history. An attempt to verify this model by changing seed crystal dendrite arm spacing within a single casting was made with samples of a single crystal Ni-base superalloy, CMSX-4. Three widely varying seed dendrite arm spacings were chosen both inside and out of predicted stable ranges. Both bars and blade shapes were cast using standard industry parameters. Each casting was sectioned to determine the time required to reach steady state and to characterize the dendrite morphology and the origin of any casting defects.

Examining the Benefits of Liquid Metal Cooling (LMC) for Directional Solidification of Large Ni-Base Superalloy Castings: *Andrew J. Elliott*[1]; Tresa M. Pollock[1]; Michael F.X. Gigliotti[2]; [1]University of Michigan, Matls. Sci. & Engrg., 2300 Hayward St., Ann Arbor, MI 48109 USA; [2]General Electric, Global Rsch., Niskayuna, NY 12309 USA

A series of experiments investigating liquid-tin assisted directional solidification (DS) of large Ni-base superalloy castings has been conducted. Compared to the conventional Bridgman (radiation cooling) process, liquid metal cooling (LMC) resulted in considerably enhanced cooling rates, refined microstructure, and reduced occurrence of casting defects, including elimination of freckle-type defects. Additionally, withdrawal rates at least two to three times those capable with the conventional process were achieved while maintaining an aligned crystal structure free from casting defects. Preliminary mechanical testing of specimens made using both the conventional and LMC processes was conducted to examine any possible mechanical benefit of the LMC

process. The effect of various LMC process parameters were also investigated through experiments and thermal modeling. The experimental casting results, microstructural analysis, mechanical comparison, and thermal modeling are examined to provide a broader understanding of how the LMC process is beneficial for producing large DS castings.

Multi-Scale Modeling of Particle-Solidification Front Dynamics: *Justin Wayne Garvin*[1]; Yi Yang[1]; Holavanahalli S. Udaykumar[1]; [1]University of Iowa, Dept. of Mechl. Engrg., 3131 Seamans Ctr., Iowa City, IA 52242 USA

The interaction between a micron-sized particle and a solidification front is inherently a multi-scale problem. For particles of microns in size or greater, the solution of the Navier-Stokes equations in the particle-front system including resolution of the nano-scale gap between surfaces would be impossible due to the fine mesh requirement needed. A lubrication model is therefore developed for the interaction between any two immersed solid surfaces moving relative to one another at arbitrary velocities. The model includes the disjoining pressure effects due to intermolecular forces as well as liquid-solid phase change at one or both of the interfaces. The solution to the lubrication model is coupled to the solution outside the gap that is solved using the Navier-Stokes equations. The interfaces are tracked using a level-set method. Results are obtained for the interaction between a particle and a solidification front and are compared to previous theoretical results for the interaction.

Impurity-Induced Instabilities: Layachi Hadji[1]; [1]University of Alabama, Dept. of Math., 345 Gordon Palmer Hall, Tuscaloosa, AL 35487-0350 USA

We examine the interaction of a minute foreign impurity with a directionally solidified interface. We show that the interaction induces the onset of morphological instability provided that the distance between the impurity and the interface falls below a critical value. This instability occurs at pulling speeds that are below the threshold for the onset of the Mullins-Sekerka instability. The expression for the critical distance reveals that this instability is manifested only for certain combination of the physical and processing parameters, and its occurence is attributed to the reversal of the thermal gradient in the melt between the impurity and the solid-liquid interface. Another instability may arise when the impurity is in near-contact with the interface. The disjoining pressure affects the interaction. We derive an expression for the film thickness at which rupture occurs. The influence of these instabilities on the capture or rejection of the impurity is discussed.

The Effects of Internal Convection on Dendritic Evolution in Stainless Steel Alloys: *Alaina B. Hanlon*[1]; Robert W. Hyers[1]; Douglas M. Matson[2]; [1]University of Massachusetts, Dept. of Mechl. & Industl. Engrg., Engrg. Lab Bldg., 160 Governors Dr., Amherst, MA 01003 USA; [2]Tufts University, Dept. of Mechl. Engrg., Rm. 025 Anderson Hall, 200 College Ave., Medford, MA 02155 USA

In certain compositions, Fe-Cr-Ni stainless steels solidify from an undercooled melt by a 2-step process in which the metastable ferrite phase transforms to a stable austenite phase. Recent experiments have shown that flow within the molten sample strongly influences the lifetime of the metastable phase. The current research will provide insight to why flow affects the metastable phase, and lead to the use of convection to control microstructural evolution. If the convective velocities are great enough to cause the dendrites to bend then low angle boundaries form at the points of collision. These result in high energy sites that could serve as nuclei for the stable phase. Numerical models are employed to evaluate interactions between fluid and microstructures during rapid solidification. Simulations yield a range of convective flow velocities that causes mechanical damage to the dendrites and can be compared to experimental results.

Phase-Field Model for Alloys with Arbitrary Phase Diagrams: Seong Gyoon Kim[1]; *Won Tae Kim*[2]; [1]Kunsan National University, Dept. Matls. Sci. & Engrg., 68 Miryong Dong, Kunsan 573-701 Korea; [2]Cheongju University, Applied Sci. Div., 36 Naedok Dong, Cheongju 360-764 Korea

Remarkable progress in phase-field simulations of alloy solidification has been made by using the thin-interface analysis and introducing an anti-trapping current into the standard phase-field model for alloys. This make us to overcome the stringent restriction on the interface width and enhance both the computational efficiency and accuracy. In this study, we extend the phase-field model with the anti-trapping current to alloy systems with arbitrary phase diagrams and find the mapping conditions of the phase-field model onto the classical moving boundary problem. A few simulations for testing the self-consistency will be shown and compared with the previous model of localized solute partitioning in diffuse interface.

Anisotropic Free Energies of Crystal-Melt Interfaces: *James R. Morris*[1]; Ruslan L. Davidchack[2]; Mikhail I. Mendelev[3]; David J. Srolovitz[4]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6115 USA; [2]University of Leicester, Dept. of Math., Leicester LE1 7RH UK; [3]Iowa State University, Ames Lab., Ames, IA 50011 USA; [4]Princeton University, Dept. of Mechl. & Aeros. Engrg., Princeton, NJ 08544 USA

Recently, there have been significant advances in approaches to calculating the free energy of crystal-melt interfaces from simulations. Current approaches can not only predict accurate values, but can resolve the anisotropies in the values on the order of 1-2%. We demonstrate these approaches, in particular the fluctuation approach applicable to rough interfaces, and compare the results. For the Lennard-Jones system, the results are in very close agreement, whereas the hard-sphere results give smaller values than in previous calculations. For more realistic models of Al, we show that different potentials have very different values, which are correlated with the liquid structure. We also include recent results for purely repulsive potentials, of the form $1/r^n$. This research has been sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract DE-AC05-00OR-22725 with UT-Battelle.

Microstructure Evaluation and Prediction During the Investment Casting of Ti-6Al-4V Plates: *Laurentiu Nastac*[1]; Kevin Klug[2]; Mehmet N. Gungor[2]; Troy Tack[2]; [1]Concurrent Technologies Corporation, 425 Sixth Ave., Regional Enterprise Tower, 28th Fl., Pittsburgh, PA 15219 USA; [2]Concurrent Technologies Corporation, Johnstown, PA 15904 USA

This paper describes the modeling and the experimental efforts for the evaluation and prediction of the microstructure and its relationship with the mechanical properties in investment casting of Ti-6Al-4V (Ti-6-4) alloy. This work supports the M777 Lightweight Howitzer (LWH) program, where investment casting was selected over machining and welding titanium plate to reduce part count and associated manufacturing expense for several LWH components. Key predictive capabilities such as shrinkage, alpha-case thickness, and microstructure were simulated. The purpose of the work was to better understand the investment casting process to eliminate/minimize defects in LWH components and provide useful insights to meet aggressive schedule requirements by minimizing experimental production trials. The modeling approach was based on the numerical solution of fluid flow and heat transfer equations and mesoscopic modeling of the microstructure. The effects of casting parameters on the plate microstructures were studied. The amount of alpha and beta phases as a function of cooling rates was experimentally measured. A relationship between the microstructure and mechanical properties was established. The influence of composition variance on both the microstructure and mechanical properties was investigated. Evaluation results showed that the HIP and heat treated Ti-6-4 plates have acceptable mechanical properties and microstructure with minimum grain coarsening and porosity. Acknowledgment. This work was conducted by the National Center for Excellence in Metalworking Technology (NCEMT), operated by Concurrent Technologies Corporation under contract No. N00014-00-C-0544 to the U.S. Navy as part of the U.S. Navy Manufacturing Technology Program. Approved for public; distribution is unlimited.

Effects of Solidification-Induced Segregation on Alloy 22 Phase Stability: *Yi-Ming Pan*[1]; Darrell S. Dunn[1]; Gustavo A. Cragnolino[1]; [1]CNWRA, Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78238 USA

Simulations have been conducted to determine the phase stability of Alloy 22 affected by elemental segregation in the solidified weld microstructure. Compositional analyses of gas-tungsten arc welded Alloy 22 using energy-dispersive x-ray spectroscopy revealed that the interdendritic regions are enriched in Mo and depleted in Ni relative to the dendrite cores. Both equilibrium and Scheil solidification simulations were performed with the Thermo-Calc Version N software and the Ni-DATA Version 5 database. The solidus, liquidus, and P-solvus temperatures were calculated for the bulk alloy composition, as well as for the measured compositions for the dendrite cores and the interdendritic regions. The equilibrium simulations predict complete solidification of liquid to γ-phase whereas the Scheil simulations predicts the formation of P-phase (the predominant topologically close-packed phase of Alloy 22) near the end of solidification. In addition, the Scheil simulation predicts a high solvus temperature for the P-phase in the interdendritic regions due to element partitioning in the

solidified weld microstructure. The implications of these results for the solution annealing treatment of Alloy 22 weldments are discussed. Acknowledgment: This paper is an independent product of the CNWRA and does not necessarily reflect the views or regulatory position of the NRC.

Phase Field Modeling of Crystal Nucleation in 3D: *Tamas Pusztai*[1]; Laszlo Granasy[1]; [1]Research Institute for Solid State Physics and Optics, POB 49, Budapest H-1121 Hungary

Homogeneous and heterogeneous nucleation is studied in the framework of the phase field theory. The properties of critical fluctuations is determined by solving the Euler-Lagrange equations. The nucleation rate is evaluated from simulations performed with Langevin-noise. The magnitude of the nucleation prefactor is estimated from simulations and compared with theoretical predictions. Sharp walls defined via the "no-flux" boundary condition are introduced into the simulations. Heterogeneous nucleation on foreign particles, rough walls, and solidification in porous media are addressed.

Multiscale Modeling of Solidification Processes: *Yan Shu*[1]; Xin Ai[1]; Ben Q. Li[1]; [1]Washington State University, Mechl. Engrg. Dept., Pullman, WA 99163 USA

Solidification processing involves multiple length and time scales. This paper discusses a multiscale computational scheme for the modeling of the microstructure development during solidification processing. The molecular simulation, phase field modeling and continuum-based fluid flow and heat transfer phenomena are integrated to cover eventually from the molecular scale all the way to the continuum domain. The modeling strategies that involve both mutual and one-way coupling of molecular, micro and macroscale phenomena are discussed. Computational results are given for some solidification systems.

A Robust and Efficient Equiaxed Grain Growth Model and Coupling To a Macro Heat Transfer Algorithm: *Andrei Starobin*[1]; David Torres[2]; Marius Stan[3]; [1]Metal Casting Simulation Consultant, 211 Maynard Dr., Santa Fe, NM 87501 USA; [2]Los Alamos National Laboratory , T-3, Los Alamos, NM 87545; [3]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545

A robust and efficient equiaxed growth model is developed. It is a variant of that suggested by Beckermann et al (1993) modified to a 2-ODE form to allow for an exactly conservative in enthalpy meso-macro coupling. A further modification is a full, fast, numerical treatment of the solute diffusion in the shrinking extradendritic region based on the work of Torres et. al. (2004). This numerical approach allows uniform treatment of growth: from just after nucleation through coalescence. A fully iterative meso-macro coupling poses significant challenges for a macroscopic enthalpy algorithm in the recalescence region where the cooling curves are highly oscillatory. These numerical difficulties are addressed and a coupling with finite-volume, enthalpy based, Jacobian matrix-free heat transfer algorithm of Knoll et al (1999) is demonstrated. Comparison is made with a faster meso-macro passive 1-way coupling.

The Influence of Fluid Flow on the Microstructure of Directionally Solidified AlSi-Base Alloys: *S. Steinbach*[1]; L. Ratke[1]; [1]Institute of Space Simulation, German Aeros. Ctr. DLR, D-51147 Cologne Germany

A quantitative understanding of the effect of fluid flow on the microstructure of cast alloys is still lacking. We therefore solidified AlSi-base alloys directionally under well defined thermal and magnetically controlled, convective boundary conditions. This was achieved with a new furnace facility that utilizes the extreme properties of transparent nanostructured silica aerogels as a crucible material, leading to flat isotherms and allowing the direct optical observation of the solidification process. Three pairs of Helmholtz coils around the sample can induce a homogeneous rotating magnetic field. The field strength can be related directly to the flow field inside the liquid and the mush using numerical modelling. The investigations show that the microstructural features like the primary dendrite, the secondary dendrite arm spacing, the eutectic spacing and the fraction solid change in a unique manner with solidification speed and rotating magnetic field strength. The experimental results are compared to literature data and accepted steady-state growth models. Simple models are derived for the effect of convection on microstructure parameters.

Analysis of the Solidification of Particle-Laden Melts: *Dawei Sun*[1]; Suresh V. Garimella[1]; Aniruddha Mukhopadhyay[2]; [1]Purdue University, Sch. of Mechl. Engrg., W. Lafayette, IN 47907 USA; [2]Fluent Inc., Lebanon, NH 03766 USA

A numerical model, based on the enthalpy-porosity method and the volume of fluid (VOF) method, has been developed for the analysis of coupled fluid flow and heat transfer processes during the casting of particle-laden fluids. The primary focus of this work is the prediction of solidification shrinkage. The influence of the suspended particles in the non-Newtonian fluid on the phase change process is taken into account. The proposed model is first validated against experimental measurements, and then used to investigate pipe formation in the casting of energetic materials. It has been found that solidification shrinkage plays an important role in the casting process, and the extent to which the non-Newtonian nature of the fluid needs to be considered depends on the concentration of the suspended particles.

Crystal Growth in Polymeric Materials: *Jing Teng*[1]; Shan Liu[2]; Rohit Trivedi[1]; [1]Iowa State University, Dept. Matls. Sci. Engrg., 225 Wilhelm Hall, Ames, IA 50010 USA; [2]Iowa State University, Ames Lab.-USDOE, Matls. & Engrg. Physics, 235 Wilhelm Hall, Ames, IA 50010 USA

In comparison with metallic alloys, polymer crystals have a much less symmetrical crystal structure and very complicated growth morphologies. Instead of crystallizing by the atomic jump across the growth interface in the small molecular and metallic alloys, polymer crystallization proceeds with the secondary nucleation and conformational change. Spherulites are the most common growth form in polymers and have been widely used for study of kinetics in undercooled growth process. In this paper, we propose to use the directional growth technique to determine the growth kinetics since the interface temperature and growth velocity can be precisely measured concomitantly. The data were compared with the nucleation theory and a growth regime change at ~16K undercooling was observed for PEG. A crossover of the growth kinetics curves for the undercooled and directional growth was found, which is believed to be due to the difference in heat transfer between the two growth configurations.

Undercooling of Liquid Iron Droplets Partly Submerged in Slags and in Contact with Oxides: *Martin Emiliano Valdez*[1]; Seetharaman Sridhar[1]; Alan W. Cramb[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Wean Hall 2325, Pittsburgh, PA 15213 USA

This paper describes a novel experimental technique to measure undercooling of metals and alloy droplets when the only interface available is a steel/slag interface. The methodology involves observing the shape change of a small droplet submerged in a slag without contacting the crucible walls through a Confocal Scanning Laser Microscope (CSLM). The results presented are for pure Fe droplets in a silicate based slag. The experiments indicate that fully submerged particles can be significantly undercooled (up to 260 K). The extent of undercooling increases after each successive re-melting of the sample, which suggests that impurity content has a strong effect on the undercooling.

Characterization of Carbide Growth in Directionally Solidified High Chromium White Cast Iron: *Dong Shyen Yang*[1]; Tien Shou Lei[1]; [1]National Taiwan University of Science and Technology, Dept. of Mechl. Engrg., 43 Keelung Rd., Sect. 4, Taipei 106 Taiwan

The characterization of carbide growth in directionally solidified castings of ASTM A532-87 Class - V high chromium white cast iron is investigated. A vertical cylindrical casting set-up which consists of two layers of Furan sand mold and sandwiched with a chilled copper mold was used for directional solidification. This solidification set-up provides three zones with different solidification direction: an upward zone above the chilled mold, a bidirectional solidification zone within the chilled copper mold, and a downward zone below the chilled mold. Due to the effect of gravity and fluid convection the solidification characteristics in these three zones are different. The morphology of primary alloy carbide M7C3 shows differences in these three zones, specifically, long rods of M7C3 carbide up to 3 mm were grown in the chilled mold. Other than SEM used for morphologies, the EBSD results will be used to study the preferred crystallographic orientation of the M7C3.

Numerical Simulation of Thermal Behavior During 3-D Laser Deposition: *Baolong Zheng*[1]; Yaojun Lin[1]; Yizhang Zhou[1]; John E. Smugeresky[2]; Enrique J. Lavernia[1]; [1]University of California, Chemical. Engrg. & Matls. Sci., 1 Shields Ave., Davis, CA 95616 USA; [2]Sandia National Laboratories, Dept. 8724, Livermore, CA 94551-0969 USA

As a result of the layer additive nature associated with the laser engineered net shaping (LENS™) process, the parts to be built would experience thermal cycles during LENS™ deposition. The laser deposition of LENS™ process can be viewed as a sequence of discrete events, and accordingly, the thermal history could be modeled numerically by using the finite difference methods (FDM). The objectives of this study are to: (1) understand the complete thermal behavior during LENS™ processing, and (2) correlate the processing parameters (e.g.,

substrate temperature, laser power, substrate traverse speed) with the microstructure and properties of parts. The numerical results from a series of designed experiments of different geometries will be compared with the experimental observations from samples processed with closed loop feedback control. Work by Sandia is supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

Interactions of Dendrites with Embedded Particles: *Yi Yang*[1]; Justin Wayne Garvin[1]; Holavanahalli S. Udaykumar[1]; [1]University of Iowa, Dept. of Mechl. Engrg., 3131 Seamans Ctr., Iowa City, IA 52242 USA

Numerical simulations of dendrite-particle interactions, which are important in metal-matrix composite production, are performed with a sharp interface method. A pure under-cooled melt is used to grow the dendrite. When the dendrite approaches the particle, appropriate conditions are applied at the particle-solid interface before and after contact. The behaviour of dendrites as they approach and grow around the particle is closely examined. For a particle-melt thermal conductivity ratio kp/kl < 1.0, the solidification front does not get close enough to the particle to activate the particle pushing mechanism. Instead, the solidification front chooses to go around the particle, and eventually the particle is engulfed by sidebranches. This result contradicts theoretical treatments of planar front interactions with a particle. Dendrite-particle interactions in alloys are also investigated.

Frontiers in Thin Film Growth and Nanostructured Materials: A Symposium in Honor of Prof. Jagdish Narayan: Semiconductors

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee

Program Organizers: N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA; Orin Wayne Holland, University of North Texas, Department of Physics, Denton, TX 76203 USA; Sungho Jin, University of California, Department of Materials Science, La Jolla, CA 92093 USA; Stephen J. Pennycook, Oak Ridge National Laboratory, Sold State Division, Oak Ridge, TN 37831 USA; Rajiv K. Singh, University of Texas, Austin, TX 78758-4455 USA

Tuesday PM Room: 3020
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Roger Narayan, Georgia Institute of Technology, Biomatls. & Bioengrg., Atlanta, GA 30332-0245 USA; Douglas B. Chrisey, Naval Research Laboratory, Washington, DC 203375-5345 USA

2:00 PM
Dilute Magnetic Semiconductors: *John T. Prater*[1]; [1]Army Research Office, PO Box 12211, Rsch. Triangle Park, NC 27709-2211 USA

The recent revelation that the magnetic spin component associated with electronic charge could be used to provide enhanced functionality in electronic devices has led to a flurry of research on dilute magnetic semiconductors. In particular, studies on the magnetic doping of various wide band-gap semiconductors have been conducted across the country in search of systems that will display room temperature ferromagnetism. An overview of the progress made to date by the various groups around the country will be presented. This overview will also include some recent findings by Professor Narayan's research group at North Carolina State University, where I have had the honor of establishing a most rewarding research collaboration. Recent breakthroughs for cobalt doped systems suggest that a viable materials solution may be near at hand. The challenge will soon turn from material discovery to materials integration. Here the concepts of domain matching epitaxy (DME), which were first forwarded by Professor Narayan in the late 1990's, will afford expanded options for device fabrication. The DME concept will be presented as it applies to spintronic device integration and the deposition of magnetic semiconductors onto such mainline commercial substrates as silicon.

2:30 PM Invited
Non-Random Distribution of Atomic Species in Mixed III-V Layers: *Subhash Mahajan*[1]; [1]Arizona State University, Cheml. & Matls. Engrg., PO Box 876006, Tempe, AZ 85287-6006 USA

We will demonstrate that atomic species in mixed III-V layers, differing in their covalent tetrahedral radii, are not distributed at random on their respective sub-lattices. Two types of deviations from randomness are observed: phase separation and atomic ordering. Both of these features evolve at the surface or near surface regions during growth, and reduce the strain energy of the system. Our recent work on mixed group III nitride layers shows that InGaN layers indeed undergo two-dimensional phase separation, and exhibit weak tendency for 1:1 ordering. Al0.5Ga0.5N layers also show 1:1 ordering that is sensitive to composition. The above microstructural features affect the electronic and optical properties of layers, and should enhance the degradation resistance of light emitting devices. The author is grateful to NSF, AFOSR, ONR and DOE for research support.

3:00 PM Invited
An Atomistic Kinetic Framework for Strain Accomodation in Lattice Mismatched Semiconductor Epitaxy: *Anupam Madhukar*[1]; [1]University of Southern California, Depts. of Matls. Sci. & Physics, Los Angeles, CA USA

The vast majority of descriptions of strain accommodation in lattice mismatched semiconductor overlayer-substrate systems rely upon comparison of the ground state energies of a (partially or wholly) strain relaxed atomic configuration with that of an assumed non-relaxed reference state, for a fixed number of particles. Tremendous strides have been made in reconciling and understanding experimental findings using such analyses, applied appropriately. Yet, growth of a film by its nature is the evolution of a system with changing number of particles. It is thus reasonable to consider an atomistic view in which the initiation, growth, and multiplication of strain relieving defects are dynamic events, tied to the changing kinetics and thermodynamics of the interacting open system. In this talk I shall present such a framework, motivated by molecular beam epitaxy of III-V compound semiconductors. It is necessarily incomplete, given the complex nature of a dynamical open system, but it does provide deeper insights into the nature of strain accommodation processes. It also lends itself to examination via multi-length and multi-time scale simulations and visualization carried out on increasingly more accessible parallel computing platforms, thus holding the promise of revealing potential pathways by which the system moves towards the experimentally observed state.

3:30 PM Invited
Core Structure of Screw Dislocation in GaN Studied by Transmission Electron Microscopy: *Z. Liliental Weber*[1]; [1]Lawrence Berkeley National Laboratory, Berkeley, CA USA

Thin film heteroepitaxy of polar materials such as GaN grown by MOCVD, MBE or HVPE Molecular Beam Epitaxy (MBE) grown on SiC or Al2O3 is frequently hampered by the formation of structural defects mostly dislocations, nanotubes and pinholes. In this presentation it will be shown that the screw dislocations present in HVPE samples are decorated by pinholes arranged on top of each other ("bamboo" structure) but these defects are not found in MBE samples grown on the top of HVPE samples. These might suggest either different core structure of a screw dislocation in HVPE and MBE grown material or suggest a different purity of these two materials. MBE samples grown under Ga-rich and Ga-lean material have been studied. A direct reconstruction of the phase and amplitude of the scattered electron wave from a focal series of high-resolution transmission electron microscopy (TEM) images were applied to learn about a core structure of screw dislocation. It will be shown that cores in MBE samples were filled and stoichiometric but in HVPE materials have an excess of Ga.

4:00 PM Break

4:15 PM Invited
Development of Separated Pulsed Laser Ablation for Oxide Light Emitting and Semiconducting Thin Films: *Kenji Ebihara*[1]; Manuel Alonso[1]; San-Moo Park[1]; Tomoaki Ikegami[1]; [1]Kumamoto University, Electl. & Computer Engrg., Kurokami 2-39-1, Kumamoto 860-8555 Japan

New type pulsed laser ablation process(separated pulsed laser deposition: SPLD) has been proposed for oxide thin film preparation using ozone and NO. The SPLD consists of the ablation chamber and the deposition chamber with the orifice. The ablation chamber is a stainless steel globe with 300 mm diameter. The deposition chamber is made with a quartz tube of 100 mm diameter and a metallic conic wall with variable orifice diameters. For the ablation process we use a KrF laser with =248 nm and 25 ns pulse duration. The differential vacuum and gas pressure in the two chambers allows us to have some control over the doping gas pressure, the plasma plume and the ion implantation by applying a variable voltage between the conic wall and the substrate. Typically this SPLD technique produces thin film ZnO at low temperature and leads to the creation of nano-hybrid thin film structures.

4:45 PM
Electrodeposition of Cd-Zn-Te Thin Films: *Krishnan Selva Raja*[1]; [1]University of Nevada, Metallurgl. & Matls. Engrg., 1661 N. Virginia St., MS 388, Reno, NV 89557 USA

Cadmium Zinc Telluride (CZT), a compound-type semiconductor, is a predominant radiation sensing material capable of detecting low to medium energy gamma radiation and hard X-rays. Conventionally, CZT (typical composition Cd0.9Zn0.1Te) is manufactured as a single crystal using Bridgman techniques and the detection efficiency of the CZT is improved by minimizing the bulk defects present in the crystal. Thin film and nano-wires of CZT material could exhibit 1or 2 D electron transport and therefore the detection efficiency can be improved substantially. In this study, thin films of CZT were electrochemically deposited in a single step process as well as two-steps process followed by annealing. Cyclic voltametry studies were carried out to understand the underlying electrochemistry of Cd-Zn-Te compound deposition. XRD and TEM studies are being carried out to characterize the thin films.

5:15 PM Invited
Structural, Optical, Magnetic and Electrical Properties of V Doped ZnO Thin Films: *Shivaraman Ramachandran*[1]; *Ashutosh Tiwari*[1]; *J. Narayan*[1]; [1]North Carolina State University, Matls. Sci., 2141 Burlington Labs, CB 7916, Raleigh, NC 27606 USA

Here we report a systematic study of structural, optical, electrical and magnetic properties of thin films of vanadium doped ZnO. Epitaxial thin films of ZnxV1-xO (x=0.01 to x=0.2) were prepared on sapphire c-plane single crystals using pulsed laser deposition technique. All the growths were done at temperatures ranging from 500-650°C. The emphasis in this work has been on determining the magnetic properties of these diluted magnetic semiconducting (DMS) systems and to correlate the properties, especially the magnetic properties with the microstructural aspects, such as occupation of the magnetic dopant in the lattice sites versus clusters and/or precipitates. It is now established that this system is devoid of any signatures of ferromagnetism either at room temperature or at low temperatures. The magnetic measurements were done using either a vibrating sample magnetometer or a superconducting quantum interference device (SQUID). The microstructural characterization was done using high-resolution transmission electron microscopy, coupled with electron energy loss spectroscopy (EELS) and STEM-Z (Scanning Transmission Electron Microscopy-Atomic Number) contrast experiments.

5:45 PM
Controlling Interface Effects in Si Solar Cells: *Bhushan Sopori*[1]; N. M. Ravindra[2]; Chuan Li[1]; Paresh Saxena[2]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA; [2]New Jersey Institute of Technology, Dept. of Physics, Newark, NJ 07102 USA

Fabrication of efficient Si solar cells requires high-quality wafers that have long minority carrier diffusion lengths and well-controlled interfaces/surfaces— interfaces that can offer very low carrier recombination. Control of interface properties is particularly important as the wafer thickness is reduced. Several approaches have been used for minimizing the carrier recombination velocity at the interfaces, which include minimizing interface state density and formation fields that can repel photogenerated minority carriers. Procedures for minimizing interface density are well established in the MOS technology. The minority carrier reflecting fields can be developed by formation of high-low junctions, dopant profiling, and by introduction of suitable surface charges, typically within a dielectric layer. In commercial Si solar cell fabrication, there can be conflicting demands between surface passivation and other cell requirements such as optical properties, processing schedules, and control of costs. This paper will discuss the effectiveness of these techniques and their compatibility with low-cost solar cell fabrication approaches.

General Abstract Session: Environmental Damage and Durability

Sponsored by: TMS
Program Organizers: Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA

Tuesday PM — Room: 2011
February 15, 2005 — Location: Moscone West Convention Center

Session Chair: Alan K. Pelton, Nitinol Devices & Components, Fremont, CA 94539 USA

2:00 PM
Environmental Durability of Coated GRCop-84 Copper Alloys: *S. V. Raj*[1]; [1]NASA Glenn Research Center, Matls. Div., MS 106-5, 21000 Brookpark Rd., Cleveland, OH 44135 USA

An advanced Cu-8(at.%)Cr-4%Nb alloy developed at NASA's Glenn Research Center, and designated as GRCop-84, is currently being considered for use as liners in combustor chambers and nozzle ramps in NASA's future generations of reusable launch vehicles (RLVs). However, past experience has shown that unprotected copper alloys undergo an environmental attack called 'blanching' in rocket engines using liquid hydrogen as fuel and liquid oxygen as the oxidizer. Potential for sulfidation attack of the liners in hydrocarbon-fueled engines is also of concern. As a result, protective overlay coatings alloys are being developed for GRCop-84. The oxidation behavior of several new coating alloys has been evaluated. GRCop-84 specimens were coated with several copper and nickel-based coatings, where the coatings were deposited by either vacuum plasma spraying or cold spraying techniques. Coated and uncoated specimens were thermally cycled in a furnace at different temperatures in order to evaluate the performance of the coatings. Additional studies were conducted in a high pressure burner rig using a hydrocarbon fuel and subjected to a high heat flux hydrogen-oxygen combustion flame in NASA's Quick Access Rocket Exhaust (QARE) rig. The performance of these coatings are discussed.

2:30 PM
Hydrogen Effects on the Phase Stability of NiTi: *Katherine C. Chen*[1]; Amanda Runciman[1]; Alan R. Pelton[2]; Andreas Wick[2]; [1]California Polytechnic State University, Matls. Engrg. Dept., 1 Grand Ave., San Luis Obispo, CA 93407 USA; [2]Nitinol Devices & Components, 47533 Westinghouse Dr., Fremont, CA 94539 USA

Shape memory and superelastic NiTi are finding its way into several commercial products (e.g., endovascular stents, cell phone antennae wire, orthodontic arch wire), as well as potential new applications (such as morphing wings). The A_f (austenite transformation finish) temperature is usually the key property to control, and is very sensitive to processing conditions. During cleaning, etching, or heat treatments, hydrogen can be inadvertently introduced into the NiTi and affect the phase stability and properties. Samples of various amounts of hydrogen in NiTi have been prepared and studied by x-ray diffraction (XRD). With only roughly 80 wppm of H in austentic NiTi, new XRD peaks appear. Structural effects of hydrogen in martensitic NiTi are also investigated. Hydrogen-induced lattice strains and the appearance of new hydride phases are discussed and correlated against previous diffusion studies.

3:00 PM
A Study on the Electrochemical Properties of Si-Cu-Carbon Composite for an Anode Material of Li-Ion Batteries: *Yong-Mook Kang*[1]; Min-Sik Park[1]; Min-Sang Song[1]; Hyun-Seok Kim[1]; Kyu-Sung Han[1]; *Jai-Young Lee*[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Engrg., 373-1, Guseong-Dong, Yuseong-gu, Daejeon S. Korea

In the previous reports, Si-Carbon composite has been suggested to improve the cycle life of Si. Even if Si-Carbon composite showed good cycle life in Si utilization of 32% (This means that 32% of the theoretical capacity was used.), its cycle life was precipitously deteriorated in Si utilization of 40%. On the other hand, Si-Cu-Carbon composite exhibited good cycle life (After 50 cycles, more than 98% of the initial capacity was maintained.) even in Si utilization of 55%. Then, the capacity of Si-Carbon composite was 679 mAh/g, while that of Si-Cu-Carbon composite came to 930 mAh/g. As shown in this result, the

cycle life of Si-Cu-Carbon composite was drastically improved compared with Si, and its Si utilization was much higher than that of Si-Carbon composite.

3:30 PM
Effect of Strain Rate and Environment on the Mechanical Behavior of the Ni-19Si-3Nb-0.15B-0.1C Intermetallic Alloy at High Temperature: *Shian-Ching Jason Jang*[1]; [1]I-Shou University, Dept. of Matls. Sci. & Engrg., 1, Sec. 1, Shiuecheng Rd., Dashu Shiang, Kaohsiung County 840 Taiwan

The effect of strain rate and environment on the mechanical behavior at different temperature for the Ni-19Si-3Nb-0.15B-0.1C was investigated by atmosphere-controlled tensile test in various conditions (different strain rate at different temperature). The results revealed that the Ni-19Si-3Nb-0.15B-0.1C alloy exhibits ductile mechanical behavior (UTS ~ 1250 MPa, ?Õ ~ 14%) at temperature below 873K in vacuum (2 x 10¡V4 torr), in air, and in water vapor (850 ppm water vapor) atmosphere. This indicates that the microalloying of boron and carbon does overcome the environmental embrittlement of water vapor at the testing temperature below 873K for the Ni-19Si-3Nb base alloy. However, the ductility of the Ni¡V19Si-3Nb-0.15B-0.1C alloy tested in air and vacuum drops significantly when the temperature increases to 973 K. In addition, the strain rate effect on the tensile strength of Ni¡V19Si-3Nb-0.15B-0.1C alloy was revealed insensitive over a wide temperature from room temperature to 1073 K. The detail of mechanical behavior related to the strain rate and temperature will be discussed in this paper.

4:00 PM Break

4:20 PM
Temperature Dependence of Delayed Hydride Cracking Velocity in Ti-6Al-4V Alloy: *Young Suk Kim*[1]; Seung Jun Choi[1]; Kyung Soo Im[1]; [1]Korea Atomic Energy Research Institute, Zirconium Team, PO Box 105, Yuseong, Daejeon 305-353 Korea

The Ti-6Al-4V sheet with the -Ti and the phase was subjected to electrolytic charging of 1000 ppm H and to constant load tests at temperatures varying from 10°C to 200°C. The delayed hydride cracking velocity (DHCV) was determined using compact tension specimens with the pre-fatigued crack growing along the rolling direction of the sheet. The DHCV of the Ti-6Al-4V alloy decreased with the test temperature increasing from 20 to 100°C and also with the test temperature decreasing below the zero temperature. This fact is quite in contrast with the DHCV of Zr-2.5Nb alloys that increases with the increasing temperature from RT to 300°C and decreases at temperatures in excess of 300°C. Based on the Kim's DHC model where the driving force for the DHC is a supersaturated hydrogen concentration or C over the terminal solid solubility of hydrogen for dissolution arising from a hysteresis of the terminal solid solubility of hydrogen, we discussed the different temperature dependency of the DHCV for the Ti-6Al-4V alloys and the Zr-2.5Nb both of which have the microstructure of the - and -phases.

4:50 PM
Performance Optimisation of Gas Turbine Power Output and Thermal Efficiency With F-Technology System: *Rama Rao Adapa*[1]; D. N. Reddy[2]; K. V. Sharma[3]; [1]VNR VJIET, Mechl. Engrg., Bachupally, Kukatpally, Hyderabad, Andra Pradesh 500 072 India; [2]Osmania University, Mechl. Engrg., Coll. of Engrg., Hyderabad, Andra Pradesh 500 007 India; [3]JNTU, Ctr. for Energy Studies, Coll. of Engrg., Hyderabad, Andra Pradesh 500 072 India

The performance optimization of Gas Turbine (GT) with F-Technology System is presented that yields higher thermal efficiency and higher power output than the existing G.T.Power Plants. F-Technology means to increase the Fire Point in the combustion chambers in G.T. which in turn increases the Turbine Inlet temperature thereby the Heat Energy (HE) converted into Kinetic Energy (KE) on the G.T. Nozzles and K.E. is converted into Mechanical Energy (ME) on the G.T.Rotor Blades which results in rotating the Generator Rotor thereby producing the higher power output and thermal efficiency. To withstand the higher temperature in the hot zones in the G.T. Sections Hastalloy material in combustion chambers, FSX 414 for G.T. Nozzles, IN 706 for G.T. hot zone parts, ASTM A 336 for shrouds, and Ni Cr MO forged steel for all the G.T. Rotor discs are preferred. Performance, calculations being done to get optimized results with relevant sources.

Hume-Rothery Symposium: The Science of Complex Alloys

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Chemistry & Materials Science, Livermore, CA 94551 USA; Thaddeus B. Massalski, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

Tuesday PM Room: 3008
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: S. Joseph Poon, University of Virginia, Dept. of Physics, Charlottesville, VA 22904-4714 USA; Jean-Marie Dubois, Ecole des Mines de Nancy, Lab. de Sci. et Génie des Matériaux et de Métall., Nancy 54042 France

2:00 PM Invited
Electronic Structure of Complex Hume-Rothery Phases in Transition-Metal Aluminides: *Guy Trambly de Laissardière*[1]; Duc Nguyen-Manh[2]; Didier Mayou[3]; [1]Université de Cergy-Pontoise, Lab. Physique Théorique et Modélisation, St. Martin, 2 av. A Chauvin, Cergy-Pontoise 95302 France; [2]UKAEA Fusion, Culham Sci. Ctr., Abingdon OX1 430B UK; [3]CNRS, Lab. D'Etudes des Propriétés Electroniques des Solides, BP 166, Grenoble, 38042 France

The discovery of quasicrystals phases and approximants in Al-Mn system has revived the interest of complex aluminides containing transition-metal atoms. On the one hand, it is now accepted that the Hume-Rothery stabilization plays a crucial role. On the other hand, TM atoms have also a very important effect on their stability and their physical properties. But, until recently, there has been no model taking into account these two aspects together. In this paper, we review a model that unifies the classical Hume-Rothery stabilization for sp electron phases with the virtual bound state model for transition-metal atoms embedded in the aluminum matrix. This new theory for "spd electron phases" is applied successfully to Al(Si)-transition-metal alloys and it gives a coherent picture of their stability and physical properties. It is compared to first-principles calculations of the electronic structure and experimental results.

2:30 PM Invited
Hume-Rothery Rules for Stable Quasicrystals: *An Pang Tsai*[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsch. for Advd. Matls., Katahira 2-1-1, Sendai 980-8577 Japan

The stability of the icosahedral quasicrystals has been studied in terms of Hume-Rothery rules. We have confirmed that most of stable quasicrystals cann be understood within the frame work of the Hume-Rothery rules. Even more interestingly, it is found that stable quasicrystals are strict electron compounds, which only form at sharp valence electron concentration. All of the quasicrystals commonly have the corresponding valence electron concentration, but the dependence of the stability on atomic size factor is different among the groups. Qualitatively, the most relevant criterion for the formation of stable quasicrystals is that the alloy should have a definite valenceelectron concentration. On top of this, a high stability of the quasicrystal is observed when the atomic size facto is favored.

3:00 PM Break

3:20 PM Invited
Hume-Rothery Rule as a Formation Condition of New Icosahedral Quasicrystals: *Tsutomu Ishimasa*[1]; [1]Hokkaido University, Div. of Applied Physics, Kita 13-jou, Nishi 8-chome, Kita-ku, Sapporo, Hokkaido 060-8628 Japan

In the past four years, several new quasicrystals have been discovered in Zn-, Cu-, Cd-, and Ag-In- based alloys. They belong to new family of quasicrystals having a unique type of atomic cluster as a local structural unit, which is different from those in the Al-based Mackay-type and the Zn-based Bergman-type quasicrystals. Many quasicrystals belonging to this family are thermodynamically stable and exhibit remarkable high degree of structural perfection, for example in the cases of Zn-M-Sc where M denotes Mg, Fe, Co, Ni, Pd and Ag. The purpose of this presentation is to review the structural properties, in particular structural relationship to the corresponding crystalline approximants, and the formation conditions of this unique type of quasicrystal. The role of Hume-Rothery rule as a formation condition is an interesting and important subject.

3:50 PM Invited
On the Formation of Quasicrystals in Zr-Based Metallic Glasses: *Uwe Koster*[1]; [1]University of Dortmund, Dept. Biochem. & Chem. Eng., Dortmund D - 44221 Germany

In a large number of metallic glasses quasicrystals form first upon devitrification. This indicates a strong influence of the short-range order in the melt, quenched-in into the metallic glass. Observations on the influence of the quenching temperature on quasicrystal formation in melt-spun Zr-Cu-Ni-Al glasses as well as on the influence of different routes of glass formation on the phase selection in Zr-Pd confirm such an assumption. Formation of quasicrystals is explained by various arguments: Quasicrystals are often considered as Hume-Rothery phases, atomic size or bond strength dependent structures, or hybrid structures built e.g. in Zr-Cu-Ni-Al glasses from Al_2Cu- and $MoSi_2$-type building blocks. In the discussion on quasicrystal formation in Zr-based metallic glasses melt temperature, quenching rate, oxygen or hydrogen contamination, alloying with small amounts of additional elements or the exchange of the late transition metals are assumed as ruling parameter for phase selection as well as their nucleation and growth rates.

4:20 PM Invited
Phase Equilibria and Thermodynamics of Ca-Based Metallic Glasses: Michael C. Gao[2]; *Gary J. Shiflet*[1]; S. Joseph Poon[3]; Marek Mihalkovic[4]; Mike Widom[4]; [1]University of Virginia, PO Box 400745, 116 Engr.'s Way, Charlottesville, VA 22904-4745 USA; [2]Northeastern University, Dept. of Physics, Boston, MA 02115 USA; [3]University of Virginia, Dept. of Physics, Charlottesville, VA 22904-4745 USA; [4]Carnegie Mellon University, Dept. of Physics, Pittsburgh, PA 15213 USA

The CALPHAD method has become a powerful tool in thermodynamic modeling of multi-component systems. However, when there is little literature information available, critical experiments are needed to validate the CALPHAD modeling. This is the case for the Al-Ca-Cu ternary phase diagram (especially on the Ca-rich side). Research in this system originates from the recent discovery, at the University of Virginia, that an unusually broad glass formation range (GFR) has been identified near the Ca-rich side that includes several bulk glass chemistries. This is in contrast to the narrow GFR reported earlier with much poorer glass forming ability on the Al-rich side. The GFR was determined using melt-spinning and die-casting techniques, and its structural and thermal stability was studied using XRD and DSC. The solid-state phase equilibria were investigated using XRD, SEM, EBSD and TEM, while DTA was used to characterize the solidus and liquidus temperatures. In order to assist this study, first-principles calculatio ns were performed on this system and have proven to be important in prediction of phase diagrams (e.g., solubility range) and minimizing experimental uncertainty, including phase chemistry and structure. In this talk, details on integration of these calculations with experiments and modeling will be emphasized. The application of such self-consistent accurate thermodynamic descriptions to explain the observed glass formation will be discussed.

Lead Free Solder Implementation: Reliability, Alloy Development, New Technology: Electromigration, and Electrical "Aging" of Lead-Free Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Mark A. Palmer, Kettering University, IMEB, Flint, MI 48504-4898 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Nikhilesh Chawla, Arizona State University, Department of Chemical and Materials Engineering, Ira A. Fulton School of Engineering, Tempe, AZ 85287-6006 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada

Tuesday PM — Room: 3014
February 15, 2005 — Location: Moscone West Convention Center

Session Chairs: Laura J. Turbini, University of Toronto, CMAP, Toronto, ON M5S 3E4 Canada; Sinn-Wen Chen, National Tsing-Hua University, Dept. of Cheml. Engrg., Hsinchu 300 Taiwan

2:00 PM Invited
The Effect of Temperature on Conductive Anodic Filament Formation: *Antonio Caputo*[1]; Laura J. Turbini[1]; [1]University of Toronto, Matls. Sci. & Engrg., 184 College St., Toronto, ON M5S 3E4 Canada

Conductive Anodic Filament (CAF) is a failure mode in printed wiring boards (PWBs) which occurs under high humidity and high voltage gradient conditions. The filament, a copper salt, grows from anode to cathode along the epoxy-glass interface. The rate limiting step is delamination of the epoxy-glass interface. This can occur due to mechanical stress or thermal stress associated with CTE mismatch between the polymer and reinforcement in the board. The move from lead-based (201-205°C) to lead-free soldering conditions (240-245°C) will accelerate epoxy-glass delamination and therefore enhance CAF growth. Bent and Turbini have shown that PWBs exposed to lead-free processing conditions have a greater incidence of CAF failure than those exposed to conventional processing temperatures. This paper presents data on new "halogen free" laminate materials being developed for lead-free soldering temperatures. It will evaluate their CAF susceptibility at both lead and lead-free soldering conditions.

2:30 PM
3-D Current Density Simulation in Flip Chip Solder Joint Under Electrical Current Stressing: *T. L. Shao*[1]; Shih-Wei Liang[2]; Chih Chen[1]; [1]National Chiao Tung University, Matl. Sci. & Engrg., 1001 Ta-Hseuh Rd., Hsin-Chu 30050 Taiwan; [2]National Chiao Tung University, Dept. of Applied Math., 1001 Ta-hseuh Rd., Hsin-Chu 30050 Taiwan

3-D current density distribution of the flip chip solder joint under electrical current stressing was simulated by finite element method. The current density distribution inside the flip chip joint was examined under 0.567 ampere current stressing. Four kinds of under bump metallurgies (UBM) structures, including the 0.7 micron Cu/0.3 micron Cr-Cu/0.1 micron Ti thin film UBM, 5 micron Cu UBM, 5 micron Ni UBM, and 3 micron Ni/5 micron Cu UBM, were investigated for the flip chip solder joints. The maximum current density, which ranges from 41,740 to 187,492 A/cm2, always occurs at the entrance of Al trace into the flip chip solder joint. The maximum current crowding ratio of the flip chip solder bump varies from 19.62 to 88.12. The flip chip solder bump of thin film UBM has the largest value of current density and current crowding ratio.

2:50 PM
In Situ Observation of Flip-Chip Solder Joints Under Current Stressing: *Chia Ming Tsai*[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matls. Engrg., Jhongli City 320 Taiwan

The effect of electron flow on the motion of Pb phase in flip chip solder joints was studied at room temperature. The solder joints had a diameter of 100 microns. The UBM (under-bump metallization) on the chip side had a Cu metallurgy, and the surface-finish on the substrate side had the Au/Ni metallurgy. In order to have in-situ observa-

tion, the samples were cross-sectioned through the centers of the solder joints before current stressing. The current density through the solder was about 3.1×10^4 A/cm^2. The dominant diffusing species was found to be Sn, as Pb had moved in the opposite direction of electron flow. The locations of the Pb grains were traced, and thus the motions of these grains were monitored. The nonuniform motion behavior of the Pb grains may correspond to the distribution of electron flow, which depends on the geometry of solder bump. The electronmigration flux and the effective charge were calculated.

3:10 PM
Effect of Current Crowding on the Failure Mechanism of Flip Chip Solder Joints Under Current Stressing: *Yen-Liang Lin*[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matls. Engrg., Jhongli City 320 Taiwan

The effect of current crowding on the flip chip solder joints during current stressing was studied. The solder was 63Sn37Pb eutectic solder, and the solder joints had a nominal diameter of 125 microns. In these joints, the UBM on the chip had a Cu/Ni/Al multiplayer structure, and that on the board side had a very thick Cu metallurgy. During current stressing, the flip chip packages were kept in an oven set at 150°C. The failure always occurred in joints that had electrons flowing from chip to substrate. Evidence of local melting was also observed. Some of the flip chip packages were placed at room temperature in order to perform in situ observation. In these in situ experiments, voids occurred in joints that had electrons flowing from chip to substrate. These voids grew wider and deeper with current stressing, and evetually could reach a depth of 36 microns.

3:30 PM Break

3:40 PM
The Effect of Applied Current on Sn-Pb Flip Chip Solder Bump: *Yeh-Hsiu Liu*[1]; Kwang-Lung Lin[1]; [1]National Cheng Kung University, Matls. Sci. & Engrg., No. 1, Ta-Hsueh Rd., Tainan 701 Taiwan

The electromigration-induced failure in flip chip solder bump of 5Sn-95Pb was studied after current stressing at 150°C with a current density of 5?~103 A/cm2 for up to 1711 h. The under-bump metallization (UBM) on the chip side was sputtered Al/Ni(V)/Cu thin films and on the board side was electroless Ni/Au surface finished. The diameter of the bumps was about 90 ?Êm. It was shown that the failure of the joints occurred at the chip side (cathode side). Simultaneously, the V layer in the UBM was damaged after current streesing up to 1711 h. Owing to no reaction occurring among V and Cu, Ni or Sn, there is no electromigration in the V layer so that its interface serves as an atomic flux divergence plane. The Cu-Ni-Sn intermetallic compound and void were found near the current crowding area in the chip side. The failure mechanism for 5Sn-95Pb flip chip solder bump will be discussed.

4:00 PM
Threshold Current Density of Electromigration in Pure Tin Films: *Hung-Chih Yu*[1]; Sue-Hong Liu[1]; Chih Chen[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Engrg., 1001 Ta Hsueh Rd., Hsin-Chu 30050 Taiwan

Electromigration in pure tin draws a lot of attention due to the implementation of Pb-free solders, in which tin weighs over 96% for most of the solders. Electromigration phenomenon is investigated under the current density of 7.5?~104?A1?~105 and 1.75?~105 A/cm2. The testing temperature were room temperature?A50?• and 75?•. Depletion and hillocks were found at the cathode side and anode side, respectively. The threshold current density at 50?• was measured to be about 4.4?~104 A/cm2 by extrapolating the plot of the applied current density versus drift velocity to zero drift velocity, and the threshold current density was approximately inversely proportional to the testing temperature. Other electromigration parameter, such as activation energy, and effective charge number, can be measured by this technique. They will be presented in details in the conference.

4:20 PM
Effect of Electromigration on Interdiffusion and Failure Mode in Composite Solder Joint: *Annie Tzu-yu Huang*[1]; Jae-Woong Nah[1]; [1]University of California, Matls. Sci. & Engrg., 6532 Boelter Hall, 405 Hilgard Ave., Los Angeles, CA 90095-1595 USA

Combination of the high melting 97Pb-3Sn or 95Pb-5Sn solder on the chip side and the low melting eutectic 37Pb-63Sn solder on the organic substrate side has recently been investigated for applications in high-end devices such as server. Our studies showed that interdiffusion of the high-Pb and the high-Sn solder is minimal during thermal annealing and the composite joint remains stable at a temperature as high as 150°C. However, when the composite solder joint is under current stressing, electromigration occurs and the composite solder joint becomes unstable due to void formation and intermetallic compound transformation. Because the dominant diffusing species (Pb or Sn) differs at different temperatures under electromigration, current stressing temperature also plays a role on failure mechanism in the composite solder joint.

4:40 PM
Study of Coupling Effect by Using Sandwich Structures of Cu/Sn/Various Basis Metals: *Shen-Jie Wang*[1]; *Cheng-Yi Liu*[1]; [1]National Central University, Cheml. & Matls. Engrg., No.300, Jungda Rd., Jhongli, Taoyuan 320 Taiwan

The coupling effect between Cu-Sn and Ni-Sn interfacial soldering reactions have been reported intensively. The interfacial reaction on Ni side would strongly be affected by the interfacial reaction on the Cu side, which further influences the reliability of solder joints. Currently, a practical solder joint structure often contains other metal layers beside Cu and Ni. Therefore, it is very important to study coupling effects for any other two different basis metal bond pads. In this study, Cu/Sn/X metals sandwich structures were used. The X metals are Au, Ag, Pd and so on. The preliminary results did show that the interfacial Cu-Sn reaction was affected simultaneously by the opposite soldering interfacial reaction. In this talk, we will report the coupling effects on the kinetics and morphology of interfacial Cu-Sn reaction.

5:00 PM
Combined Effect of Electromigration and Applied Stress on Solder Joint Failure: *Fei Ren*[1]; Jong-ook Suh[1]; Jae-Woong Nah[1]; King-Ning Tu[1]; Bingshou Xiong[2]; Luhua Xu[2]; John Pang[2]; [1]University of California, Dept. of Matls. Sci. & Engrg., 405 Hilgard, Los Angeles, CA 90095-1595 USA; [2]Nanyang Technological University, Dept. of Mechl. & Production Engrg., 50 Nanyang Ave., Singapore 639798 Singapore

The combined effect of electromigration and applied stress on Pb-free solder joints has been investigated. For tensile and creep tests, samples of Pb-free solder balls with a diameter of 300 ¥ìm were reflowed between two Cu wire-electrodes to form one-dimensional line structures of Cu-solder-Cu. For shear tests, flip chip samples were used. The tensile tests at a strain rate of about 3 ¥ìm/min were conducted after the samples were undergone electromigration at 1~5¡¿103 A/cm2 and at 100~150¨¬C. We observed that the failure always occurs at the cathode interface. Same kind of failure also occurs in a daisy chain of flip chip solder jumps when tested by shear after electromigration. Creep and electromigration were conducted simultaneously using the line samples, with the applied stress at 1 to 15 MPa. The combined effect of electrical and mechanical forces on solder joint failure under these different conditions will be discussed.

5:20 PM
Grain Growth of Tin Under Electromigration Studied by Synchrotron X-Ray Microdiffraction: *Albert T. Wu*[1]; King-Ning Tu[1]; J. R. Lloyd[2]; N. Tamura[3]; [1]University of California, Matls. Sci. & Engrg., 1677 Boelter Hall, Los Angeles, CA 90095 USA; [2]IBM T. J. Watson Research Center, PO Box 218, Yorktown Hgts., NY 10598 USA; [3]Lawrence Berkeley National Laboratory, Advd. Light Source, Berkeley, CA 94720 USA

When a white Sn (â-Sn) thin film stripe is subjected to electromigration, it shows a voltage drop typically about 10%. The reason might be due to the dependence of resistivity on anisotropic crystal structure of â-Sn; the c-axis is about 35% higher than a- and b-axes. Synchrotron x-ray micro-diffraction (~1µm in beam diameter) has been used to achieve grain-by-grain analysis of the changes of grain orientation in the Sn stripe before and after electromigration. Grain growth, with a mechanism which seems different from normal grain growth, was observed under electromigration; the low-resistance grain grew at the expense of a neighboring high resistance grain. We propose a different mechanism of grain growth under electromigration.

Magnesium Technology 2005: Magnesium Alloy Development

Sponsored by: Light Metals Division, International Magnesium Association, LMD-Magnesium Committee
Program Organizers: Ramaswami Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA

Tuesday PM Room: 2004
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Alan A. Luo, General Motors Corp, R&D Ctr., Warren, MI 48090-9055 USA; Alok Singh, National Institute for Materials Science, Matls. Engrg. Lab., Tsukuba, Ibaraki 305-0047 Japan

2:00 PM
Magnesium Castings in Aeronautics Applications - Special Requirements: *Achim Wendt*[1]; Konrad Weiss[1]; Arye Ben-Dov[2]; Menachem Bamberger[3]; Boris Bronfin[4]; Nir Moscovitch[4]; [1]RWP GmbH, Am Muensterwald 11, Roetgen 52159 Germany; [2]Israel Aircraft Industries, Tel Aviv Israel; [3]Technion, Haifa Israel; [4]Magnesium Research Institute, Beer-Sheva Israel

Although weight reduction in aircrafts is a fundamental matter use of Magnesium alloys is not widespread in the interior of airplanes due to "traditional" problems with corrosion resistance. In spite of the creation of new Magnesium alloys with improved corrosion resistance, these alloys are mostly designed for automotive industry. Alloys for aerospace industry must combine high performance regarding mechanical properties and corrosion resistance. A review is given on the use of Magnesium in aircrafts in the past. The paper then presents requirements of modern aircraft industry to Magnesium castings regarding to mechanical properties as well as corrosion properties. Today's drawback is the lack of a sufficient number of available alloys fulfilling the needs of modern aerospace industry. The methodology used in an international research project (IDEA) to develop new magnesium alloys for special use in aircrafts is described as well as the experimental and virtual methods utilised in this development.

2:20 PM
Effects of Aluminum Addition on Properties of Magnesium-Lithium Alloys: *Hsin Chin Lin*[1]; Kun Ming Lin[2]; Hsin Man Lin[2]; Cheng Hsiu Yang[2]; Ming Tang Yeh[3]; [1]National Taiwan University, Dept. of Matls. Sci. & Engrg., No.1, Sec. 4, Roosevelt Rd., Taipei 106 Taiwan; [2]Feng Chia University, Dept. of Matls. Sci. & Engrg., No. 100 Wenhwa Rd., Seatwen, Taichung 40724 Taiwan; [3]Hsu-Yang Technologies Co. Ltd, R&D Ctr., 80 Erh-Chia Rd., Ying-Keh, Taipei-Hsian Taiwan

The products of Mg alloys are mainly manufactured by using the die-casting because of their poor formability. However, the high ratio of defect products in die-casting of Mg alloys will reduce their manufacturing efficiency. Therefore, the developments of new Mg alloys with high formability of rolling, pressing and forging are important issues to improve the manufacturing ability of thin plates of Mg alloys. The two-phase Mg-Li alloys are reported to have excellent formability. However, their mechanical property and corrosion resistance are not well understood. In this study, we aim to study the microstructure, crystal structure, mechanical behavior and corrosion resistance of these two-phase Mg-Li alloys. The effects of Al addition on the Mg-Li alloy are also investigated. Preliminary results reveal that Al addition will effectively improve the mechanical property and corrosion resistance of two-phase Mg-Li alloys.

2:40 PM
Effects of Al Addition on Microstructure and Mechanical Properties of Mg-Zn-Y Alloys: *H. K. Lim*[1]; J. Y. Lee[1]; W. T. Kim[2]; D. H. Kim[1]; [1]Yonsei University, Ctr. for Non-Crystalline Matls., Dept. of Metallurgl. Engrg., 134 Shinchon-dong, Seodaemun-gu, Seoul 120-749 S. Korea; [2]Cheongju University, Dept. of Applied Sci., 36, Naedok-dong, Sangdang-gu, Cheongju, Chongbuk 360-764 S. Korea

Recently, efforts on quasicrystal reinforced magnesium alloys have been focused in order to overcome the disadvantages of magnesium alloys. The quasicrystal reinforced Mg-9Zn-2Y (in wt. %) alloy has been reported to exhibit a combination of high strength and ductility, and good formability at high temperature.[1] In the present study, we investigated the variation of microstructures and mechanical properties when the Zn/Y ratio from 4.5 to 2 (Mg-9Zn-2Y, Mg-6Zn-2Y and Mg-4Zn-2Y) and Al is added in Mg-Zn-Y alloy (Mg-(9-x)Zn-4Al-2Y (x=0, 3, and 5)). The microstructural dependence upon composition variations of Mg-Zn-RE alloys is discussed in terms of structural identification and the corresponding mechanical properties. [1]D.H. Bae, S.H. Kim, D.H. Kim, W.T. Kim, Acta Mater. 50 (2002) 2343.

3:00 PM
The Behavior of CaO in Magnesium Alloys: Jin-Kyu Lee[1]; Won Ha[2]; *Shae K. Kim*[1]; Young-Jig Kim[2]; [1]Korea Institute of Industrial Technology, Advd. Matl. Ctr., 994-32 Dongchun-dong, Yeonsu-Gu, Incheon 406-130 Korea; [2]Sungkyunkwan University, Dept. of Advd. Matl. Engrg., 300 Chunchun-dong, Jangan-gu, Suwon, Gyounggi-do 440-746 Korea

Mg alloys present a number of interesting properties, such as high specific strength, good castability, low density, etc. Despite these properties, Mg alloys are used with many precautions of high chemical reactivity, limited high strength and creep resistance at elevated temperatures. Research has been directed to improve these properties by alloying and/or composites manufacturing. It is well known that Ca, though its high cost, is effective for oxidation resistance and particle reinforced Mg composites have desirable high mechanical properties. The aim of this study is to manufacture CaO particle reinforced Mg composites in terms of improving oxidation resistance and mechanical properties. The Mg composites were manufactured by RCM (Rotation Cylinder Method) and the effects of CaO on oxidation resistance, fluidity and mechanical properties of Mg composites were investigated.

3:20 PM
The USCAR Structural Cast Magnesium Development Project: *Naiyi Li*[1]; Richard Osborne[2]; Bruce Cox[3]; Donald Penrod[4]; [1]Ford Motor Company, Mfg. & Processes, 2101 Village Rd., MD3135, Dearborn, MI 48124 USA; [2]General Motors Corporation; [3]DaimlerChrysler Corporation; [4]Manufacturing Services & Development, Inc.

The Structural Cast Magnesium Development Project is a jointly sponsored effort by the US Department of Energy (DOE) and the US Council for Automotive Research (USCAR) to identify and resolve technical and manufacturing issues that limit the light weighting opportunities of applying large-scale structural cast magnesium automotive components. This project, starting at the end of year 2001, comprises General Motors, Ford, DaimlerChrysler and thirty-four other North America companies and organizations to set its overall objective to determine the technical feasibility and practicality of producing and implementing an one-piece front engine cradle casting. This paper provides an overview of the project scopes, magnesium technology development, scientific understanding and up-to-date accomplishments. The front engine cradle made of AE alloy is introduced in various aspects including magnesium alloys property database development, component design and analysis, corrosion protection and coating strategy.

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3:55 PM
The Role of Rare Earth Elements in Structure and Property Control of Magnesium Die Casting Alloys: *Per Bakke*[1]; Hakon Westengen[1]; [1]Hydro Aluminium, Mg Competence Ctr., PO Box 2560, Porsgrunn N-3908 Norway

The performance of magnesium die cast parts is governed by the microstructure and by the distribution of structural features which occur as a result of the chemical composition and processing history of the alloy. The elevated temperature properties, especially mechanical strength under creep conditions, are primarily determined by the grain structure, the elements in solid solution and the effectiveness of second phase particles in stabilizing the grain boundaries. The current emphases in alloy development focus on the utilization of elements with low solubility in the solid state, leading to the formation of stable precipitates during solidification. Such elements include the rare earths, as well as silicon, strontium and calcium. A detailed analysis of the various microstructural features and attributes is given for a new family of rare earth-containing alloys. The optimization of alloy composition is addressed in terms of blending advantageous microstructural characteristics with phase equilibria considerations.

4:15 PM
Assessment of Superheating in Mg-Al Alloys by Thermal Analysis: *Peng Cao*[1]; Qian Ma[2]; David H. StJohn[1]; [1]University of Queensland, CRC for Cast Metals Mfg., Sch. of Engrg., St. Lucia, Queensland 4072 Australia; [2]Brunel University, Brunel Ctr. for Advd. Solidification Tech., Uxbridge, Middlesex, London UB8 3PH UK

The superheating grain refinement of Mg-9%Al alloy has been assessed by thermal analysis. The results obtained from the thermal analysis showed that after superheating nucleation temperature in-

creased while nucleation undercooling decreased. The grain size of the Mg-9%Al alloys decreased significantly after superheating. Microscopic examination showed that the particles present in the samples contained Al and Fe, no matter whether these samples were superheated or not. It is suggested that superheating causes grain refinement by adjusting the shape, size or distribution of the nucleant particles, rather than changing the composition of the nucleant particles.

4:35 PM
The Influence of Alloying Elements Upon Properties of Elektron 21 Alloy: *Paul Lyon*[1]; *Timothy E. Wilks*[1]; [1]Magnesium Elektron, TSD, PO Box 23, Swinton, Manchester M27 8DD England

Elektron 21 (ASTM Designation EV31) is a Mg-RE-Zn-Zr alloy designed for Aerospace and Speciality Applications. The alloy is capable of operating up to approximately 200C (400F). To achieve successful applications, Elektron 21 has had to exhibit not only good mechanical properties but also good corrosion performance and ease of castability. When developing the alloy, the effect of the Rare Earth (RE) constituents and Zinc were optimised to achieve these requisite alloy characteristics. Changes in the Rare Earth and Zinc content affect age hardening response. Control of individual Rare Earth components also influences castability. The level of Rare Earth component and, more importantly Zinc content, must be limited and controlled to achieve good and consistent corrosion performance. Finally, the effects of heat treatment variables were assessed to ensure that they are as broad as possible to aid produceability.

4:55 PM
The Effects of Grain Refinement on the Castability of Magnesium Permanent Mould Castings: *Timothy Loughnane*[1]; Deliang Zhang[2]; Darius P.K. Singh[1]; Thomas Neitzert[2]; [1]ION Automotive Ltd, Light Metal Castings Div., Auckland New Zealand; [2]University of Waikato, Dept. of Matls. & Process Engrg., Hamilton New Zealand

A gravity filled permanent mould casting has been designed to simulate the geometry and feeding habits of a typical automotive wheel. The casting geometry reflects the areas of a wheel casting that are most prone to defect formation, that is, the hub and the transition from the spoke to the rim where hot spots are often experienced. Different magnesium alloys were trialled along with various melt additions to facilitate the refinement of the microstructure during solidification. The effect of grain refinement on the castability of the alloys was investigated by measuring the resulting grain sizes, porosity levels, porosity types, as well as levels of hot tearing and surface slumping defects in the castings produced. It is expected that the conclusions drawn from this, and future investigations can be applied to the permanent mould casting of magnesium alloy wheels. The aim of this paper is to present and discuss the results of the investigation to date.

5:15 PM
Modern Melting Devices for Mg-Alloy for Die Casting Industry (Hot and Cold Chamber): Christoph Scheer[1]; *Roger Rapp*[1]; [1]MELTEC Industrieofenbau GmbH, Guggenberg 15-17, Kirchham A-4656 Germany

Modern melting devices for Mg-Alloy for die casting industry (Hot and Cold Chamber) – consisting of preheating, automatic charging, melting and gas protection for the molten Mg. In particular about accurate and safe dosing of Mg-Alloy.

Materials Processing Fundamentals: Smelting & Refining II

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Princewill N. Anyalebechi, Grand Valley State University, Padnos School of Engineering, Grand Rapids, MI 49504-6495 USA; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Tuesday PM Room: 3001
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Prince N. Anyalebechi, Grand Valley State University, Padnos Sch. of Engrg., Grand Rapids, MI 49504-6495 USA

2:30 PM
Heat Pipe Cooling of a Slag Tapblock: *Pietro Navarra*[1]; Hujun Zhao[1]; Frank Mucciardi[1]; Daniel Cheret[2]; Peter Verguts[2]; Karel Verscheure[3]; [1]McGill University, Metals & Matls. Engrg., 3610 Univ. St., Wong Bldg., Rm. 2160, Montreal, Quebec H3A 2B2 Canada; [2]Umicore Research, Dept. Pyro-Metall., Kastelstraat 7, Olen B-2250 Belgium; [3]K.U. Leuven, Afd. Chemische Materiaalkunde, Kasteelpark Arenberg 44, Heverlee B-3001 Belgium

The proximity of operators to copper tapholes in the metallurgical industry has caused a drive to seek an improved cooling mechanism vis-à-vis forced convection water cooling, which can fail catastrophically in the unlikely event of a failure. The purpose of the research presented is to develop an industrial-scale copper tapblock using novel heat pipe technology, which is fundamentally safer than conventionally cooled tapblocks. A tapblock cooling system incorporating two independent heat pipes was designed and assembled at Umicore Research in Belgium, each containing a total of 4 kg of water. Pilot tests achieved a heat load of 140 kW into a single heat pipe, with the potential for an 80% reduction in cooling water requirements compared to a forced convection system. Modeling indicates a maximum local heat extraction rate of 2.4 MW/m^2, with no indication of film boiling. The dry-out limitation was reached during testing and was subsequently circumvented.

2:55 PM
Modelling for Viscosities of Imperial Smelting and Lead Smelting Slags: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Mount Isa Mines Limited, Metallurgl. Plants Bldg., Private Mail Bag 6, Mt. Isa, Queensland 4825 Australia

A program 'SlagVis' has been developed to simulate the viscosities of Imperial Smelting slag and lead smelting slag, combined with a thermodynamic database and the mathematical model of slag viscosities based on a modified Urbain formalism. The modelling results have been validated by a number of measurements. The effects of slag compositions on the liquidus temperature and viscosities of Imperial Smelting slag have been simulated and discussed.

3:20 PM Cancelled
Investigation on the Synthesis of Titanate Barium in Molten NaOH-KOH by the A. C. Impedance Technique

3:45 PM
Preparation of Monodispersed NiO Precursor Particles by Induced Precipitation of Solid Oxalate Acid: *Huang Kai*[1]; Guo Xueyi[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

By adding the solid oxalate acid powder into the solution of nickel chloride or its ammonia coordinated solution, the uniform compound particles were prepared and the precipitation particles were transferred to be mono-dispersed NiO particles by calcination. In the study, the various experimental conditions were investigated and the characteristics of the corresponding obtained particles were analyzed, and the formation mechanism of the particles was elucidated. It was found that the solid oxalate acid provided the solid-solution interface for the precipitation and induced the nucleation and growth of the product particles.

4:10 PM Break

4:25 PM
Thermodynamic Modeling of Desilverising of Lead Bullion by Zinc During Lead Refining: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Mount Isa Mines Limited, Metallurgl. Plants Bldg., Private Mail Bag 6, Mt. Isa, Queensland 4825 Australia

A thermodynamic model and database has been developed to model the desilverising of lead by the reagent zinc during lead refining, whereby silver-zinc intermetallic compounds is formed, and floats out of the dissolved zinc/silver in the lead bath. The model predictions have been validated by the industrial data. The solubility of silver-zinc intermetallic compounds in lead decreases with falling temperature. A number of parameters of the Davey desilverising process are modeled, and applied to a wide range of silver contents in lead bullions in the industry.

4:50 PM
Optimization of the Pig Iron Desulfurization Inside a Torpedo Car by Physical Modeling Techniques: *Varadarajan Seshadri*[1]; Ildeu Alves Souza[2]; Carlos Antonio Silva[3]; Itavahn Alves Silva[3]; Versiane Albis Leão[3]; Diego Canez Fernandes[3]; [1]Universidade Federal de Minas Gerais, Metallurgl. Engrg., Rua Espirito Santo, 35, Belo Horizonte, Minas Gerais Brazil; [2]Belgo Mineira, Joâo Monlevade, Minas Gerais Brazil; [3]Universidade Federal de Ouro Preto, Escola de Minas/Engrg. Metalúrgica, Campus do Morro do Cruzeiro, Ouro Preto, Minas Gerais 35400-00 Brazil

A 1:6 torpedo car model was built in order to assess the refining conditions during pig iron desulfurization. The industrial conditions

are those used at Belgo-Mineira, João Monlevade, Brazil. At this plant the refining is made by injection of a CaO-CaF2-Mg powder. Similarity was accomplished by following criteria devel-oped from Froude and turbulent Reynolds groups. Mixing times were evaluated by pulse tracer addition and the kinetics of metal/slag interactions by assessing the iodine transfer from kerozene to water. The influence of parameters such as level of metal, gas flowrate, lance penetration, lance orientation was evaluated.

5:15 PM
Synthesis of Uniform Cobalt Oxalate Particles in the Micro-Fluid Tubular Reactor: *Huang Kai*[1]; Guo Xueyi[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

Micro-fluid tubular reactors with the inner diameter of 2mm and 3mm were used to synthesis the cobalt oxalate particles from ammonia oxalate and cobalt chloride solutions. Influences of the characteristic size of the tubular reactor, residence time, and reaction temperature on the particle properties were studied. SEM, XRD, TG-DTA, and Size analyzer were used to characterize the particles. The results of this study is helpful for the preparation of uniform particle with high value products obtained from precipitation process.

Mechanical Behavior of Thin Films and Small Structures: Fatigue, Fracture, and Reliability of MEMS and Thin Structures I

Sponsored by: Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Nanomechanical Materials Behavior

Program Organizers: Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA; Brad L. Boyce, Sandia National Laboratories, Materials and Processes Sciences Center, Albuquerque, NM 87185 USA; Evan Ma, Johns Hopkins University, Department of Materials Science & Engineering, Baltimore, MD 21218 USA; Andrew Minor, Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Berkeley, CA 94720 USA; Christopher L. Muhlstein, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Judy A. Schneider, Mississippi State University, Department of Mechanical Engineering, Mississippi State, MS 39762 USA

Tuesday PM Room: 2024
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Brad L. Boyce, Sandia National Laboratories, Dept. 1851, Albuquerque, NM 87185 USA; Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA

2:00 PM Invited
A Mechanistic Understanding of Fatigue in Polysilicon Structural Thin-Films: *Robert O. Ritchie*[1]; Daan Hein Alsem[1]; Eric A. Stach[2]; Christopher L. Muhlstein[3]; [1]University of California, Matls. Sci. & Engrg., One Cyclotron Rd., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, One Cyclotron Rd., Berkeley, CA 94720 USA; [3]Pennsylvania State University, Matls. Sci. & Engrg., 310 Steidle Bldg., Univ. Park, PA 16802 USA

While bulk silicon is not susceptible to fatigue failure, silicon micron-scale structural thin films are. This has important implications for microelectromechanical systems (MEMS) applications, where parts are subjected to a large number of loading cycles with amplitudes below their (single-cycle) fracture stress, which may arise due to vibrations intentionally induced in the structure (i.e. a resonator) or those which arise from the service environment. While the system-reliability of MEMS has received increasing attention, the physical mechanisms responsible for these failure modes have yet to be conclusively determined. This is particularly true for fatigue, where the mechanisms have been subject to intense debate. Our past studies have shown that fatigue is a result of a "reaction-layer" process, whereby high stresses induce a thickening of the post-release oxide at stress concentrations - such as the root of a notched cantilever beam - which subsequently undergoes moisture-assisted cracking. More recent results obtained in high vacuum (~2.0×10^{-7} mbar) reveal absolutely no evidence of premature fatigue failures (i.e. no fatigue failure in vacuo and no oxide thickening). Moreover, fatigue tests conducted in ambient air on polysilicon samples from a more recent fabrication run confirm that the fatigue behavior and oxide-layer thicknesses observed in earlier experiments were not an artifact of that particular fabrication run. We believe that both of these results add further confirmation to the "reaction-layer" mechanism for fatigue of micron-scale polysilicon.

2:25 PM
Long-Term Reliability of Single-Crystal Silicon Thin Films: The Influence of Environment on the Fatigue Damage Accumulation Rate: O. N. Pierron[1]; *C. L. Muhlstein*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg. & the Matls. Rsch. Inst., 310 Steidle Bldg., Univ. Park, PA 16803 USA

The fatigue behavior of single-crystal silicon thin films tested in various environments (300.1 °C at 251, 401 or 501% relative humidity (R.H.), medium vacuum, and medium vacuum with pre-bake) and fully-reversed loading conditions at high frequency (~40 kHz) was investigated in this study. The fatigue characterization structures, consisting of a notched cantilever beam attached to a plate-shaped mass, were electrostatically actuated at resonance. The resonant frequency was periodically monitored, and its evolution during cycling could be used for interpretation of fatigue damage accumulation. Particularly, damage accumulation rate and therefore fatigue life were strongly affected by both relative humidity and stress amplitude. Fracture surface examination showed highly localized, distinct features in long-life fatigued specimens tested in both air and vacuum that were not found in short-life fatigued specimens. The reaction-layer fatigue mechanism, implying a process of sequential, mechanically-induced oxidation and environmentally-assisted cracking of the surface oxide layer, appears to be the most appropriate to fit this experimental evidence.

2:40 PM
An Electron Microscopy Study of Wear in Polysilicon Microelectromechanical Systems: *Daan Hein Alsem*[1]; Eric A. Stach[2]; Michael T. Dugger[3]; Robert O. Ritchie[1]; [1]University of California, Matls. Sci. & Engrg., One Cyclotron Rd., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, One Cyclotron Rd., MS 72/150, Berkeley, CA 94720 USA; [3]Sandia National Laboratories, Matls. & Process Scis. Ctr., PO Box 5800, Albuquerque, NM 87185 USA

Wear is an important failure mechanism in microelectromechanical systems (MEMS) and is heavily affected by the tribological properties of the structural materials used. The most widely used material in MEMS is silicon, which is a brittle material. Because of the use of mostly ductile materials in macro-scale mechanical applications, a majority of bulk wear models focus on wear in these type of materials and can therefore not automatically be used for MEMS reliability. Surface characterization of silicon structural thin films has received a growing amount of attention and some cautious attempts to propose micron-scale wear models have been made. However, the exact physical mechanisms for micron-scale wear in silicon have yet to be conclusively determined. We have used polycrystalline silicon side-wall test specimens in combination with electron microscopy to study active mechanisms in sliding wear. In-situ experiments in the scanning electron microscope were performed, as well as transmission electron microscopy (TEM) of worn MEMS parts. After running the devices, worn parts were prepared for TEM using a dual-beam focused ion beam system. Analytical TEM was performed on wear debris and worn parts to investigate their morphology and microstructural changes. Debris particles were observed varying in size from below 100 nm to greater than 600nm and consist completely of silicon-dioxide. Furthermore, both ploughing wear tracks and surface cracks perpendicular to the wear direction were found, suggesting that fracture and fatigue of surface asperities could play an important role.

2:55 PM Invited
Fatigue Failure of Metal Thin Films in MEMS: *Oliver Kraft*[1]; [1]Forschungszentrum Karlsruhe, Inst. für Materialforschung II, Postfach 3640, Karlsruhe 76021 Germany

Continuous and patterned metal thin films with thicknesses well below 1 µm are widely used in micro-electro-mechanical systems (MEMS). Applications range from reflective coatings in micro-optics to current carrying metallizations in sensors or actuators. In these applications, thin film materials are often stressed in a cyclic manner with loading frequencies ranging from well below 1 Hz, e.g. related to usage cycles, up to GHz, e.g. in communication devices. In this paper, the effect of fatigue failure as a result of mechanical or thermomechanical cyclic loading conditions will be reviewed. Damage has been observed to consist of extrusions, voids, and cracks where details of the damage morphology depend on loading conditions, e.g. frequency and temperature, as well as film thickness and microstructure. Further, a general trend has been found that fatigue lifetime increases with decreasing film thickness. The insights gained from

these studies into lifetime prediction and mechanism of fatigue in thin film devices will be discussed.

3:20 PM
Nanoscale Wear Testing of LIGA Nickel Samples: *Neville R. Moody*[1]; John M. Jungk[2]; Marion S. Kennedy[3]; Soumari V. Prasad[4]; Thomas E. Buchheit[4]; David F. Bahr[3]; William W. Gerberich[2]; [1]Sandia National Laboratories, PO Box 969, MS9409, Livermore, CA 94551-0969 USA; [2]University of Minnesota, Minneapolis, MN 55455 USA; [3]Washington State University, Pullman, WA 99164 USA; [4]Sandia National Laboratories, Albuquerque, NM 87158 USA

Strength, friction, and wear are dominant factors in the performance and reliability of materials and devices fabricated using nickel based LIGA and silicon based MEMS technologies. However, the effects of frictional contacts and wear are not well-defined. We have therefore begun a program employing nanoscratch and nanoindentation on electrodeposited LIGA nickel samples. Nanoscratch techniques were used to generate wear patterns as a function of load and number of passes. Nanoindentation was then used to measure properties in each wear pattern. The results show a systematic increase in hardness with applied load with surprisingly little effect of repeat passes on sample deformation. In this presentation, we will use measured hardness values and Johnson's cavity model for plasticity to show how flow stress and the extent of plasticity evolve under sliding contacts. The authors gratefully acknowledge the support of Sandia National Laboratories under contract DE-AC04-94AL85000.

3:35 PM Break

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Fatigue of Electrodeposited Nickel Films: M. C. Zapata[1]; A. Minor[2]; *C. L. Muhlstein*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg. & Matls. Rsch. Inst., 310 Steidle Bldg., Univ. Park, PA 16803 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, Berkeley, CA USA

The fatigue resistance of the metallic thin films used in microelectronic circuits, microelectronic packages, and microelectromechanical systems (MEMS) can be a fundamental limitation to product performance. However, our fundamental understanding of how metallic thin films degrade under fatigue loading conditions is limited. In this work the stress-life (S/N) fatigue behavior of a 25mm-thick electrodeposited nickel film was evaluated by mechanical testing of subsized samples under tension-tension loading conditions. The fracture surfaces and dislocation substructure of the failed samples were then evaluated using electron microscopy. The ultrafine grained (i.e., grain sizes <100 nm) nickel exhibited extraordinarily high strength (s_{ult} >1.5 GPa) and its fatigue resistance was consistent with the trends for nickel films established in the literature. The implications of the electron microscopy observations in understanding the fatigue mechanisms will be discussed.

4:05 PM
Roles of Surface Oxide and Morphology in LIGA Ni Fatigue Behavior: Yong Yang[1]; *Jikou Zhou*[1]; S. Allameh[1]; W. O. Soboyejo[1]; [1]Princeton University, Dept. of MAE & PRISM, E-Quad, Olden St., Princeton, NJ 08540 USA

Fatigue life of LIGA Ni MEMS materials has been reported by several groups, but the failure mechanism has never been investigated for such single-layer film materials. In this work, we studied the effects of surface oxide scales on fatigue behavior. We found that surface oxide layer retards crack initiation by blocking the accessibility of water vapor to dislocation slip bands. Film surface morphology evaluation was also studied using AFM, and was found to play an important role in the film fatigue failure.

4:20 PM Invited
Mechanical Properties of Microscale Materials for MEMS Applications: *Yakaichi Higo*[1]; Kazuki Takashima[1]; [1]Tokyo Institute of Technology, P&I Lab., 4259 Nagatsuta, Midori-ku, R2-18, Yokohama 226-8503 Japan

For future application in micro/nano electro mechanical systems (MEMS/NEMS) devices, the elemental size for MEMS devices is thought to be 1/1000 to 1/10000 that of traditional structures. This down sizeing can causes many difficulties, called "Size Effects", which are described as follows, (1) Preparation Process, (2) Handling and Fixing, (3) Accuracy of Dimensions, (4) Accuracy of Measurement Method, (5) Surface Effect, (6) Materials Strengthening Methods and (7) Mechanical Properties. In order to understand these size effects and to overcome the difficulties associated with them, it is essential to develop mechanical testing methods for micro-sized specimens in order to obtain fundamental information for the design of durable and reliable MEMS devices, and to allow comparison of materials of different origin and the processing methods used to produce them. These methods can then be used to provide new strengthening methods to further advance the capabilities of MEMS devices.

4:45 PM
Application of General Weibull Statistics to MEMS: *I. Chasiotis*[1]; A. McCarty[1]; [1]University of Virginia, Mechl. & Aeros. Engrg., PO Box 400746, Charlottesville, VA 22904-4746 USA

The Weibull statistics have been used extensively to describe strength data for uniformly stressed MEMS specimens. In this study, we investigate the relevance of Weibull statistics to more general situations of MEMS-scale specimen failure. The applicability of the Weibull cumulative function to describe failure of (a) specimens with a single flaw distribution and variable geometry and (b) multiple flaw populations and a specific geometry is studied. In the first case, we examine the potential of Weibull statistics to provide a universal description for failure of specimens with non-uniform cross-sections. The uniaxial tension specimens tested and analyzed in this work included variable size perforations at the center of their gauge section, which allowed for twelve combinations of stress intensity factor and radii of curvature. The strength data for each of these geometries were used to calculate the material scale parameter and the Weibull modulus. The approach followed here took into account the non-uniform stress state resulting from the perforations in each specimen through a finite element model and the use of the general integral form for the Weibull function. The computed material scale parameter and the Weibull modulus allow for a general description of brittle failure for MEMS devices with different stress distributions, which can be further used in design and reliability of miniature components with complex geometries. In the second case, we investigated polysilicon MEMS-scale specimens that simultaneously possessed two different flaw populations, both of which were responsible for failure. The microtension specimens characterized in this investigation possessed one flaw population introduced by Reactive Ion Etching (RIE) during surface micromachining and a second population that was the effect of a surface treatment conducted during post-processing. The study also included specimens with the first of the two flaw populations only, which allowed the effects of the second source of failure to be isolated. The data show that the application of Weibull statistics with the assumption of a single flaw population is not appropriate. Instead, the failure behavior of the specimens with two flaw populations is better described by bi-Weibull statistics to incorporate the contribution of both flaw populations to the material strength. Such a statistical treatment provides insight into the origin of material failure and supports more reliable predictions for component failure.

5:00 PM Invited
The Use of Atomic Force Microscopy to Study Crack Tips in Glass: *S. M. Wiederhorn*[1]; J.-P. Guin[1]; [1]National Institute of Standards and Technology, Matls. Sci. & Engrg. Lab., Gaithersburg, MD 20899-8500 USA

Crack tips in glass are difficult to resolve by most techniques. Transmission electron microscopy is not feasible because glass is amorphous and shows no contrast. Optical techniques are not useful because the surface separation is of the order of nanometers for considerable distances from the actual tip of the crack. In this study we used the Atomic Force Microscope to resolve crack tips in glass specimens subjected to high stresses below the static fatigue limit. Experiments were conducted by first propagating a crack in glass and then reducing the applied stress intensity factor to a value, K_{Ih}, that is less than or equal to the expected fatigue limit, and holding it for a period of time. The load was then increased to the original stress intensity factor and the time measured to restart the crack. After breaking the fracture specimen in two, the "upper" and "lower" fracture surfaces were mapped and compared using atomic force microscopy. Fracture surfaces matched to an accuracy of better than 1 nm normal to the fracture plane and 5 nm within the fracture plane. Displacements between the upper and lower fracture surfaces that developed after a critical holding time were independent of distance from the crack tip, but increased with holding time, approaching an upper limit of about 25 nm for very long times. Despite the surface displacement, cracks tips appeared to be sharp. Results are discussed in terms of a hydronium ion-alkali ion exchange along the crack surfaces and corrosion of the glass surface near the crack tip by hydroxyl ions.

5:25 PM
Fracture Strength of Silicon Carbide Microspecimens Versus Weibull Size Effect Predictions: *N. N. Nemeth*[1]; O. M. Jadaan[2]; M. A. Trapp[3]; W. N. Sharpe[4]; G. D. Quinn[5]; G. M. Beheim[6]; [1]NASA Glenn Research Center, Life Prediction Branch, Cleveland, OH 44135-3191 USA; [2]University of Wisconsin, Math. & Sci., Platteville, WI 53818 USA; [3]Carnegie Mellon University, Dept. of Mechl. Engrg., Pitts-

burgh, PA 15213 USA; [4]Johns Hopkins University, Dept. of Mechl. Engrg., Baltimore, MD 21218 USA; [5]National Institute of Standards & Technology, Ceram. Div., Gaithersburg, MD 20899 USA; [6]NASA Glenn Research Center, Sensors & Elect. Tech. Branch, Cleveland, OH 44135 USA

Notched and un-notched micro-sized silicon carbide tensile specimens were fabricated in order to investigate the effect of stress concentration on the strength. Measured strengths in the polycrystalline material were typically below 1 GPa with a Weibull modulus of about 3. A size effect whereby the notched specimens were stronger than the un-notched specimens was observed. Fractographic examination showed the source of failure to be a combination of etching grooves along the specimen side-walls and large sized grains. Strength results were compared to predictions based on the Weibull distribution by using the NASA CARES/Life code. CARES/Life over-predicted the strength of the notched specimens versus the un-notched specimens (the size effect) but correctly predicted that some specimens would fail in the grip section. Correlation of predicted strength compared to experimental results improved when tighter dimensional control was achieved.

5:40 PM
A Numerical Analysis of Flexure Induced Cylindrical Cracks During Indentation of Thin Hard Films on Soft Substrates: Sampath K. Vanimisetti[1]; *R. Narasimhan*[1]; [1]Indian Institute of Science, Dept. of Mechl. Engrg., Bangalore 560012 India

Thin hard films deposited on soft substrates are employed in engineering applications where mechanical loads like those due to contact are experienced (for example, in cutting tools). It is important to understand the mechanics of fracture of these films in order to design better coating-substrate systems. Recent experimental work has shown that during spherical indentation, circumferential cracks may form outside the contact zone on the film surface or at the film-substrate interface directly underneath the indenter. The latter occur because the film experiences bending due to the contact load and behaves as a thin plate resting on a plastic foundation. The objective of this paper is to examine the behavior of these flexure induced cracks. To this end, finite element analyses of spherical indentation of a thin TiN film perfectly bonded to a steel substrate and containing circumferential (cylindrical) cracks as mentioned above are performed. The film is taken to be linear elastic while the substrate obeys an elastic-plastic constitutive model with linear strain hardening. The results show that when the crack length is small, predominantly mode I conditions prevail due to the flexure stresses near the interface. As the crack length increases, the mode mixity gradually changes from mode I to mode II, for cracks located away from the indenter axis. It is observed that the crack growth process is stable up to a crack length of about a third of the film thickness and thereafter becomes unstable. However, due to the presence of a strong compressive radial stress field near the contact zone, the mode I component of loading vanishes for crack length greater than 60% of the film thickness leading to crack closure and arrest. Finally, the role of the substrate yield strength on the above issues is systematically investigated.

Metallurgical Technology for Waste Minimization: Session I

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; Toru Okabe, University of Tokyo, Institute of Industrial Science, Tokyo Japan; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA

Tuesday PM Room: 2012
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Toru Okabe, University of Tokyo, Tokyo Japan; Ji-Whan Ahn, Korea Institute Geoscience & Mineral Resources Korea

2:30 PM Invited
Electrochemical Copper Removal from Semiconductor Industry Waste Streams: Ran Ding[1]; *James W. Evans*[1]; Fiona M. Doyle[1]; [1]University of California, Matls. Sci. & Engrg., Berkeley, CA 94720 USA

In the semiconductor industry copper is replacing tungsten and aluminum metallization for devices. That poses new challenges in waste-water treatment. Specifically, the manufacturing of current microprocessors generates waste from both the copper electroplating solution and the copper CMP/post-CMP cleaning processes. Traditionally, the concentrated depleted electrolyte is treated separately and there has been little work on the recovery of copper from the dilute waste solutions. Our project is aimed at recycling copper and water from all the waste solutions by a combination of ion-exchange and electrodeposition. Our first-stage work has been to examine the effects of typical electrolyte additives on copper deposition kinetics by the use of a rotating disc electrode. A few results from cells using porous carbon electrode for copper removal are also described. We have also conducted research on using the electrochemical quartz crystal microbalance (EQCM) to determine the adsorption of certain additives on the copper crystal surface. Finally, a fluidized bed electrode has been built and its performance in copper deposition is described. Research supported by US Environmental Protection Agency.

3:00 PM Invited
A Hydrometallurgical Process for Recovery of Vanadium and Molybdenum from Wasted Catalysts for Hydrotreating Desulfurization of Heavy Oil: *Toshiaki Akaboshi*[2]; Yoshiaki Umetsu[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsch. for Advd. Matls., 1-1 Katahira 2-chome, Aoba-ku, Sendai, Miyagi 980-8577 Japan; [2]YSK Techno-system, Kamisu, Ibaraki Japan

The fundamental reactions have been investigated in order to develop a hydrometallurgical process for recovery of molybdenum and vanadium from wasted catalysts generated in the hydrotreating desulfurization of heavy oil. The principal steps of the process are a mild oxidation roasting of the waste catalysts at temperature around 500°C, leaching of the target metal components with dilute ammoniacal aqueous leachates, selective precipitation of vanadic acid, and ammonium molybdate precipitation. The selective recovery ofvanadium as vanadic acid is successfully performed using ammonium sulfate as a salting-out agent. Recovery of ammonium molybdate is based on large dependence of solubility of ammonium molybdate in ammoniacal solutions upon temperature and ammonium salt concentration. In this process, the solution after recovery of vanadium and molybdenum is recycled to the leaching stage of the roasted materials, and the ammonium salts in the process liquor is in the repeated use. Employment of ammoniacal system makes it possible to operate the roasting at much lower temperature than that for the currently operating process.

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Assessment of Costs and Revenues for an Electronic Waste Materials Recovery Facility: *Hai-Yong Kang*[1]; Julie M. Schoenung[1]; [1]University of California, Cheml. Engrg. & Matls. Sci., One Shields Ave., 3118 Bainer Hall, Davis, CA 95616 USA

At a Materials Recovery Facility (MRF) electronic waste (e-waste) can become marketable output products including resalable systems/components, and recyclable materials such as plastics and metals. The recovery process can be divided into four basic unit operations; sorting/testing, dismantle, size reduction and separation by materials. A spreadsheet model has been developed to estimate the costs and revenues in each unit operation. Input costs include machine costs, labor costs and recycling fees for CRTs, and output streams include revenues from resale systems/component, and recovery of scrap metals and plastics. By estimating the costs and revenues for each unit operation, the model calculates the total processing costs and revenues in operating an MRF that handles e-waste. Sensitivity analysis is used to validate the modeling results. Furthermore, critical cost and revenue drivers are identified, and the viability of utilizing an MRF to handle both current and future types and volumes of e-waste is evaluated.

4:05 PM
Static and Dynamic Synergistic Effects for Ni(II) Extraction and the Application to Waste Solution Containing Ni(II): *Junji Shibata*[1]; Yoshinori Motoda[1]; Norihiro Murayama[1]; Hideki Yamamoto[1]; [1]Kansai University, Cheml. Engrg., 3-3-35, Yamate, Suita, Osaka 564-8680 Japan

Waste solution from the Ni non-electric plating process contains 4.0kg/m3 Ni (II), 0.1kg/m3 Fe (III), 0.01kg/m3 Zn (II), 48kg/m3 SO42-, 98kg/m3 HPO32- and 31kg/m3 lactic acid as a typical composition. Solvent extraction, cementation, ion exchange resin and precipitation methods may be used for the treatment of this kind of solution. In this study, solvent extraction of Ni (II) is investigated by using two kinds of extractants in order to clarify the relationship between extraction equilibrium (static synergistic effect) and extraction rate (dynamic synergistic effect) for several extractant mixtures. The static synergistic effect does not take place significantly for the extractant mixtures of acidic extractants and neutral organphosphorous compounds, while a large static synergistic effect is confirmed when the extractant mixtures of acidic extractants and nitrogen-containing

compounds are used. Nickel ions can be extracted in the wide pH range of 3.0-8.0 without any pH adjustment by using the several extractant mixtures. At the same time, the extraction rate remarkably increases by using the extractant mixtures of acidic extractants and nitrogen-containing compounds. The mechanism for static and dynamic synergistic effects was investigated. It is possible to extract Ni (II) from artificial waste liquor using a mixture of D2EHPA and LIX860 without pH adjustment. Ni (II) can be stripped with 0.1 mol/dm3 sulfuric acid from the Ni (II)-loading mixtures of D2EHPA and LIX860. The recovery process of Ni (II) from the waste solution in the Ni non-electric plating was proposed from the results of this research.

4:25 PM
Analysis of Extraction Rate of Phosphorous Acid by Tri-n-Octylamine in Toluene: *Yoshinobu Kawano*[1]; Yasuo Hatate[2]; Koichiro Shiomori[1]; Takashi Sana[1]; Hideshi Tanaka[1]; [1]Miyazaki University, Applied Chmst., 1-1 Gakuen-KIbanadai, Miyazaki 889-2192 Japan; [2]Kagoshima University, Chem & Chem. Engrg., Korimoto, Kagoshima 890-0065 Japan

Phosphorous acid presented in the bath of electroless nickel-plating reduce the efficiency of nickel plating. Solvent extraction process is designed to remove the acid in bath. Extraction rate of phosphorous acid by Tri-n-octyl amine in toluene was investigated using a Lewiss type transfer cell having a stagnant interface. The dependencies of concentrations on the rates were analyzed considering the interfacial reaction steps and extraction equilibrium constants for the system. The results for the extraction rate in vibro mixer type extractor having small droplets formed in mixing of fluids were analyzed by the rate equation derived in Lewiss type transfer cell. The mass transfer rates constants was enlarged about 3x103 times of that in Lewiss type transfer cell. We propose the application of the extraction for phosphorous acid by tri-n-octylamine in vibro mixing extractor.

4:45 PM
Solvent Extraction of Precious Metals Using Thiodiglycolamide: *Hirokazu Narita*[1]; Mikiya Tanaka[1]; Kazuko Morisaku[1]; [1]National Institute of Advanced Industrial Science and Technology, Rsch. Inst. for Environml. Mmgt. Tech., 16-1 Onogawa, Tsukuba, Ibaraki 305-8569 Japan

Since precious metals are in extensive use for catalysts, electronic devices and so on, the recovery of precious metals from their wastes has recently received much attention. Especially, the separations of precious metals by solvent extraction are one of the most important studies on their recycling. In this study, we have investigated the extraction properties of some precious metals (Au(III), Pd(II), Pt(IV) and Rh(III)) and base metals (Fe(III), Cu(II) and Zn(II)) from hydrochloric acid solution using the N,N'-dimethyl-N,N'-dioctylthiodiglycolamide (MOTDA). The extraction percentages of Au(III) and Pd(II) using 0.05 M MOTDA are almost 100% over a wide HCl concentration range, while the other metals are hardly extracted. Additionally, the extraction of Pd(II) is extremely fast.

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Iron Removal From Titanium Ore Using Selective Chlorination and Effective Utilization of Chloride Wastes: *Ryosuke Matsuoka*[1]; Toru H. Okabe[2]; [1]University of Tokyo, Dept. of Matls. Engrg., Grad. Sch. of Engrg., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656 Japan; [2]University of Tokyo, Internatl. Rsch. Ctr. for Sustainable Matls., Inst. of Industl. Sci., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8505 Japan

This study investigated iron removal from titanium ore using selective chlorination, and effective utilization of chloride wastes generated from titanium production process (Kroll process). The thermodynamic analyses of the chlorination reactions in the Ti-Fe-O-Cl system were carried out prior to the experimental work, and the conditions for chlorination experiments were optimized. The iron in the Ti ore was selectively chlorinated by reacting low-grade Ti ore and metal chloride at 1100 K under a nitrogen atmosphere, and low-iron Ti ore and iron chloride (FeClx) were obtained. Chlorine in the FeClx produced by selective chlorination was recovered as TiCl4 by reacting FeClx and metallic titanium at 1100 K under an argon atmosphere. This recycling process investigated in this study, which utilizes low-grade ore or chloride wastes, is useful because the Ti scrap and chloride wastes are expected to increase. This process has the potential for developing a new environmentally sound chloride metallurgy.

5:40 PM
Process Modelling of Rotary Kiln Treating EAF Dust: *Pengfu Tan*[1]; Pierre Vix[1]; [1]Mount Isa Mines Limited, Metallurgl. Plants Bldg., Private Mail Bag 6, Mt. Isa, Queensland 4825 Australia

Electric arc furnace (EAF) dust from the steel industries is listed by EPA as a hazardous waste under the regulations of the Resource Conservation and Recovery Act. The disposal of the approximately 650,000 tons of EAF dust per year in the U.S. and Canada is an expensive and unresolved problem for the majority of steel companies. Waelz process has been considered as the best process treating EAF dust. A process model, combined thermodynamic modelling, chemical kinetics with heat transfer, has been developed to simulate the chemical reactions, mass and heat transfer and heat balance in the kiln. The injection of air into the slag and the temperature profile along the kiln have been modelled. The effect of (CaO+MgO)/SiO2 on the solidus temperature of slag has also been modelled and discussed. Some optimising results have been presented in this paper.

Micromechanics of Advanced Materials II (Symposium in Honor of James C.M. Li's 80th Birthday): Microstructure and System Stability

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Fuqian Yang, University of Kentucky, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA; C. C. Chau, Pactiv Corporation, Canandaigua Technology Center, Canandaigua, NY 14424 USA; Sung Nee George Chu, Multiplex Inc, South Plainfield, NJ 07080 USA; M. Ashraf Imam, Naval Research Laboratory, Materials Science & Technology Division, Washington, DC 20375-5343 USA; Teh-Ming Kung, Eastman Kodak Company, Rochester, NY 14650 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; B. B. Rath, Naval Research Laboratory, Materials Science and Component Technology Directorate, Washington, DC 20375-5341 USA

Tuesday PM Room: 3000
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: R. J. Arsenault, University of Maryland, Dept. of Matl. Sci. & Engrg., College Park, MD 20742 USA; Changxu Shi, National Natural Science Foundation of China, Beijing 100085

2:00 PM Invited
Negative Creep of Metallic Glasses as an Externally-Catalysed Dissipative Structure Within Ginzburg-Landau Kinetics: *J. S. Kirkaldy*[1]; [1]McMaster University, Brockhouse Inst. for Matls. Rsch., Hamilton, ON L8S-4M1 Canada

The process to which negative creep is applied concerns annealing temperatures of metallic glasses approaching ambient where normal densification is negligible, but negative dimensional change is dramatically catalysed as a response to a LeChatelier-like load perturbation and initially accelerated against the load by temperature and load increase. This obtains on the isotherm up to the load point where normal creep in the opposite direction must begin to counter the anomalous effects. The proposed dissipative structure, originally regarded by J.C.M. Li as analogous to uphill spinodal decomposition, is based upon strain as a global average order parameter satisfying the time-dependent Ginzburg Landau (TDGL) equation and more generally following the experimentally verified Ostwald Step Rule which subsumes autonomous selection for near-reversibility, minimum dissipation and equivalent high free energy configuration en route to equilibrium. This self-organizing phenomenon can be conceived as a continuous stroke, potential energy-increasing heat engine driven by the quenched-in thermal energy. It is remarkable in that a significant part of the stored free energy is strictly macroscopic, where the usual TDGL reactions such as order-disorder, clustering, Ostward Ripening and grain growth the unused free energy is microscopic or mesoscopic. The Ginzburg-Landau analogy which comes to mind is Type II superconductivity under irreversible flux creep where the conserved free energy corresponding to minimum dissipation is the macroscopic kinetic energy of the Cooper pairs which constitute the supercurrent. In this case the externally applied magnetic field plays the thermodynamic role of the applied stress field. This analogy is suggestive of the possibility that the incredible molecular collaboration involved in negative creep ultimately has a quantum mechanical explanation in much the same way that classical semiconductor theory transforms to a more rigourous quantum mechanical version.

2:25 PM Invited
Thermodynamic Stability of Co/Cu Multilayered Nanostructures: W. W. Cao[1]; Y. Yang[1]; J. Zhu[1]; W. A. Oates[2]; *Y. A. Chang*[1]; [1]University of Wisconsin, Dept. Matl. Sci. & Engrg., Madison, WI 53706 USA; [2]University of Salford, Inst. for Matls. Rsch., Salford M5 4WT UK

When the size of a system decreases to the nanoscale, interfacial energy becomes significant. We can no longer ignore the contribution of the interfacial energy to the total free energy of the system. In deed a recent study suggested that intermixing occurs in multilayered nanostructures of Co/Cu at temperatures lower than the critical temperature for the bulk case. In this presentation, we report the simulation results as a function of the thickness of the metal layers using several thermodynamic models, the regular solution model, the pair approximation of the Cluster Variation Method (CVM) and the cluster/site approximation (CSA). In all these models, the results show that decreases in the thickness of the layer lower the critical temperatures.

2:50 PM Invited
Energy Balances and Stability of Electro-Capillary Systems: *S. J. Burns*[1]; [1]University of Rochester, Mechl. Engrg., Matls. Sci. Prog., Rochester, NY 14627-0133 USA

Wetting between a dielectric liquid and a flat conducting surface is analyzed using energy balances. The wetted surface area, the interfacial surface energy, the voltage, charge, temperature and entropy are considered the state variables in the system. Reversible, equilibrium mechanics are used to obtain the voltage - charge relations on isothermal lines of constant interfacial surface energy. Nonlinear, charged systems are included in these general energy balances. Most frequently, the voltage is linear with charge, i.e., a linear capacitor. For this example, experimental observations shows that the electrostatic energy release rate is proportional to the voltage squared and thus AC or DC voltages create equal driving forces for these systems. The stability of linear systems for a parallel plate capacitor when the capacitance changes linearly with wetted surface area, is considered. This system is neutrally stable at constant voltage, and absolutely stable at constant charge. Measurements establish that the voltage squared is proportional to the interfacial surface energy's driving force using charge control while the charge added to the system is proportional to the total wetted surface area. It is experimentally demonstrated that system stability allows wetting in electro-capillary systems so systematic volumes of liquid can be delivered in micro-liter units. Applications to ink-jet printers and drug delivery systems are explored.

3:15 PM Invited
The Long-Term Stability of Electroplated Thin Films: *Paul T. Vianco*[1]; Garry Bryant[1]; Gary Zender[1]; Paul F. Hlava[1]; [1]Sandia National Laboratories, PO Box 5800, MS0889, Albuquerque, NM 87185-0889 USA

Electroplated Au films are used with 50In-50Pb (wt.%) solder for level 2 interconnections. Solid-state reactions form the AuIn2 intermetallic compound (IMC) layer at the Au/solder interface. Rate kinetics were measured for thin film Au/In-Pb solder couples having contaminated and non-contaminated Au layers. The AuIn2 layer growth was described by A(t^n) exp(Q/RT). The time exponent, n was equal to one and showed no dependence upon Au layer contamination. The value of Q (50 - 70 kJ/mol) was slightly higher for the non-contaminated Au layer. Formation of the AuIn2 layer was not detrimental to interface integrity. However, contaminants in the Au layer caused voids at the AuIn2/Au interface that degraded adhesion strength. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Dept. of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

3:40 PM Break

3:45 PM Invited
Kinematic and Dynamic Characterization of Plastic Instabilities Occurring in Nano- and Microindentation Tests: *Chinh Quang Nguyen*[1]; András Juhász[1]; Gyõzõ Horváth[1]; György Bérces[1]; János Lendvai[1]; [1]Eotvos University, Dept. of Gen. Physics, Pázmány P. sétány 1/A., Budapest 1117 Hungary

This paper surveys the phenomenon of plastic instabilities occurring in depth sensing indentation measurements, during which a stepwise increase has been observed in the indentation depth vs. load (d-F) curves measured in constant loading rate mode, indicating hardness-oscillations around a nearly constant value of the conventional dynamic microhardness. These oscillations are correlated with Portevin-Le Chatelier type plastic instabilities starting from the contact surface between the sample and the indenter head. Taking into account the experimental observations, a macro-mechanical dynamic model was also proposed for the characterization of indentation instabilities. The characteristics of serrated flow - plastic instabilities - in constant loading rate indentation are compared with those often-observed in conventional, uniaxial compression and tensile tests.

4:10 PM
Stress-Induced Surface Instability of Cylindrical Layer: *Wei Song*[1]; *Fuqian Yang*[1]; [1]University of Kentucky, Chem. & Matls. Engrg., Lexington, KY 40506 USA

The lateral surface stability of a cylindrical layer subjected to a constant uniaxial stress σ_0 and frictionless condition between the layer and cylindrical rigid substrate was analyzed by using the theory of linear elasticity and thermodynamics. The mechanism controlling the surface instability was the gradient of chemical potential associated with surface energy and elastic energy. A new dispersion equation describing the surface evolution was derived. It was found that surface instability depends on the elastic parameter $(2\sigma_0^2 r_1)/(\gamma E)$, Poisson's ratio and film thickness h_0, where r_1 is the radius of the substrate, γ is surface tension, E is Young's modulus and h_0 is the initial thickness of film. For the thin films $h_0/r_1 \to 0$, the critical frequency and the maximum-growth frequency are inversely proportional to the square root of film thickness, while for thick films $h_0/r_1 \to \infty$, they are independent of the thickness.

4:30 PM
Surface Evolution of Crystalline Tubes: Fuqian Yang[1]; *Wei Song*[1]; Jun Zhang[2]; [1]University of Kentucky, Dept. of Cheml. & Matls. Engrg., Lexington, KY 40506 USA; [2]University of Kentucky, Dept. of Computer Sci., Lexington, KY 40506 USA

The surface evolution of an annular tube has been established on the basis of lattice diffusion and linear stability analysis. Without surface disturbance the annular tube will shrink to reduce the surface energy while the cross-sectional area of the tube remains constant. For an annular tube having infinitesimal thickness, the time dependence of the tube radius follows a linear law. When surface energy is significant, a new dispersion relation describing the morphological stability of crystalline tubes due to longitudinal surface perturbation has been formulated. A criterion has been obtained on the dependence of perturbation growth rate on perturbation frequency. The perturbation will grow when the perturbation frequency is less than the critical frequency, which is equal to the inverse of the inner surface radius. To our surprise, the critical frequency for an annular tube of infinitesimal thickness is the same as that given by Nichols and Mullins [Trans. Metall. Soc. AIME 233(1965) 1840] for an infinitely cylindrical rod. A finite spatial frequency for maximum growth rate was also obtained, which depends on the ratio of the inner surface radius to the outer surface radius. The surface instability will lead to the formation of closed end of crystalline tubes.

4:50 PM
Surface Morphology Evolution in Thin Films Via Diffusional Creep: *K. Jimmy Hsia*[1]; [1]University of Illinois, Theoretl. & Applied Mech., 111B Talbot Lab., MC 262, 104 S. Wright St., Urbana, IL 61801 USA

Deformation mechanisms involving mass transport by stress driven diffusion influence a large number of technological problems. We study the formation of undulations on surfaces of stressed films at high temperature by exploring the deformation kinetics governed by volume and surface diffusion. A governing equation is derived that gives the amplitude change of such surfaces as a function of time. A parametric study is then carried out using a range of practically important input values of the film material properties. The results show that at the dominant instability wavelength, under high average stresses (GPa range), only surface diffusion contributes to film surface morphology evolution; whereas under low stress and high temperature conditions, both surface diffusion and volume diffusion contribute to film surface morphology evolution. Furthermore, the contribution of volume diffusion depends on the sign of the film stress, with compressive stress promoting surface roughening while tensile stress promoting surface smoothing.

5:10 PM
Micromechanics Analysis of Elastic Strain and Ferroelectric Domain Texture in Tetragonal PZT Ceramics: *Tsutomu Mori*[1]; David Anthony Hall[1]; Hans Kungl[2]; Philip J. Withers[1]; [1]University of Manchester, Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK; [2]University of Karlsruhe, Inst. for Ceram. in Mechl. Engrg., Karlsruhe D-76131 Germany

We demonstrated previously that high energy X-ray diffraction provides a useful technique for determining the elastic strain of a grain 'family' in a poled ferroelectric ceramic as a function of its orientation Ψ relative to the macroscopic polar axis. Specifically, it was

shown that the elastic strain normal to {111} planes and the ferroelectric domain texture, measured by the intensity ratio $I_{(002)}/(I_{(002)}+I_{(200)})$, both exhibit a linear dependence on $\cos^2\Psi$. In the present paper, these observations are analysed on the basis of a simple micromechanical model, which makes use of the unique characteristics of the {111} planes in tetragonal ferroelectrics i.e. that the {111} lattice spacing of a grain is independent of the internal ferroelectric domain population in the 'free' (unconstrained) state. In this approach, individual grains are modelled as spherical inclusions within a surrounding polar matrix, which are characterised in terms of the transformation strain (eigenstrain) associated with the ferroelectric domain fractions. The analysis successfully explains the linear dependence of the elastic strain and the ferroelectric domain texture in a quantitative manner. The results obtained using high energy synchrotron XRD for a range of tetragonal PZT ceramics, having a systematic variation in chemical composition and tetragonality, are evaluated and compared with their macroscopic polarisation-electric field and strain-electric field relationships.

TUESDAY PM

Microstructural Processes in Irradiated Materials: Microstructure Evolution and Segregation

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Tuesday PM Room: 3011
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Brian Cockeram, Bechtel Bettis Laboratory, W. Mifflin, PA 15122-0079 USA; Yuri Osetsky, Oak Ridge National Laboratory, Computer Sci. & Math., Oak Ridge, TN 37831 USA

2:00 PM
In-Situ TEM Analysis of FePt Ordering Reactions and Grain Growth Via Ion Irradiation: *Nicholas William Morgan*[1]; Gregory B. Thompson[1]; Robert C. Birtcher[2]; [1]University of Alabama, Metallurgl. & Matls. Engrg., Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]Argonne National Laboratory, Matls. Sci. Div., 9700 S. Cass Ave., MSD/212, Argonne, IL 60439-4838 USA

We report the use of in-situ TEM analysis of 10 nm and 100 nm thick FePt films irradiated with 500 keV Kr^+ ions. FePt is a potential material for next-generation magnetic recording. When processed, FePt adopts an A1 phase requiring an anneal to order to L10. Irradiation has been proposed to reduce the ordering temperature and maintain small grains. The as-deposited films, grown on SiO, were amorphous and crystallized at room temperature at dosages < $1x10^{14}$ ions/cm^2 with minimal grain growth. The films were then simultaneously annealed and irradiated. Onset of long-range ordering occurred at ~300°C. At elevated temperatures and dosages of ~10^{14} ions/cm^2, the 100 nm film exhibited rapid grain coarsening where as the 10 nm film dewetted and formed an interconnected island pattern. We will address how ion irradiation dosage and temperature can be used to engineer the phase as well as grain morphology of FePt nanostructures.

2:20 PM
Unique Defect Processes at Irradiation-Assisted Stress Corrosion Crack Tips: *Edward P. Simonen*[1]; Larry E. Thomas[1]; Stephen M. Bruemmer[1]; [1]Pacific Northwest National Laboratory, Matls. Div., PO Box 999, Richland, WA 99352 USA

Concurrent point defect processes affect alloy composition in advance of a crack tip during irradiation-assisted stress corrosion cracking (IASCC). Crack-tip corrosion can affect solute redistribution in addition to radiation-induced solute segregation because IASCC occurs at the intersection of the alloy matrix, alloy grain boundary and the aqueous solution. Selective dissolution or oxidation can promote vacancy injection and affect equilibrium concentrations at crack-tip grain boundaries. Competition between radiation-induced segregation, radiation-enhanced diffusion and corrosion-induced vacancy injection has been quantified for conditions relevant to austenitic stainless steels in light-water reactor environments. Corrosion-induced vacancy injection has the greatest potential for changing crack-tip alloy compositions during IASCC propagation. Measured composition changes at crack tips are compared to calculated changes for light-water-reactor component relevant temperatures, doses and dose rates. This work was supported by the Materials Sciences Branch, Office of Basic Energy Sciences and the Office of Nuclear Energy, Science and Technology, U.S. Department of Energy, under Contract DE-AC06-76RLO 1830.

2:40 PM
Phosphorus Interaction with Point Defects in bcc Fe: Electronic Structure Calculation Contribution: Christophe Domain[1]; *Charlotte S. Becquart*[2]; [1]EDF R&D, MMC, Les Renardières, Moret sur Loing F-77250 France; [2]Universite de Lille I, Lab. de Métall. Physique & Génie des Matériaux - UMR CNRS 8517, Bat C6 - 2eme étage, Villeneuve d'Ascq Cedex F-59655 France

The main effect of phosphorus in steels is the possible enhanced embrittlement at grain boundaries. Consequently, the segregation of P under irradiation is an important issue, and furthermore P may influence the evolution of the microstructure under irradiation. The modelling of the P behaviour requires the knowledge of the interaction with point defects created under irradiation, and the possible transport mechanisms. In this work, these interactions have been investigated using ab initio density functional theory. The interactions of P with vacancies and interstitial atoms have been determined and found to be significant. The consequences of these large interactions are discussed.

3:00 PM
Theoretical Investigation of Phosphorus Segregation to Grain Boundaries in Alpha-Iron Under Irradiation: *Alexander V. Barashev*[1]; [1]University of Liverpool, Dept. of Engrg., Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK

In pressure vessel steels of nuclear reactors, a big enhancement of the concentration of phosphorus (P) atoms at grain boundaries (GBs) is observed and this leads to a decrease in the GB cohesion and to a shift of the ductile-to-brittle transition temperature. In this paper we present the results of molecular dynamics and Monte Carlo studies of phosphorus atom diffusion in bcc Fe-P alloy in the dilute limit. An EAM type potential set developed by Ackland et al. (2004, J. Phys.: Condens. Matter 16) is used. The diffusion coefficients of point defects and P atoms are evaluated. The rate theory analysis of the P segregation under irradiation conditions is presented. Possible effect of carbon atoms on the P segregation in steels is also discussed.

3:20 PM
Effect of Hafnium Addition on Radiation-Induced Inter-Granular Segregation in Ferritic Steel: Zheng Lu[1]; *Roy G. Faulkner*[1]; N. Sakaguchi[2]; H. Kinoshita[2]; H. Takahashi[2]; Peter E.J. Flewitt[3]; [1]Loughborough University, Inst. of Polymer Tech. & Matls. Engrg., Ashby Rd., Loughborough, Leicestershire LE11 3TU UK; [2]Hokkaido University, Ctr. for Advd. Rsch. of Energy Tech., N. 13, W. 8, Sapporo 060-8628 Japan; [3]Magnox Electric, Berkeley Ctr., Berkeley, Gloucestershire GL13 9PB UK

9%Cr ferritic steels with and without hafnium (Hf) addition were irradiated by 250 keV nickel-ions at 300°C in a ion accelerator attached to the Multi-beam High Voltage Electron Microscopy (ARM1300) to study the influence of Hf addition on radiation-induced segregation in ferritic steel. Grain boundary segregations of phosphorus, silicon, chromium and molybdenum were measured by field emission gun transmission electron microscopy with an energy dispensive analyser. The results show that radiation induces the enrichment of undersized atoms (P, Si) and the depletion of oversized atoms (Cr, Mo) in the materials without Hf addition. The addition of Hf suppressed radiation-induced undersized atom enrichment and oversized atom depletion. A radiation-induced non-equilibrium segregation model is developed to predict radiation-induced oversized atom depletion at grain boundary. The microstructural factors, such as dislocation density, grain size, grain boundary misorientation, and stress effect are taken into consideration in the model. Effect of hafnium on freely migrating defect population is discussed and estimated. The predicted results show a good agreement with experimental data.

Microstructural Processes in Irradiated Materials: Poster Session

Sponsored by: Structural Materials Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Brian D. Wirth, University of California, Department of Nuclear Engineering, Berkeley, CA 94720-1730 USA; Charlotte S. Becquart, Ecole Nationale Superieure de Chimie de Lille, Laboratoire de Metallurgie Physique et Genie des Materiaux, Villeneuve d'Ascq cedex 59655 France; Hideki Matsui, Tohoku University, Institute for Materials Research Japan; Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA

Tuesday, 4:00-6:30pm Room: 3011
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: David Hoelzer, Oak Ridge National Laboratory, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; Arthur Voter, Los Alamos National Laboratory, Theoretl. Div., Los Alamos, NM 87545 USA

An MEAM Interatomic Potential for the Fe-Cu Alloy System and Cascade Simulation on Pure Fe and Fe-Cu Alloy: *Byeong-Joo Lee*[1]; Junhyun Kwon[2]; Sang Chul Kwon[2]; Jun-Hwa Hong[2]; [1]Pohang University of Science and Technology, Dept. of Matls. Sci. & Engrg., Pohang 790-784 Korea; [2]Korea Atomic Energy Research Institute, Nucl. Matls. Tech. R&D Team, Taejon 305-353 Korea

The microstructural changes occurring during irradiation are essential factors of radiation effects on materials properties, and can be well investigated using atomistic simulation approaches such as molecular dynamics or Monte Carlo simulation. To be able to predict the changes of materials properties more correctly, important is that the interatomic potentials for the atomistic simulation should be more reliable ones and be applicable to practical multicomponent alloy systems. The MEAM interatomic potentials are highly applicable to alloy systems because wide range of elements can be described using a common formalism. In the present study, an interatomic potential for the Fe-Cu binary system was developed using previously developed MEAM potentials of Fe and Cu. The procedure of parameter optimization and transferability of the potential will be presented. Some results of cascade simulations on pure Fe and Fe-0.5at%Cu alloy will also be presented.

Atomic Scale Modelling of the Primary Damage State of Irradiated fcc and bcc Nanocrystalline Metals: *Maria Samaras*[1]; Peter Michael Derlet[1]; Helena Van Swygenhoven[1]; Maximo Victoria[2]; [1]Paul Scherrer Institut, Villigen, Argau 5232 Switzerland; [2]Lawrence Livermore National Laboratory, Livermore, CA USA

Experimental studies of ion irradiated nanocrystalline materials have revealed that that the high density of grain boundaries present in these materials can affect the final damage production. Further differences are seen in defects produced by the different metallic lattice structures, with small vacancy clusters and voids forming in bcc Fe and stacking fault tetrahedra predominately forming in higher energy irradiation of fcc Ni. In the nanocrystalline regime there exists a lengthscale overlap (around the 50nm grain size scale), where experiments and computation are comparable. Large scale molecular dynamics computer simulations of the irradiation of nanocrystalline fcc Ni and bcc Fe with grains sizes in a range of 6- 50nm are presented. The resultant primary damage state after irradiation is discussed in terms of grain boundary structure and grain size, and how these microstructural parameters can affect the primary damage state.

Molecular Dynamics Simulation of Displacement Cascades in α-Fe: A Review: *Lorenzo Malerba*[1]; [1]SCK-CEN, RMO, Boeretang 200, Mol 2400 Belgium

Molecular dynamics has been extensively used to simulate displacement cascades in iron, using different interatomic potentials. The results of this type available from the literature are compared in order to discuss up to what extent they are consistent with each other. It is found that, while generally the number of Frenkel pairs versus recoil energy is the same for most potentials, yielding a defect production efficiency in agreement with experimental estimates, large differences exist concerning the defect clustered fractions. In the case of self-interstitial atoms the criterion used to define clusters is largely responsible for the discrepancies, but sensible differences seem to exist also as a consequence of the different properties of the used interatomic potentials. Very little data have been published concerning vacancy clusters, but in this case too different potentials seem to provide different predictions. These differences may be significant and influence the outcome of, for example, kinetic Monte Carlo studies, depending on what set of input primary damage state is used.

Computer Simulation of Cascade Damage in Alpha-Iron: *Andrew F. Calder*[1]; David J. Bacon[1]; Alexander V. Barashev[1]; Yuri N. Osetsky[2]; [1]University of Liverpool, Dept. of Engrg., Brownlow Hill, Liverpool L69 3GH UK; [2]Oak Ridge National Laboratory, Computer Scis. & Math. Div., PO Box 2008, Oak Ridge, TN 37831-6158 USA

MD simulation has been applied widely to study displacement cascades in metals. Since it is necessary to use ~1M atoms, the interatomic potentials have simple form and there may be inconsistency between different potentials for the same metal. Iron is of concern because numerous simulations have used potentials for which the <110> dumbbell interstitial is only marginally stable over the <111> crowdion, whereas recent ab initio calculations, (e.g. Fu et al. (PRL 92 (2004)), show that the difference in energy of these defects is ~0.7eV. To investigate this, cascades have been simulated using a new potential (Mendelev et al., Phil. Mag. 83 (2003)) for which the energy difference is close to the ab initio value. A large number of cascades of different energy have been simulated for temperature up to 600K to allow statistical treatment of the number of point defects, the fraction that cluster and cluster properties. Comparisons will be made with previous results.

Similarities and Differences of Point Detect Cluster Formation, Stability and Behaviour in Copper and α-Zirconium: *Roman E. Voskoboinikov*[1]; Yuri N. Osetsky[2]; David J. Bacon[1]; [1]University of Liverpool, Matls. Sci. & Engrg., Dept. of Engrg., Brownlow Hill, Liverpool, Merseyside L69 3GH UK; [2]Oak Ridge National Laboratory, PO Box 2008, MS-6138, Oak Ridge, TN 37831 USA

Atomic-scale simulation of displacement cascades in two metals with close-packed crystalline structures, namely copper (fcc) and α-zirconium (hcp), has been conducted for a wide range of temperature 100 K ≤ T ≤ 900 K and primary knock-on atom (PKA) energy 5keV ≤ E_{pka} ≤ 25 keV. Our study of more than 700 displacement cascades is the largest yet reported for both metals. Using four different identification techniques we identified point defects and their clusters at the end of simulation and carried out comprehensive statistical treatment of the results. For both metals the number of Frenkel pairs and fraction of point defects in clusters versus temperature and PKA energy were obtained. Cluster yield per cascade was also evaluated for both vacancy and self-interstitial atom (SIA) clusters. Typical vacancy and SIA clusters arising in displacement cascades were classified, and the dependence of their mean size on simulation parameters was found. Similarities and distinctions in cluster variety, structure, mobility, thermal stability and transformations in the two metals were investigated in detail.

Copper Precipitates in Alpha-Iron and Their Interaction with Irradiation Produced Defects: *Alexander V. Barashev*[1]; David J. Bacon[1]; Alan C. Arokiam[1]; [1]University of Liverpool, Dept. of Engrg., Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK

Precipitation of copper atoms is one of the main reasons for the hardening increase observed in reactor pressure vessel steels during either ageing or irradiation. Theoretical treatment of the experimental data requires knowledge of the diffusion characteristics of copper atoms and interaction properties of copper atoms, vacancies and self-interstitial atom (SIA) clusters with copper precipitates. In this paper, the results of molecular dynamics and Monte Carlo studies of these interactions are presented. The equilibrium concentration of vacancies inside precipitates, the binding energy of a copper atom with a precipitate as a function of precipitate size, the binding energy of a SIA-cluster with a precipitate and transport coefficients are estimated. The calculations were performed using the many-body potential set of Ackland et al. (1997, Phil. Mag. A 75) and ab initio calculations by Becquart and Domain (2003, Nucl. Inst. Meth. B 202).

Mobility of Self-Interstitial Atom Clusters in bcc-Fe: A New Molecular Dynamics Study: *Dmitry A. Terentyev*[1]; Lorenzo Malerba[1]; Marc Hou[2]; [1]SCK-CEN, RMO, Boeretang 200, Mol 2400 Belgium; [2]Université Libre de Bruxelles, Physique des Solides Irradiés & des Nanostructures CP234, Bd du Triomphe, Bruxelles B-1050 Belgium

The mobility of self-interstitial atom (SIA) clusters, characterised by migration energy, prefactor as a function of size and dimensionality of the motion (1D versus 3D) is known to determine largely the microstructural evolution of irradiated metals. The ideal tool to study SIA cluster mobility is molecular dynamics (MD). Yet, so far the description of SIA provided for bcc-Fe by the empirical potentials used in MD studies was at variance with both experimental and ab initio data. Recently, an Embedded Atom Method (EAM) potential capable of

providing a clearly improved description of SIA in á-Fe has been proposed (Mendelev et al., Phil. Mag. 83(35), 2003, 3977 3994). This potential has been used in the present work for dynamical calculations of the diffusion coefficient of SIA clusters in bcc-Fe. The results of the study are expected to help in the definition of the parameter set for kinetic Monte Carlo simulations of radiation damage evolution in ferritic alloys.

Simulations of Elastic Electron Diffuse Scattering from Small Defects: Z. Zhou[1]; S. L. Dudarev[2]; *M. L. Jenkins*[1]; A. P. Sutton[1]; M. A. Kirk[3]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxfordshire OX1 3PH UK; [2]EURATOM/UKAEA Fusion Association, Theory & Modlg. Dept., Culham Sci. Ctr., Oxfordshire OX14 3DB UK; [3]Argonne National Laboratory, Matls. Sci. Div., Argonne, IL 60439 USA

We have recently shown that elastic diffuse scattering patterns obtained from single small defect clusters carry information both on the morphology and nature of the defect. Potentially this is an important new method for characterising small clusters. A possible drawback of the our earlier work was the use of kinematical diffraction theory for the simulation of the experimental patterns. A dynamical model of elastic electron diffuse scattering has now been used to calculate the distribution of diffuse scattered electrons in reciprocal space from small dislocation loops and stacking-fault tetrahedra. Simulations were carried out for similar conditions used in experiments to investigate the influence of experimental parameters such as deviation parameters, beam coherence, and the sample conditions, for example, crystal structures, sample thicknesses and the depths of defects. The dynamical and kinematical models will be compared and the validity of the kinematical model assessed. It is shown that the database built from simulated results according to various conditions is essential for the characterization of defects by experimental diffuse scattering patterns, and theoretical analysis and simulations are also very useful for optimizing the experiments.

Effect of the Internal Displacement Cascades Morphology on the Growth of Point Defect Clusters Under Irradiation: *Charlotte S. Becquart*[1]; Christophe Domain[2]; Lorenzo Malerba[3]; Marc Hou[4]; Roger E. Stoller[5]; A. Soudi[6]; [1]Universite des Sciences et Technologies de Lille, LMPGM, UMR 8517, Batiment C6, Villeneuve d'Ascq cedex 59700 France; [2]EDF-R&D Département MMC, Les renardières, Moret sur Loing Cédex F-77818 France; [3]SCK CEN, Reactor Matls. Rsch. Unit, Mol B-2400 Belgium; [4]Physique des Solides Irradiés, CP234, Université Libre de Bruxelles, Bd du Triomphe, Brussels B-1050 Belgium; [5]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S, MS-6138, PO Box 2008, Oak Ridge, TN 37831-6138 USA; [6]Centre Universitaire de Saida, BP138, En-nasr, Saida 20000 Algeria

The influence of the internal cascade structures on the long term evolution of the primary damage is demonstrated by an Object Kinetic Monte Carlo Method and using displacement cascades produced by molecular dynamics as input for long term damage prediction. Displacement cascades obtained in the binary collision approximation as well as random point defect distributions having all similar overall morphologies to the molecular dynamics cascades are used to determine which characteristics of the point defect distributions (spatial extension, the level of agglomeration, etc.) are the most important. Different interatomic potentials are used to generate cascades by MD, allowing the influence of the potential on the results to be discussed.

Kinetic Monte Carlo Simulation of Model Experiments in Iron Based Alloys: *Charlotte S. Becquart*[1]; Christophe Domain[2]; Lorenzo Malerba[3]; [1]Universite de Lille I, Lab. de Métall. Physique & Génie des Matériaux - UMR CNRS 8517, Bat C6 - 2eme étage, Villeneuve d'Ascq Cedex F-59655 France; [2]EDF R&D, MMC, Les Renardières, Moret sur Loing F-77250 France; [3]SCK-CEN Belgian Nuclear Energy Research Centre, Reactor Matls. Rsch. Unit, Boeretang 200, Mol B-2400 Belgium

Object kinetic Monte Carlo methods, based on elementary diffusion and reaction mechanisms, is a very powerful tool to simulate radiation damage and damage accumulation in various configuration (thin slabs, 3D box ?). Model experiments can be used to parameterise the OKMC codes and we have performed ion irradiation simulations of Fe based alloys remaining as close as possible to the model experiments. The purpose is to study the influence of environmental parameters such as temperature, irradiated specimen shape and chemical composition on the microstructure evolution. The model used to simulate the ion irradiation is discussed and the sensitivity of some key parameters is also evaluated.

Exact Kinetic Monte Carlo Simulations Without the Lattice Hops: *Vasily V. Bulatov*[1]; Wei Cai[2]; George Gilmer[1]; Tomas Oppelstrup[1]; Malvin Kalos[1]; Babak Sadigh[1]; [1]Lawrence Livermore National Laboratory, University of California, Livermore, CA 94550 USA; [2]Stanford University, Stanford, CA 94305 USA

This presentation will discuss a new algorithm for kinetic Monte Carlo simulations applicable to a wide range of physical situations where multiple Brownian particles of finite dimensions diffuse, collide and otherwise react with each other. In its spirit, the new approach is reminiscent of the so-called event-based Monte Carlo algorithm (JERK) developed over the years in SACLAY: similar to JERK, it alleviates the need to simulate every single diffusional hop but focuses on more significant changes in the system's configuration. Yet, unlike JERK, the new algorithm is approximation-free and its accuracy is limited only by the quality of the rate coefficients.

Monte Carlo Simulation of Point Defect Recombination During the Initial Stages of Cascade Aging in Fe: *Bor Kae Peter Chang*[1]; Brian D. Wirth[1]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA

Structural materials in nuclear energy applications are exposed to intense neutron fields that create atomic displacements leading to microstructural evolution and property changes. At the smallest scales, radiation damage is continually initiated with the formation of energetic primary knock-on atoms through collisions between high-energy neutrons and lattice atoms. Primary damage production in high-energy displacement cascades has been extensively studied by MD simulations and reveals intra-cascade recombination, spatial separation of vacancy and self-interstitial atoms (SIA), and SIA clustering. We have studied the subsequent recombination in the "early" stages of cascade aging as the SIA migrate through and away from the cascade volume. The simulations investigate the effect of PKA energy (from 500 eV to 100 keV), recombination radius (1 to 3 lattice parameters), one dimensional versus three dimensional SIA cluster migration, and temperature (50 - 500°C). The results indicate increasing recombination with cascade energy, which saturates for the highest energy cascades.

Modeling Cascade Aging and Dose Rate Effects in Dilute Fe-Cu Alloys: *Brian D. Wirth*[1]; Jae-Hyeok Shim[1]; G. Robert Odette[2]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA; [2]University of California, Santa Barbara, CA USA

Fundamental understanding of defect production in displacement cascades is required to model and predict long-term neutron irradiation induced microstructural evolutions. Defect production is generally treated in terms of primary events, occuring in cascades over time scales of less than 100 ps. We describe the development of advanced kinetic lattice Monte Carlo (KMC) methods to simulate the long-term rearrangement (aging) of displacement cascades as well as cascade aging effects on overall damage accumulation in neutron irradiated dilute Fe-Cu alloys. Special algorithms have been developed to model self-interstitial atom-vacancy recombination in cascades and long range point defect and solute diffusion. The simulations reveal the formation of a continuous distribution of three dimensional cascade vacancy-Cu cluster complexes and demonstrate the critical importance of spatial, as well as short and long-time, correlated processes, that mediate the effect of dose rate on microstructural evolution under conditions relevant to reactor pressure vessel embrittlment.

Kinetic Monte Carlo Simulation of Substitutional He Diffusion in Fe: *Brian D. Wirth*[1]; Jae-Hyeok Shim[1]; Rick J. Kurtz[2]; G. Robert Odette[3]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA; [2]Pacific Northwest National Laboratory, Richland, WA 99352 USA; [3]University of California, Santa Barbara, CA USA

Ferritic and martensitic steels will experience severe irradiation induced degradation of many important performance sustaining mechanical properties as well as potential dimensional instabilities in fusion environments, driven by simultaneous production of displacement defects and high concentrations of helium. Thus a key issue is the coupled transport and fate of all defect, gas and solute species, including the effects of radiation enhanced diffusion, segregation and cluster aggregation. As part of a broader multi-scale modeling effort, we present the results of kinetic Monte Carlo (KLMC) simulations of substitutional helium diffusion in iron, based on vacancy jump frequencies in the vicinity of substitutional helium. The jump frequencies are obtained from molecular statics simulations of the potential energy barriers based on semi-empirical Fe-He interatomic potentials. The KLMC results are compared to theoretical descriptions of substitutional solute diffusion in bcc alloys and are also extended to model the diffusion of small helium-multiple vacancy complexes.

Helium and Hydrogen Clustering in Radiation Damaged Iron Studied by Stochastic Simulations: *Chaitanya Suresh Deo*[1]; Michael I. Baskes[1]; Srinivasan G. Srivilliputhur[1]; Stuart Maloy[1]; Michael James[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los

Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, AAA-TDO, MS H809, Los Alamos, NM 87545 USA

Many micro-structural defects are introduced into materials upon irradiation with energetic particles. These defects can cause degradation of mechanical properties and contribute to material failure. Transmutation products such as hydrogen and helium in irradiated stainless steels may exert deleterious effects on material properties. A description of the atomic mechanisms governing the process and their correlation to material properties will result in better understanding of the mechanisms by which iron and iron-based alloys respond to helium and hydrogen implantation by radiation and will suggest methods of alloy improvement to withstand irradiation damage. We have performed kinetic Monte Carlo (KMC) simulations of point defect diffusion and clustering in bcc alpha iron. The model consists of the following entities in bcc iron: interstitial and substitutional helium and hydrogen atoms, interstitial iron atoms, vacancies, vacancy-clusters, and sinks for the trapping of point defects (dislocations and grain boundaries). Input to the simulations includes the migration energies of the point defects (interstitial iron, vacancy, interstitial and substitutional helium and hydrogen), formation energies of the HenVm clusters, dissociation energies of the point defects from the HenVm clusters and initial concentrations and configurations of point defects and defect ratios. These quantities are obtained from experimental data and molecular dynamics (MD) simulations using embedded atom and modified embedded atom potentials. The defect ratios and configurations can be obtained from the post-cascade data of large MD runs. We employ the KMC simulations to investigate the time evolution of the point defect configuration leading to defect clustering and bubble formation. The composition of embryonic defect clusters as a function of time and operating temperatures is determined. It is found that almost all the transmuted helium and hydrogen is trapped in sink configurations (clusters, dislocations or grain boundaries) within a fraction of a microsecond.

The Diffusion of He Atoms and Small He Clusters in Grain Boundaries in Alpha-Fe: *Fei Gao*[1]; Richard J. Kurtz[1]; Howard L. Heinisch[1]; [1]Pacific Northwest National Laboratory, Fundamental Sci., MS K8-93, PO Box 999, Richland, WA 99352 USA

A systematic study of the migration and diffusion mechanisms of He atoms and small He clusters in grain boundaries in α-Fe is presented. Two grain boundaries, the Π11<110> {323}, and the Π<110> {111}, were used for the current investigations. The migration of He atoms and small He clusters were followed from 10 to 30 ns, at temperatures between 400 and 1000 K. The diffusivity and self-diffusion coefficient of He atoms and small He clusters were obtained, and the effective migration energies were determined. Also, the lowest energy paths of He atoms and He clusters were traced out by the Dimer method. He atoms diffuse quickly in the grain boundaries at low temperatures with one-dimensional behavior, but a few directional changes were observed at higher temperatures. The different activation energies and diffusion mechanisms in these two representative grain boundaries suggests that the varying atomic structures of the grain boundaries are important for the diffusivity of He.

Atomistic Modeling of Helium Clustering and Diffusion Along Dislocations in Alpha-Iron: *Howard L. Heinisch*[1]; Fei Gao[1]; Edward A. Le[1]; Richard J. Kurtz[1]; [1]Pacific Northwest National Laboratory, Matls. Sci. Div., PO Box 999, P8-15, Richland, WA 99352 USA

An important first step in mitigating helium effects on the mechanical properties of metals is to understand the fate of helium with respect to its interaction with various microstructural features. Molecular statics, molecular dynamics and the dimer method of potential surface mapping are being used to study the fate of helium in the vicinity of a/2<111>{110} edge dislocations in alpha-iron. Interstitial He atoms can easily migrate to dislocations, where they are strongly bound (>2eV) in crowdion positions in the layer of atoms just above the slip plane. A He-divacancy complex can also migrate and be trapped near the core, but with a much lower binding energy (~0.5 eV). These complexes may be a primary mode for the collection of He and its transport along dislocations. Various aspects of the mobility, clustering, and stability of He-divacancy complexes in and near dislocations and their important role in He disposition will be discussed.

Helium Behavior in Metals Characterized by Thermal Helium Desorption Spectroscopy: *Stephen C. Glade*[1]; Brian D. Wirth[1]; Henk Schut[2]; [1]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720-1730 USA; [2]Delft University of Technology, Interfaculty Reactor Institute, Mekelweg 15, 2629 JB Delft The Netherlands

The behavior of helium in metals must be understood for their future use in fusion applications. In fusion reactors, the helium concentration in metals increases by direct implantation of helium or by nuclear transmutation reactions. Helium is insoluble in most metals alloys and precipitates in vacancy clusters and voids, forming helium bubbles at high concentrations, causing high-temperature intergranular embittlement. We present the design and initial performance of a thermal helium desorption spectrometer that we are constructing at the University of California, Berkeley. We plan to study microstructural effects on the helium behavior in iron. The information that can be obtained from analyses of the desorption spectra will be discussed.

Positron Annihilation Characterization of Nanostructural Features in High Nickel Copper Free Model Alloys: *Stephen C. Glade*[1]; Brian D. Wirth[1]; G. Robert Odette[2]; Michael K. Miller[3]; [1]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA; [2]University of California, Santa Barbara, CA USA; [3]Oak Ridge National Laboratory, Oak Ridge, TN USA

Irradiation embrittlement of reactor pressure vessel (RPV) steels results from formation of a high density of nm-scale precipitates. In RPV steels with >0.1%Cu the dominant hardening features are copper-rich precipitates (CRPs) alloyed with manganese, nickel and silicon. But as theoretically predicted long ago, manganese-nickel(-silicon) rich precipitates (MNPs) can form in both copper bearing and copper free alloys, containing large amounts of these elements. Large volume fractions of so-called late blooming MNPs (LBP), cause severe hardening and embrittlement. The presence LBP-MNPs and large hardening in low copper and copper free alloys has been demonstrated recently by a variety of techniques. We present positron annihilation spectroscopy results of neutron irradiated model alloys containing high nickel and low copper. The results provide insight into composition and magnetic properties of the MNPs. The positron results are compared to corresponding small angle neutron scattering, combined electricial resistivity-Seebeck coefficient and atom probe tomography data.

Positron Anihilation in Fe and Fe-Cu After Neutron Irradiation: *Abderrahim Almazouzi*[1]; [1]SCK.CEN, Reactor Matls. Rsch., LHMA, Boeretang 200, Mol 2400 Belgium

In the frame of the european project PERFECT, dealing with the modelling of irradiated materials. Well controlled irrdiations have been performed at 300°C for doses ranging from 0.05 to 0.2dpa. The materials that have been irradiated are ranging from pure Fe to technological steels. In this paper, the Lifetime and coincidence doppler measurements that have been performed on neutron irradiated pure Fe and Fe-Cu containing 0.1 and 0.3% will be reported. the results demonstrate the effect of Cu on reducing the matrix damage and enhancing the Cu-precipitation.

Apt Characterization of High Nickel RPV Steels: *Michael K. Miller*[1]; Mikhail A. Sokolov[1]; Randy K. Nanstad[1]; Kaye F. Russell[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, MS 6136, Oak Ridge, TN 37831-6136 USA

The microstructures of high and low copper reactor pressure vessel steels with high nickel contents have been characterized in the local electrode atom probe to investigate the influence of high nickel levels on the response to neutron irradiation. Atom probe tomography revealed nickel-, manganese-, and silicon-enriched precipitates in VVER 1000 base (0.05% Cu,1.26% Ni, 0.46% Mn) and weld (0.07% Cu,1.78% Ni, 0.80% Mn) materials after neutron irradiation. A high number density of copper-, nickel-, manganese-, silicon- and phosphorus-enriched precipitates were observed in the Palisades reactor weld (0.20% Cu, 1.20% Ni, 1.27% Mn) after neutron irradiation. Research at the Oak Ridge National Laboratory SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the U. S. Department of Energy.

Precipitation in Neutron Irradiated Fe-Cu-Mn-Ni Model Alloys: *Michael K. Miller*[1]; Kaye F. Russell[1]; Robert Odette[2]; Brian D. Wirth[3]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, MS 6136, Oak Ridge, TN 37831-6136 USA; [2]University of California, Dept. of Cheml. Engrg., Santa Barbara, CA 93106 USA; [3]University of California, Nucl. Engrg. Dept., Berkeley, CA 94720-1730 USA

A series of Fe-Cu-Ni-Mn model alloys have been characterized by atom probe tomography and small angle neutron scattering to determine whether any precipitation occurs in low (0.05%) and copper-free alloys during neutron irradiation and to determine the influence of nickel. These alloys contained 0, 0.05 or 0.1% Cu, 0.8 or 1.6% Ni and 1.6% Mn and were neutron irradiated to a fluence of ~1.3 x 10^{23} n m^{-2} (E > 1 MeV) at a temperature of 290°C. After irradiation, precipitates were detected in all alloys. Some 1-2 nm diameter precipitates were

enriched in Ni and Mn and some were enriched in Cu, Ni and Mn. Copper-enriched precipitates were observed in the 0.05% Cu alloys indicating that 0.05% Cu is above the solubility limit under these irradiation conditions. The Cu, Ni and Mn atom distributions were not located at the same center-of-mass in agreement with Monte Carlo simulation predictions. Research at the Oak Ridge National Laboratory SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the U. S. Department of Energy. Research at UCSB was sponsored by the U. S. Nuclear Regulatory Commission NRC-04-01-064.

Characterization of Precipitation in MA/ODS Ferritic Steels: *Michael K. Miller*[1]; Kaye F. Russell[1]; David T. Hoelzer[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6136 USA

Mechanically alloyed, oxide dispersion strengthened (MA/ODS) ferritic steels exhibit excellent high-temperature creep and tensile properties. These ODS ferritic steels are attractive for fusion reactor applications because of their potential for higher operating temperatures and also because the dispersed oxide particles may provide a trap for helium. These ferritic alloys are fabricated by mechanically alloying a pre-alloyed Fe, Cr, Y and Ti powder with a small amount of yttria powder. Atom probe tomography has revealed that these MA/ODS alloys contain a high number density of nanometer scale Ti-, Y- and O-enriched particles in the as-processed condition. These particles were found to be extremely resistant to coarsening at temperatures up to 1300°C (0.85 Tm). Research at the Oak Ridge National Laboratory SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

Irradiated Microstructure of Alloy 800H: *Jian Gan*[1]; James I. Cole[1]; Todd R. Allen[2]; Shuttha Shutthanandan[3]; Suntharampillai Thevuthasan[3]; [1]Argonne National Laboratory, Nucl. Tech., PO Box 2528, Idaho Falls, ID 83403 USA; [2]University of Wisconsin, Dept. of Engrg. Physics, 1500 Engrg. Dr., Madison, WI 53706 USA; [3]Pacific Northwest National Laboratory, Environml. Molecular Sci. Lab., 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

Alloy 800H has the same basic composition as INCOLOY alloy 800 (Fe-20Cr-32Ni) but with significantly higher creep-rupture strength. It is one of the high temperature candidate alloys being considered for the Generation IV nuclear reactor system. The radiation resistance of 800H has not previously been studied. This work provides information on the microstructural changes in 800H after irradiation using 5.0 MeV Ni ions at 500°C to 5 and 50 dpa. Following irradiation, changes in microstructure and phase stability were studied using transmission electron microscopy. At the dose of 50 dpa, no voids were found and the density and size of the faulted loops were measured to be 2.3x1016 cm-3 and 8.4 nm, respectively. There are fine precipitates distributed in 800H with an average size approximately 6 nm and a density greater than 9.1x1015 cm-3. The high Ni content and the presence of precipitates are believed to be responsible for the resistance to void formation at dose up to 50 dpa.

Damage Accumulation Under Cascade Damage Conditions with Specific Emphasis on the Evolution of Stacking Fault Tetrahedra: *Stanislav Ilijch Golubov*[1]; Bachu N. Singh[2]; Helmut Trinkaus[3]; Steve J. Zinkle[1]; Roger E. Stoller[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bathel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6138 USA; [2]Risø National Laboratory, Matls. Rsch. Dept., Roskilde DK 4000 Denmark; [3]Forschungszentrum Jülich, Inst. Für Festkörperforschung, Jülich D-2425 Germany

By now it is well established that clusters of vacancies and self-interstitial atoms (SIAs) are produced directly in the displacement cascades. Furthermore, some of these SIAs clusters diffuse one-dimensionally and occasionally may change their direction of diffusion. In addition, they and in addition may diffuse transversely by conservative climb. While diffusing, these clusters are likely to interact with all other defects and their clusters present in the crystal. Over the years, these features of the primary damage production and the ensuing consequences have been incorporated in the production bias model (PBM). During the last ten years or so, various aspects of defect accumulation and microstructural evolution under cascade damage conditions have been treated both analytically and numerically within the framework of the PBM. However the impact of the evolution of stacking fault tetrahedra (SFTs) on damage accumulation in FCC metals, has so far not been subjected to a serious and systematic theoretical treatment. Recently the available experimental data on the evolution of SFTs under irradiation and aging have been reviewed. The review suggests that interactions of SIAs and their clusters with SFTs, transformation of SFTs into Frank loops and direct impingement of cascades/subcascades on existing SFTs may play significant role in determining the evolution of SFTs under different irradiation conditions. These aspects must be taken into account in the theoretical treatment of the microstructural evolution and this is main objective for the present work. The results thus obtained will be implemented to further calculations, for example, of the temperature dependence of microstructural evolution in FCC metals.

The Influence of Fast Neutron Irradiation and Irradiation Temperature on the Tensile Properties of Wrought LCAC and TZM Molybdenum: *Brian V. Cockeram*[1]; Richard W. Smith[1]; Lance L. Snead[2]; [1]Bechtel Bettis Laboratory, PO Box 79, ZAP 05R/MT, W. Mifflin, PA 15122-0079 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN USA

Molybdenum alloys are generally known to be susceptible to embrittlement following neutron irradiation at temperatures < 800C, which can restrict the broad use of Mo-base alloys in nuclear applications. However, irradiation of molybdenum-base alloys at higher temperatures to relatively low neutron fluence (<60 X 10^20 n/cm^2 (E>0.1 MeV)) can result in little radiation hardening and no embrittlement1. The undesireable changes in the mechanical properties of molybdenum alloys following irradiation can be affected by altering the microstructure and base composition. These variables are evaluated in this work by the irradiation of commercially available wrought Low Carbon Arc Cast (LCAC) and TZM molybdenum in the High Flux Isotope Reactor (HFIR) at 300C, 600C, and 1000C to neutron fluences between 10.5 to 200 X 10^20 n/cm^2. The wrought LCAC sheet has a fine grain size and a high carbon to oxygen ratio, which results in high levels of ductility at room-temperature prior to irradiation. Wrought TZM molybdenum also has a fine grain size with coarse carbides and titanium + zirconium in solid solution that results in higher non-irradiated strength. The change in the Ductile to Brittle Transition Temperature (DBTT), which is based on tensile properties and failure mode, was used as the basis for quantifying radiation embrittlement. Irradiation at 300C is shown to result in evaluation of the DBTT from a pre-irradiated value of -100C to a post-irradiated value of 800C for both LCAC and TZM with a similar increase in fracture stress for both alloys (91% to 25% increase). Irradiation at 600C also resulted in a comparable increase in fracture stress for both alloys (90% to 47% increase), but the post-irradiated DBTT for LCAC (300C) was much lower than observed for TZM (700C). Irradiation of both LCAC and TZM at 935-1100C resulted in little radiation hardening (15% to 0% increase in yield strength) and approximately a -50C DBTT for both LCAC and TZM molybdenum. The finer grain size, low oxygen content, high carbon to oxygen ratio, and absence of coarse carbides may explain the slightly improved embrittlement resistance for LCAC compared to TZM at the 600C irradiation temperature. The post-irradiated fracture modes are shown to correlate with the tensile properties of LCAC and TZM molybdenum. 1. B.V.Cockeram, J.L.Hollenbeck, and L.L.Snead, J. Nuclear Materials, 324 (2004) pp. 77-89.

Strain-Rate Effects on Microstructural Deformation in Irradiated 316 SS: *James I. Cole*[1]; Hanchung Tsai[2]; Todd R. Allen[3]; Tsunemitsu Yoshitake[4]; Naoaki Akasaka[4]; Ichiro Yamagata[4]; Yasuo Nakamura[4]; [1]Argonne National Laboratory, Nucl. Tech. Div., PO Box 2528, Idaho Falls, ID 83403 USA; [2]Argonne National Laboratory, Energy Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [3]University of Wisconsin, Engrg. Physics, Coll. of Engrg., Engrg. Rsch. Bldg., 1500 Engrg. Dr., Madison, WI 53706 USA; [4]Japan Nuclear Cycle Development Institute, Fuels & Matls. Div., Oarai Engrg. Ctr., Oarai-machi, Ibaraki 311-1393 Japan

A series of studies have been performed to investigate the post-irradiation deformation and failure behavior of 12% cold worked 316 stainless steel following irradiation to variety of doses and temperatures. In the current phase of the study, three sets of samples with distinctly different radiation-induced microstructures have been characterized with transmission electron microscopy following tensile testing to failure at a "fast" strain-rate (1 x 10^{-3} s^{-1}) and a "slow" strain-rate (1 x 10^{-7} s^{-1}). The samples were tested at 430°C. The influence of the defect microstructures and strain-rate on deformation and failure behavior is discussed. In particular, mechanisms that might lead to transitions in deformation mode from heterogeneous to localized are analyzed with respect to the irradiation and testing temperatures.

Deformation Mode Maps of Irradiated 316 Stainless Steels in True Stress-Dose Space: *Thak Sang Byun*[1]; Naoyuki Hashimoto[1];

Kenneth Farrell[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6151 USA

Deformation modes in type 316 stainless steels after low temperature neutron and ion irradiation have been mapped into the true stress-irradiation dose coordinate system. In the irradiated type 316 stainless steels various deformation microstructure features, such as dislocation tangles and pileups, dislocation channels, stacking faults, twins, and martensite particles, have been observed depending on irradiation and test conditions. With increasing radiation dose the deformation microstructure changed progressively from a dislocation network-dominant to a stacking fault/twin band-dominant or to a dislocation channel-dominant microstructure. Much of this variety in deformation microstructure is believed to result from the low stacking fault energy of the austenitic steels. It was found that the most important external factor for controlling the deformation microstructure was the true stress. In the mapping deformation modes, therefore, the boundaries were drawn by stress-based criteria for deformation modes; the stress criteria for twinning, for channeling, and for macroscopic plastic instability have been proposed and applied in the mapping. Indeed, irradiation hardening and interactions between dislocations and radiation-induced defects plays an important role in determining the deformation mode at high doses; the helium ion-irradiated specimens showed a tendency for twinning due to the non-shearable gas bubbles, while the neutron-irradiated specimens experienced heavy dislocation channeling due to the removable defect clusters. This study also showed the possibility that the stress-based mapping can be expanded to other conditions with minimal experimental efforts.

Molecular Dynamics Simulation of Screw Dislocation Motion in bcc Fe: *Christophe Domain*[1]; Ghiath Monnet[1]; [1]EDF R&D, Dept. MMC, Les Renardieres, Moret sur Loing F-77250 France

At low temperature, the plasticity of steels is controlled by the motion of screw dislocations. Screw dislocation in bcc Fe is studied by classical molecular dynamics using different embedded atom method potentials for Fe. The motion of the screw dislocation under applied strain is presented. It operates through a double kink mechanism. The results are compared to experimental data obtained in extremely pure iron in order to assess the quality of the cohesive models used. Preliminary results regarding the interaction between the screw dislocation and small defects formed under irradiation such as void or interstitial loops is also presented.

Brittle-Ductile Transition in Ferritic Steels Subjected to Irradiation: A Discrete Dislocation Simulation: *Silvester J. Noronha*[1]; Nasr M. Ghoniem[1]; [1]University of California, Mechl. & Aeros. Engrg., 420 Westwood Plaza, #48-121, Los Angeles, CA 90095-1597 USA

Two dimensional discrete dislocation dynamics simulations, incorporating 3D mechanisms are used to study the evolution of plasticity at crack-tip. The result of dislocation reactions: like dislocation annihilation, dislocation junction formation and dynamic source generation are taken into account apart from the long range elastic interaction between dislocations and with the crack. The method is used to determine the brittle-ductile transition in ferritic steels where cleavage propagation from microcracks around hard carbide precipitates or other microcrack initiation sites plays a critical role in the fracture behavior. The dependence of ductile-to-brittle transition temperature (DBTT) with irradiation is studied using the variation of yield stress with irradiation. The micro mechanisms that lead to the sharp increase in the fracture toughness with temperature around DBTT will be discussed. The simulation results are compared with available experimental data.

Dynamics of Edge and Screw Dislocations in Copper in the Environment of Radiation-Induced Clusters: *Yuri N. Osetsky*[1]; David J. Bacon[2]; Bachu N. Singh[3]; [1]Oak Ridge National Laboratory, Computer Scis. & Math., PO Box 2008, MS-6138, Oak Ridge, TN 37831 USA; [2]University of Liverpool, Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK; [3]Risoe National Laboratory, Matls. Dept., PO Box 49, Roskilde DK-4000 Denmark

Commonly, the level of radiation hardening in the post-irradiation tensile tests is related to the density of defect clusters accumulated during irradiation by assuming that these clusters act as obstacles to motion of dislocation produced by applied stress. In a dynamic rector experiment, on the other hand, both defect clusters and dislocations will be generated simultaneously. In an effort to understand the hardening behaviour under these conditions, the resistance to the motion of dislocations due to glissile and sessile defect clusters continuously generated during irradiation is evaluated.

Void Hardening in BCC and FCC Metals Studied by Atomic-Scale Modeling: *Yuri N. Osetsky*[1]; David J. Bacon[2]; [1]Oak Ridge National Laboratory, Computer Scis. & Math., PO Box 2008, Oak Ridge, TN 37831 USA; [2]University of Liverpool, Matls. Sci. & Engrg., Brownlow Hill, Liverpool L69 3GH UK

We present results of large-scale atomic modeling of interaction between moving edge and screw dislocations and voids in model crystals of BCC iron and FCC copper. We have considered dislocations with the Burgers vector ½<111> in Fe and ½<110> in Cu, and voids of up to 6nm diameter at T=0. In Fe, where a dislocation is non-dissociated, the critical resolved shear stress (CRSS) is similar to estimates obtained by continuum modeling when dislocation self-stress is taken into account. In Cu, where dissociation occurs, the CRSS for small (<4nm) void is lower than that predicted by the continuum modeling, whereas for larger voids the results are in good agreement. We explain this by the structure of the dissociated dislocation and features of its interaction with small obstacles, when each partial interacts independently, and large obstacles, when the effect of the dissociation becomes negligible. Other atomic-scale features are also demonstrated and discussed.

An Assessment of the Dislocation Obstacle Strength of Precipitates and Defect Cluster: *Takuya Yamamoto*[1]; G. Robert Odette[1]; Brian D. Wirth[2]; [1]University of California, Dept. of Mechl. Engrg., Santa Barbara, CA 93106 USA; [2]University of California, Dept. of Nucl. Engrg., Berkeley, CA 94720-1730 USA

Small angle neutron scattering size (r) and volume fractions (f) characterization of copper rich (CRP) and nickel-manganese rich (MNP) precipitates in irradiated RPV steels were combined with yield stress changes to assess the precipitate-dislocation obstacle strength. The database covers a wide range of compositions (Cu, Ni, Mn,..) for RPV steels and model alloys irradiated over a range of flux, fluence and temperature. The yield stress/square root of volume fraction are compared to both the Russell-Brown model (RBM) and recent results of molecular dynamics simulations. Analysis of complex steels requires first de-superimposing the individual precipitate contribution to the overall yield stress. Precipitate strengths are generally consistent with the RBM peaking at 1.2 nm at values from 2800±200 to 4500±400 MPa. The peak hardening increases between pure copper precipitates and MNPs that may be partially ordered. The corresponding strength of matrix defects that harden low copper steels is also assessed.

On the Interactions Between a Vacancy and Interstitial Loops in Metals: M. Angels Puigvi[1]; *Anna Serra*[1]; Nieves de Diego[2]; Yuri N. Osetsky[3]; David J. Bacon[4]; [1]Polytechnic University of Catalonia, Applied Math. III, Jordi Girona 1-3, Barcelona 08034 Spain; [2]Universidad Complutense, Física Materiales, Facultad de Física, Ciudad Universitaria, Madrid Spain; [3]Oak Ridge National Laboratory, Computer Sci. & Math. Div., PO Box 2008, Oak Ridge, TN 37831-6158 USA; [4]Liverpool University, Matls. Sci. & Engrg., Dept. of Engrg., Liverpool L69 3GH UK

Interactions between point defects and defect clusters and between them and with existing microstructure features cause microstructure evolution and lead to changes in mechanical and physical properties of the irradiated materials. We present results of atomic-scale computer modelling of interactions between a cluster of self-interstitial atoms (SIAs) and a single vacancy in models of bcc, fcc and hcp metals. This type of reaction is considered to be one of the most frequent because formation of SIA clusters, particularly glissile ones, is commonly observed in high energy displacement cascades in all metals. The interaction depends strongly on the dislocation nature of the cluster and is therefore different in the three crystal structures. Vacancy-SIA recombination, in particular, is inhibited by dissociation of the SIA loop on its glide prism.

Multicomponent Multiphase Diffusion Symposium in Honor of John E. Morral: Diffusion in Oxide Systems

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2:00 PM
Substitutional and Interstitial Diffusion in α_2-$Ti_3Al(O)$: *E. Copland*[1]; D. J. Young[2]; B. Gleeson[3]; [1]Case Western Reserve University, Cleveland, OH USA; [2]University of New South Wales, Sydney, NSW Australia; [3]Iowa State University, Ames, IA USA

The reaction between Al_2O_3 and α_2-Ti_3Al was studied with a series of Al_2O_3 / α_2Ti_3Al multiphase diffusion couples annealed at 900, 1000 and 1100°C. The diffusion-paths were found to strongly depend on α_2-$Ti_3Al(O)$ composition. For alloys with low oxygen concentrations the reaction involved the reduction of Al_2O_3, the formation of a γ-TiAl reaction-layer and diffusion of Al and O into the α_2-Ti_3Al substrate. Measured concentration profiles across the interaction-zone showed "up-hill" diffusion of O in α_2-$Ti_3Al(O)$ indicating a significant thermodynamic interaction between O and either Al or Ti. As O takes interstitial sites in α_2-$Ti_3Al(O)$ the diffusion coefficients for O were determined independently from the interdiffusion of Ti and Al on the substitutional lattice. Diffusion coefficients are reported for α_2-$Ti_3Al(O)$ and γ-TiAl. Finally this is related to the subsequently measured activities of Al, Ti and O in α_2-$Ti_3Al(O)$ in an attempt to better understand the nature of the thermodynamic interaction observed in the measured diffusion-paths.

2:25 PM
Diffusion and Interface Stability During Solid-State Displacement Reactions: *S. N.S. Reddy*[1]; L. B. Wiggins[1]; [1]IBM Corporation, Sys. & Tech. Div., 2070 Rte. 52, B/330, Z/81A, Hopewell Junction, NY 12533 USA

The displacement reaction, A (metal) + BO (oxide) = AO (oxide) + B (metal), at high temperatures is controlled by the diffusion in product phases, namely cation diffusion in AO and oxygen diffusion in B. The stability of the reactive interface is dependent on which of the diffusion step is rate controlling, and was first demonstrated by Rapp, Ezis and Yurek. In this paper, the transition from stable to unstable reactive interface is studied by controlled variation in cation diffusion during the reaction between Copper Oxide and Co-Fe alloys. The transition to interface instability occurs when the product oxide, (Co,Fe)O, can support a cation flux that exceeds the maximum possible Oxygen flux in Copper. The concepts are illustrated using reaction kinetics and product zone microstructure.

2:50 PM
Uphill Diffusion and Zero Flux Planes in Garnets: An Experimental and ATEM Study: *D. Vielzeuf*[1]; A. Lupulescu[2]; A. Baronnet[1]; A. Addad[3]; [1]Université Marseille – CNRS, CRMCN, 5 rue Kessler, 63038 Clermont-Ferrand, France; [2]RPI, Matl. Sci. & Engrg. Dept., Troy, NY 12180 USA; [3]Université de Lille – CNRS, LSPES, France

In order to determine calcium diffusivity in garnets, we carried out self-annealing experiments at 1.3 GPa, 1050-1250°C, from 5 to 36 days, in piston-cylinder apparatus. Polished and calibrated (250-350µm) garnet spheres (Fe51Mg45Ca3Mn1) were incorporated in a powder of clinopyronene and garnet in a graphite and Pt container. At HP and HT, the powder partially melted, recrystallized and developped a calcic overgrowth (Fe28Mg54Ca16Mn2) around the garnet spheres. Long duration experiments were performed to provide measurable relaxation profiles at the interface. Diffusion profiles are narrow (<1µm) and were measured using ATEM techniques. Ca–(Fe,Mg) interdiffusion coefficients in the range 1*10-19 and 1*10-21 m2s-1 were determined at 1250°C and 1050°C, respectively. Some penetration curves are indicative of uphill diffusion. Modelling of these curves indicates the presence of zero flux planes. As far as we know, it is the first time uphill diffusion is evidenced in silicates.

3:15 PM Break

3:30 PM
Internal Displacement Reactions in Multicomponent Oxide Solid Solutions: *S. N.S. Reddy*[1]; D. N. Leonard[2]; L. B. Wiggins[1]; K. T. Jacob[3]; [1]IBM Corporation, Sys. & Tech. Grp., 2070 Rte. 52, B/330, Z/81A, Hopewell Junction, NY 12533 USA; [2]North Carolina State University, Dept. of Matls. Sci., Raleigh, NC 27695 USA; [3]Indian Institute of Science, Dept. of Metall., Bangalore 560012 India

Internal displacement reactions in oxide solid solutions are of the type: A(metal)+(B,C)O(oxide)=B(metal)+(A,C)O(oxide). The oxides AO,BO and CO are iso-structural and form complete solid solutions with A,B and C occupying the same sub-lattice. During reaction, cation B in oxide is displaced by A and B is precipitated as internal metal phase. The cation C does not participate in the chemical exchange reaction. At low concentrations of BO in the starting oxide, the kinetics are controlled by cation diffusion in the product oxide. The diffusion of cations A,B and C in the product oxide are interrelated through cross-coefficient terms in the flux equations. Concentration gradients develop for all cations in product zone. The concepts are illustrated by the following reactions at 1273 K : Fe+(Ni,Mg)O=Ni+(Fe,Mg)O and Fe+(Co,Mg)O=Co+(Fe,Mg)O.

3:55 PM
Oxygen Diffusivity in Nonstoichiometric Cerium Dioxide: *Petrica Cristea*[1]; Marius Stan[1]; [1]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

The diffusion-mediated interactions between intrinsic point defects in nonstoichiometric cerium dioxide is investigated. It is shown that the electric charge carried by the oxygen ions, the presence of reduced Ce(3+) cerium ions, and the orientation of defect pairs influence the frequencies and the local configurations generated by the diffusion jumps. The oxygen self-diffusivity and chemical diffusivity are calculated as functions of temperature and partial oxygen pressure.

4:20 PM
Simulations of High Temperature Internal Oxidation: *Yali Li*[1]; John E. Morral[2]; [1]University of Connecticut, Dept. of Metall. & Matls. Engrg., Storrs, CT 06269-3136 USA; [2]Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH 43210 USA

In order to help design commercial alloys with improved oxidation resistance, mathematical models are being developed to predict internal oxidation as a function of physical properties and boundary conditions. Two methods being used are: Error Function Modeling using Mathematica and Finite Difference Modeling using DICTRA. Mathematica can be applied in the limit of small supersaturations while DICTRA can be applied to more general cases, except when superficial oxide scales form. One limitation is that all simulations assume local equilibrium conditions. Corresponding experimental results on internal oxidation of Cu-Ni alloys will be compared with simulation predictions.

4:45 PM
Anomalous Oxidation of Ferritic Stainless Steels in Air/Hydrogen Fuel Dual Environments: *Z. Gary Yang*[1]; Prabhakar Singh[1]; Jeff Stevenson[1]; Gordon Xia[1]; [1]Pacific Northwest National Laboratory, Matls. Sci. Div., 902 Battelle Blvd., Richland, WA 99352 USA

Ferritic stainless steels are among the most promising candidate alloys to construct interconnects in the intermediate temperature (650-800°C) solid oxide fuel cells (SOFCs). During SOFC operation, the interconnects are working in a dual environment, i.e. simultaneously exposed to air at cathode side and a fuel (e.g. hydrogen) at the anode side. Our recent studies found that the oxidation behavior of stainless steels in the dual environments can be significantly different from that in a single exposure, either an oxidizing or reducing atmosphere. The anomalous oxidization is attributed to the hydrogen diffusion flux from the airside to the fuel side under the influence of a hydrogen (as well as water) gradient across the stalinless steel interconnects. This paper will present the details of our study on a number of selected alloys under the dual environments and discuss mechanistic understanding on the anomalous oxidation.

Neutron Diffraction Characterization of Mechanical Behavior: Deformation III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Hahn Choo, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA; Peter K. Liaw, University of Tennessee, Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Xunli Wang, Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37831 USA

Tuesday PM Room: 3004
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sean R. Agnew, University of Virginia, Dept. of Matls. Sci. & Engrg., Charlottesville, VA 22904 USA; Raj Vaidyanathan, University of Central Florida, AMPAC/MMAE, Orlando, FL 32816 USA

2:00 PM Invited
Measurement and Modeling of Internal Stresses in Directionally Solidified High Volume Fraction Superalloys at High Temperatures: *B. S. Majumdar*[1]; S. Ma[1]; D. W. Brown[2]; [1]New Mexico Tech, Matls. & Metallurgl. Engrg., Socorro, NM 87801 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

Superalloys represent remarkable achievement in the engineering of coherent microstructures, and are being pushed for application at temperatures exceeding 90% of the melting point. Much of the creep life in this regime is dominated by deformation being confined to narrow 50 - 100 nm wide ?× channels, with the 300-500 nm ?×' phase remaining primarily elastic, except at high stresses. The large confinement, and the different elastic/inelastic response of the phases, induce significant internal stresses. These stresses, in turn, play a large role in microstructural stability, and final fracture of the alloy. In order to track the internal stress development, we conducted insitu creep experiments on a columnar grain directionally solidified superalloy using the pulsed neutron source at the SMARTS facility at LANSCE. Tests were conducted at a range of stresses at 900 C, and a rocking technique was successfully used to monitor the elastic strains in the two phases as a function of time. The diffraction measurements showed that the misfit in the loading direction increased fairly rapidly with time, while it decreased in the transverse direction. These results are rationalized in terms of the development of dislocation network at the gamma/gamma-prime interface. TEM analysis was conducted to characterize the interface dislocation structure, and it was observed that these networks formed fairly early in life. However, they reacted into a configuration that appeared to reduce the overall misfit between the gamma and gamma-prime phases at high temperature. The evolution and reaction of these networks will be discussed, and calculations will be presented to show how these networks might influence overall creep rate. Results from finite element method analysis will also be presented, with the overall goal of developing a realistic life prediction methodology based on actual micromechanisms of deformation. We thank Dr. Venkat Seetharaman of Pratt & Whitney for kindly providing us the DS superalloy, and Drs. Bjorn Clausen and Mark Bourke of LANL for their help in accomplishing the neutron diffraction measurements.

2:20 PM Invited
Interaction of Stress on Phase Stability in a Single Crystal Nickel Base Superalloys: S. S. Babu[1]; *Edward Andrew Payzant*[1]; D. W. Brown[2]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., PO Box 2008, MS 6064, Oak Ridge, TN 37831-6064 USA; [2]Los Alamos National Laboratory, Lujan Ctr., Los Alamos, NM 87545 USA

Turbine blades used in both land-based gas turbines and in aircraft engines are made up of single-crystal nickel-base superalloys. Their high-temperature strength is related to the presence of coherent, hard L12 ordered γ' precipitates within the γ (fcc crystal structure) matrix. The important microstructural parameters (volume fraction, size, shape, and composition of the γ' phase, as well as lattice mismatch between γ' and γ phases) are affected by the bulk composition, heat-treatment conditions, stress, and time. Prior research suggests that on application of stress at high temperature, the hard γ' phase does not take part in the plastic deformation, which is concentrated in the soft γ phase. A critical requirement is to describe the above phenomenon under in situ thermomechanical conditions and in different crystallographic directions. In this research, the lattice parameters of γ' and γ phases in a single-crystal nickel-base superalloy were investigated by neutron-diffraction while subjected to high-temperature stress during continuous cooling and isothermal conditions using the SMARTS beamline at LANSCE.

2:40 PM Invited
Elevated Temperature Uniaxial and Cyclic Loading of 316 Stainless Steel: *Mark Daymond*[1]; P. John Bouchard[2]; [1]Queen's University, Mechl. & Matls. Engrg., Nicol Hall, Kingston, ON K7L 3N6 Canada; [2]British Energy Generation Ltd., Barnwood, Gloucester GL4 3RS UK

The macroscopic stress-strain response of type 316 stainless steel subjected to cyclic loading at high temperature evolves from the virgin material state to a cyclically hardened state. In practical applications however, the material is not simply cycled, but undergoes cycling with dwell interruptions at various points around the cycle. A knowledge of the start-of-dwell stress alone is insufficient to describe the creep relaxation behaviour observed; the location of the dwell event around the cycle is important. We have carried out experiments investigating the response of the material at a range of elevated temperatures in order to explore the interaction between microscopic internal stresses developed during cyclic loading with the high temperature creep response of the material during dwell periods at different positions around the cycle. The grain orientation strains obtained by diffraction are compared with an elasto-plastic polycrystal deformation model to clarify the role of internal stresses in the material response.

3:00 PM
Creep Relaxation Near a Notch in a Specimen Loaded in Tension: *Nicolas Christodoulou*[1]; Brian W. Leitch[1]; Ronald Rogge[2]; [1]Chalk River Laboratories, AECL, Deformation Tech. Branch, Chalk River, Ontario K0J 1J0 Canada; [2]National Research Council of Canada, Steacie Inst. of Molecular Scis., Chalk River Labs., Chalk River, Ontario K0J 1J0 Canada

A notch is usually defined as a relatively smooth discontinuity or a stress concentration feature that elevates the stress around the region of the notch by a factor of 2 or 3. The stress elevation around a notch is not as severe as that created by the sharp re entrant geometry of a crack. A notch can be a consequence of the manufacturing process or it can be created by an operating event, such as contact with another component. Depending on the far-field stress and the material properties, stress analysis of notches in components subjected to cyclic operating conditions could indicate that the locally elevated stress levels are sufficient to induce cracks that are formed at the notch root and perhaps limit the useful life of the component. However, the incidence of these life-limiting events appears to be relatively low and therefore the high stresses around the notch region must be relaxed in some manner. This paper describes an investigation to ascertain and predict this stress re-distribution with time. Experimental measurements of the stresses around a large radius notch in a tensile specimen were determined using neutron diffraction techniques. A large radius is defined as a radius that is a substantial portion of the specimen cross section. Experimental creep tests on notched, Zr-2.5Nb specimens were carried out at 25 and 300°C. Neutron diffraction measurements were taken during the loading sequence and at regular time intervals during the creep portion of the experiment. The experimental findings are compared with the results of a finite element model where a viscoplastic constitutive law was implemented to simulate the creep relaxation of the notch region stresses. Generalized constitutive creep laws have been developed for un-irradiated and irradiated zirconium materials. Only the un-irradiated material behaviour is considered here and a good agreement is found between the experimental findings and the analytical results.

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Tension/Compression Asymmetry During Fatigue: *Alexandru Dan Stoica*[1]; Xun Li Wang[1]; James W. Richardson[2]; Donald W. Brown[3]; Bjorn Clausen[3]; Hongbo Tian[4]; Michael Lee Benson[4]; Peter K. Liaw[4]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, Bldg. 8600, MS 6474, Oak Ridge, TN 37830 USA; [2]Argonne National Laboratory, Intense Pulsed Neutron Source, 9700 S. Cass Ave., Argonne, IL 60439-4163 USA; [3]Los Alamos National Laboratory, Los Alamos Neutron Sci. Ctr., MS H831, Los Alamos, NM 87545 USA; [4]University of Tennessee, Dept. of Matls. Sci. & Engrg., 319 Dougherty Engrg. Bldg., Knoxville, TN 37996 USA

The residual strain dependence of grain orientation, developing during fatigue life in 316LN stainless steel, was determined by time-of-flight neutron diffraction, as well as longitudinal and transversal lattice

strain dependence of tensile or compressive load on pre-fatigued specimens. The analysis of both types of strain data involves the general concept of strain/stress orientation distribution function, which takes into account of texture symmetry and elastic anisotropy. A phenomenological, model- independent representation of lattice strains was developed for uniaxial loading of cylindrical untextured specimens. The contribution of plastic deformation induced intergranular strains is clearly separated from elastic anisotropy behavior. A marked tension/compression asymmetry was observed to develop during fatigue life. A dramatic difference in residual intergranular strain state measured after the compression and tension half-cycle appears as soon as the fatigue cracks are generated. In late stage, the residual strains vanish for fatigue tests ending in tension and remain high for fatigue tests ending in compression. It is shown that the lattice strain behaviors in incremental in-situ loading experiments match the residual strains in ex-situ samples, if the creep deformation during in-situ loading is taken into account. This research was supported by U.S. Department of Energy, Basic Energy Sciences, Division of Materials Science and Engineering, under contract DE-AC05-00OR22725 with UT-Battelle, LLC. SNS is a partnership of six national laboratories: Argonne, Brookhaven, Jefferson, Lawrence Berkeley, Los Alamos, and Oak Ridge.

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Internal Strain and Texture Development During Cyclic Loading of Haynes® 230®Nickel Based Superalloy: *Tarik A. Saleh*[1]; Hahn Choo[1]; Donald W. Brown[2]; Bjorn Clausen[2]; Peter K. Liaw[1]; Sven K. Vogel[2]; Dwaine L. Klarstrom[3]; Raymond A. Buchanan[1]; [1]University of Tennessee, Dept. of Matls. Sci., 434 Dougherty Engrg., Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, Los Alamos Neutron Sci. Ctr., Los Alamos, NM USA; [3]Haynes International, Inc., Kokomo, IN USA

Haynes 230 is a solid solution strengthened, face centered cubic (FCC), nickel based superalloy. This alloy is frequently used in fatigue intensive applications such as turbine engines. In situ neutron studies can reveal changing internal and residual strains in a material during monotonic or cyclic loading. Additionally, neutron diffraction experiments can reveal the texture of the microstructure, before and after deformation has taken place. Haynes 230 alloy was subjected to monotonic and tension-tension cyclical loading at a σmax of 700 MPa. Internal strains were measured during cycling using in situ neutron diffraction at the Spectrometer for Materials Research at Temperature and Stress (SMARTS) at the Los Alamos Neutron Science Center (LANSCE). Texture was measured before and after cycling using the High Pressure Preferred Orientation (HIPPO) diffractometer at LANSCE. During the fatigue experiment, internal strains were seen to relax over the first 1000 cycles (2.4% of the fatigue life) and then hold steady over the remainder of the fatigue experiment. The initial texture of the fatigue specimens was found to be random. After cycling, the specimen had a texture, 2.5 times random, with the normal to the 111 plane oriented parallel to the loading direction and a two-fold symmetry. The experimental data is being compared to finite element modeling and viscoplastic self-consistent (VPSC) modeling, to develop a mechanistic understanding of the effects of fatigue and deformation on the internal strains and microstructural texture. The present work is supported by the National Science Foundation (NSF), the Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, and the International Materials Institutes (IMI) Program under DMR-0231320, and the Combined Research-Curriculum Development (CRCD) Program, under EEC-9527527 and EEC-0203415, with Drs. P. W. Jennings, L. S. Goldberg, L. Clesceri, C. Huber and Ms. M. Poats as contract monitors.

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Understanding Lüders-Band Evolution During Fatigue by Thermography and Neutron Diffraction Techniques: *Bing Yang*[1]; Yinan Sun[1]; Peter K. Liaw[1]; Choo Hahn[1]; Mark R. Daymond[2]; J. Y. Huang[3]; R. C. Kuo[3]; J. G. Huang[4]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [2]Rutherford Appleton Laboratory, ISIS Facility, Chilton, Didcot OX11 0QX UK; [3]Institute of Nuclear Energy Research, PO Box 3-14, 1000 Wenhua Rd., Chiaan Village, Lungtan 325 Taiwan; [4]Taiwan Power Company, Taipei Taiwan

In tensile testing, yielding, initialized with discontinuous and localized plastic zones or bands, are called the Lüders-band effect, which is commonly observed in steels containing interstitial elements. In engineering applications, Lüders bands often indicate the onset of the plastic deformation at the vicinity of stress concentrations and inclusions, which will contribute to crack initiations - a critical issue in fatigue damage processes. However, up to now, few studies have been performed to understand the Lüders-band evolutions during cyclic loading. In the current study, a detailed investigation on the Lüders band evolution processes during fatigue has been conducted by a combination of the state-of-the-art infrared thermography and neutron scattering techniques. While thermography detection demonstrated in-situ the initiation and propagation of Lüders bands, neutron diffraction technique provides quantitative analyses of the residue stresses in the Lüders band and disclosed the internal mechanism of Lüders band evolution during fatigue, which will be essential to understand the early stages of material fatigue behaviors.

4:40 PM

Measurement and Modeling of the Effect of Hydrogen on the Mechanical Behavior of a Zircaloy-4 Alloy: *E. Garlea*[1]; A. Ionita[1]; H. Choo[1]; D. W. Brown[2]; R. A. Buchanan[1]; P. K. Liaw[1]; C. R. Hubbard[3]; [1]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

Hydride formation is one of the main degradation sources of zirconium alloys in hydrogen-rich environments. When sufficient hydrogen is available in these alloys, zirconium-hydride precipitates can be formed. The brittle hydrides near a crack tip can initiate crack growth leading to a premature failure of the material. Hydride formation is believed to be enhanced by the presence of residual or applied stresses. Therefore, the increase in stress field ahead of a crack tip may promote precipitation of additional hydrides. In order to verify this behavior, we investigated the effect of hydrogen charging on the lattice dilation, hydride formation, and mechanical behavior of Zircaloy-4 alloy using neutron diffraction. Spatially-resolved internal strain measurements were made on fatigue pre-cracked compact-tension specimens using in-situ neutron diffraction under applied loads of 667 and 4,444 newtons with or without the presence of hydrogen. The results show that the internal strain profile near the crack tip changes significantly when the specimen is charged with hydrogen. The neutron diffraction results will be compared to the theoretical predictions obtained using finite element modeling.

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Measurement and Modeling of Crack Tip Strains During Tensile Loading and Unloading Cycles: *Y. Sun*[1]; A. Ionita[1]; H. Choo[1]; P. K. Liaw[1]; Y. L. Lu[1]; B. Yang[1]; D. W. Brown[2]; [1]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

The changes in the elastic-lattice strain profiles and plastic zone around a fatigue crack in a compact-tension (CT) specimen were investigated during tensile loading and unloading cycles using neutron diffraction. Spatially-resolved strain measurements were performed to determine the in-plane and through-thickness lattice-strain profiles ahead of the crack tip under a constant tensile load. The strain scanning was repeated under various applied loads ranging from 667 to 6,667 N. Subsequently, overload at 8,889N was applied, and the strain scans repeated. After overload, large compressive strain fields were observed close to the crack tip indicative of the crack-closure phenomena. Diffraction-peak broadening related to the plastic zone was also investigated. The finite element model predictions of the strain (both elastic and plastic) distributions ahead of the crack tip showed good agreements with the experimental data. The results provide fundamental understanding of the micro-mechanics around the crack tip during fatigue deformation.

5:20 PM

Elastic Modulus of B19' Shape-Memory NiTi from Neutron Diffraction, Instrumented Nanoindentation and Extensometry: *S. Rajagopalan*[1]; A. L. Little[1]; M. A.M. Bourke[2]; Raj Vaidyanathan[1]; [1]University of Central Florida, AMPAC/MMAE, Orlando, FL 32816 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

In situ neutron diffraction during loading and instrumented nanoindentation experiments, using a spherical indenter, were conducted on a B19' shape-memory NiTi alloy to study its elastic behavior. Lattice plane and elastic moduli obtained from the two approaches were comparable but significantly higher than Young's moduli obtained from extensometry in this work and previously widely reported. The lower values from extensometery were attributed to twinning at low stresses that was observed in the diffraction measurements but was suppressed in the spherical nanoindentation experiments. This work was supported by grants from NASA (NAG3-2751) and NSF (CAREER DMR-0239512) to UCF.

Neutron Scattering in Materials Research: Diffraction: Instruments and Nanostructure

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Brent T. Fultz, California Institute of Technology, Department of Materials Science, Pasadena, CA 91125 USA; Michael Atzmon, University of Michigan, Department of Materials Science & Engineering, Ann Arbor, MI 48109 USA

Tuesday PM Room: 3022
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Michael Atzmon, University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109-2136 USA; Brent Fultz, California Institute of Technology, Matls. Sci., Pasadena, CA 91125 USA

2:00 PM Invited
Neutron Scattering Instruments and Opportunities at the Spallation Neutron Source: *R. Kent Crawford*[1]; [1]Oak Ridge National Laboratory, Experimental Facilities Div., Bldg. 8600, Oak Ridge, TN 37830 USA

The unprecedented power and best-in-class neutron scattering instruments of the new Spallation Neutron Source (SNS) facility at Oak Ridge National Laboratory bring many new opportunities for materials science. The SNS has provisions to accommodate up to 24 neutron beam instruments which will cover a broad spectrum of science, and 14 of these have already been funded. Design and construction of these instruments are already well along, and installation of some components is already underway. First instrument commissioning is scheduled for the spring of 2006. This presentation will provide an overview of these instrumentation activities, and will also indicate the performance expected from many of these instruments.

2:30 PM Invited
POWGEN3: A High Resolution Third Generation TOF Powder Diffractometer Under Construction at the SNS: *Jason P. Hodges*[1]; [1]Oak Ridge National Laboratory, Experimental Facilities Div., Spallation Neutron Source, PO Box 2008, Oak Ridge, TN 37831-6474 USA

POWGEN3 is a fundamental departure from previous designs for a time-of-flight powder diffractometer at a spallation neutron source. POWGEN3 may be considered the world's first third-generation time-of-flight powder diffractometer. The combination of a supermirror neutron guide system with a very large modular detector array means POWGEN3 is a very efficient instrument. The high count rates thus achieved together with high-resolution characteristics present a big leap forward in performance over previous diffractometer designs. POWGEN3 will thus provide unprecedented opportunities for new science in the study of polycrystalline materials.

3:00 PM Break

3:20 PM Invited
Nanoscale Structures in Complex Crystals Using Neutron Pair Distribution Function Methods: *Simon J.L. Billinge*[1]; [1]Michigan State University, Physics & Astron., 4268 Biomed. Phys. Sci. Bldg., E. Lansing, MI 48824 USA

We are increasingly interested in complex materials for their unique functional properties. Complex materials often exhibit nanoscale local structures that are important in determining their properties. These come about from defects but often are intrinsic, coming from competing interactions in the materials. It is important to characterize these "nanostructures" but this is difficult because they are not, by their nature, long-range ordered and cannot be studied using traditional crystallographic methods. Imaging probes such as STM and TEM are useful but do not give bulk average information and it is difficult to get quantitative atomic positions in this way. An important complement to these probes are bulk local probes. I will describe the atomic pair distribution function (PDF) analysis method which has proven to be a powerful quantitative probe of nanostructures. I will use examples where neutron diffraction data gives either unique data, or data complementary to x-ray information, resulting in successful understanding of nanostructures in crystals. I will also discuss prospects for the future.

3:50 PM Invited
Medium-Range Atomic Order Studied by Neutron Scattering: *Takeshi Egami*[1]; [1]University of Tennessee/Oak Ridge National Laboratory, MSE/Physics, 208 S. College, 1413 Cir. Dr., Knoxville, TN 37996 USA

Long-range atomic order, or the lattice structure, can readily be studied by diffraction, while the local structure can be accessed by the local probes, such as the EXAFS and NMR. However, there is a dearth of techniques that can probe the medium-range atomic order in the range from 0.5 to 2 nm. Note that TEM sees the structure in this range, but averaged over the sample thickness. We discuss the method of atomic pair-density function (PDF) analysis, which can bridge this gap. In particular, by the upgrading of a pulsed neutron diffractometer, NPDF, of Los Alamos, we now can determine the PDF from zero to up to 20 nm or more. We discuss recent examples, including the nanoscale domains in LiNiO_2. It is also possible to determine the dynamic PDF, by inelastic neutron scattering. An example of local lattice dynamics of relaxor ferroelectric PMN will be discussed.

4:20 PM Invited
Local Atomic Environments in NiPt and SiGe Alloys: *J. Lee Robertson*[1]; [1]Oak Ridge National Laboratory, Ctr. for Neutron Scattering, Bldg. 7962, Oak Ridge, TN 37831-6393 USA

We have investigated two alloys, Ni52-Pt48 and a dilute alloy of 7 atomic percent Ge in Si, using the neutron disk chopper spectrometer (DCS) at NIST. For the Ni-Pt study two crystals were used, a null-matrix crystal using isotopic 62Ni chosen so that the components of the scattered intensity that depend on the average lattice are suppressed leaving only those components that depend on the local atomic configurations that deviate from the average lattice, and a much larger crystal made with natural Ni for studying the dynamics of the alloy. The chemical short-ranged order and static displacement (size effect) scattering are quite prominent for the null-matrix crystal. The Ge-Si crystal showed pronounced fans of diffuse scattering emanating strongly on the low angle side of the Bragg peaks indicating that the Si lattice is expanded around the Ge "defects" and the static displacement field extends to distant neighbors.

Phase Stability, Phase Transformation and Reactive Phase Formation in Electronic Materials IV: Phase Equilibria, Interfacial Energy and Wetting Phenomena in Solder Joints

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee
Program Organizers: Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Katsuaki Suganuma, Osaka University, Department of Nanomaterials and Environmental Conscious Technology, Ibaraki, Osaka 567-0047 Japan

Tuesday PM Room: 3016
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Herbert Ipser, University of Vienna, Dept. of Inorganic Chmst., Wien A-1090 Austria; Douglas J. Swenson, Michigan Technological University, Dept. of Matls. Sci. & Engrg., Houghton, MI 49931 USA

2:00 PM
Thermochemistry of the Quaternary System Ag-Cu-Ni-Sn: *Hans Christian Flandorfer*[1]; Christoph Luef[1]; Herbert Ipser[1]; [1]University Vienna, Inorganic Chmst., Waehringer Str. 42, Vienna 1090 Austria

The quaternary intermetallic system Ag-Cu-Ni-Sn and its binary and ternary constituents are of importance because Ag-Cu-Sn is a key systems for lead-free soldering and Ni is a frequently used contact material. The aim is to establish reliable thermochemical and phase relation data from literature and our own experiments. First we determined partial and finally integral enthalpies of mixing of liquid alloys by calorimetric measurements performed at different temperatures.

The experimental results were fitted using the Redlich-Kister-Muggiano formula in order to provide a numerical description of the integral enthalpies of mixing according to the CALPHAD method. The experimental results were also compared with extrapolations and theoretical values from pseudo-potential calculations. Our work is considered as a contribution to the COST-Action 531 "Lead-free solder materials". The main objective of the action is to increase the basic knowledge on possible alloy systems that can be used as lead-free solder materials and to provide a scientific database on possible lead-free solder materials and soldering processes. Experimental thermochemical data are important for the optimization of phase diagram calculations as well as the evaluation of physicochemical properties like e.g., diffusion and surface tension.

2:30 PM

Bi-From Woods Metal to Lead Free Solders: *Sabine Knott*[1]; Adolf Mikula[1]; [1]University of Vienna, Inst. of Inorganic Chmst., Währingerstraße 42, Vienna 1090 Austria

Bismuth, which was discovered as a metal in 1753, offers a wide range of applications. It is a component of Woods metal, named after the American metallurgist B. Wood, which is widely used in electrical fuses and in automatic fire alarm and sprinkler systems. Bismuth is environmentally friendly and non toxic and it is already used as a replacement of lead in solder alloys. The investigated thermodynamic data of the ternary Ag-Bi-Sn and the ternary Cu-Bi-Sn systems will be presented. The Ag-Sn-Bi solder is especially used for the Wave soldering and Reflow soldering processes. No data of the ternary Cu-Bi-Sn system are available within the literature although the knowledge of the properties of this system might be necessary not only for a possible application as a lead free solder but also as a base for the investigation of the quarternary Ag-Bi-Cu-Sn system.

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Interaction of Ag-In-Sn Solders with Palladium Substrates: A Phase Diagram Approach: Adela Zemanova[2]; Ales Kroupa[3]; Jan Vrestal[2]; *Herbert Ipser*[1]; Christoph Luef[1]; Hans Flandorfer[1]; [1]University of Vienna, Dept. of Inorganic Chmst., Waehringerstrasse 42, Wien A-1090 Austria; [2]Masaryk University, Dept. of Physl. & Theoretl. Chmst., Kotlarska 2, Brno CZ-61137 Czech Republic; [3]Institute of Physics of Materials AS CR, Zizkova 22, Brno CZ-616 62 Czech Republic

In order to understand the interaction of lead-free Ag-In-Sn solders with palladium substrates one has to know the quaternary Ag-In-Pd-Sn phase diagram, and for that all the constituent binaries and ternaries. For this purpose, enthalpies of mixing were determined calorimetrically for liquid ternary Ag-Pd-Sn and In-Pd-Sn and quaternary Ag-In-Pd-Sn alloys. These experimental results were taken into account as an input for the optimization in CALPHAD-type phase diagram calculations of the above-mentioned two ternary phase diagrams. These were then combined with an earlier optimization of the Ag-In-Pd system as well as with literature data on the Ag-In-Sn system to obtain a first idea of the phase diagram of the quaternary Ag-In-Pd-Sn alloy system. The calculations were supported by a number of specifically devised experiments.

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Phase Equilibria in the Sn-Rich Corner of the Sn-Cu-Ni Ternary Alloy System: Chia-Ying Li[1]; *Jenq-Gong Duh*[1]; Sue-Yueh Tsai[2]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., No. 101, Sec. 2, Kuan Fu Rd., Hsinchu 300 Taiwan; [2]National Tsing Hua University, Instrument Ctr., No. 101, Sec. 2, Kuan Fu Rd., Hsinchu 300 Taiwan

The interfacial reactions between solders and under bump metallization (UBM) are of highly interests recently in flip chip technology. Intermetallic compounds (IMCs), i.e. $(Cu,Ni)_6Sn_5$ and $(Ni,Cu)_3Sn_4$, formed between solders and UBM. To fully understand the interfacial reactions and phase transformation phenomenon, a suitable phase diagram concerning solder, IMCs and UBM material is required. As a result, Sn-riched phase in Sn-Cu-Ni ternary phase diagram is very critical in determining the concentration tendency of x and y values in $(Ni_{1-x},Cu_x)_3Sn_4$ and $(Cu_{1-y},Ni_y)_6Sn_5$ compounds. In this study, ternary Sn-Cu-Ni alloys were prepared and annealed at 240°C. Three equilibrium phases, Sn, Ni_3Sn_4 and Cu_6Sn_5, were identified by XRD analysis, and also evidenced in BEI micrograph. Using EPMA quantitave analysis, three acme compositions of the ternary region in the Sn-Cu-Ni isotherm near the Sn-riched corner were determined as 98.5 at.%Sn, $(Ni_{0.80},Cu_{0.20})_3Sn_4$ and $(Cu_{0.59},Ni_{0.41})_6Sn_5$. Furthermore, the degree of composit ion homogeneity and the distribution of the forming phases were evaluated by X-ray color mapping and phase analysis technique. By way of software program to convert the concentration measured with EPMA, the exact compositions could be mapped on the Sn-Cu-Ni ternary isotherm. In addition, the solubility of the Cu and Ni in $(Ni,Cu)_3Sn_4$ and $(Cu,Ni)_6Sn_5$ compounds was evaluated. Finally, the isothermal section of the ternary Sn-Cu-Ni system at 240°C was proposed on the basis of experimental results in this study. Furthermore, the related phase transformation was also discussed with respect to the formation of the IMCs around 240°C.

3:30 PM Break

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Driving Force of Reactive Wetting in the Au-Sn System: *Liang Yin*[1]; Timothy J. Singler[1]; [1]SUNY Binghamton, Dept. of Mechl. Engrg., 4400 Vestal Pkwy. E., Binghamton, NY 13902-6000 USA

Isothermal sessile drop experiments were performed in a gaseous reducing atmosphere to identify the driving force for the reactive wetting in the Au-Sn system. Two alloys, pure Sn and a eutectic Au-Sn alloy (80 wt.% Au), were used to wet pure Au and an intermetallic compound ζ substrates at 430°C. Spreading on pure Au substrates was accompanied by ζ phase formation and exhibited a near perfect wetting behavior. Spreading on ζ substrates was dissolutive and driven by uncompensated Young's force coupled with solute concentration gradients in the liquid.

4:00 PM

Experimental Wettability Studies Combined With the Related Properties from Data Bases for Tin Based Alloys With Silver, Copper, Bismuth and Antimony Additions: *Moser Zbigniew*[1]; Gasior Wladyslaw[1]; K. Ishida[2]; I. Ohnuma[2]; X. J. Liu[2]; K. Bukat[3]; J. Pstrus[1]; J. Sitek[3]; R. Kisiel[4]; [1]Polish Academy of Sciences, Inst. of Metall. & Matls. Sci., 30-059 Kraków, Reymonta Str. 25 Poland; [2]Tohoku University, Grad. Sch. of Engrg., Dept. of Matls. Sci., Obayama 02, Sendai 980-8579 Japan; [3]Tele and Radio Research Institute, 03-450 Warszawa, Ratuszowa Str. 11 Poland; [4]Warsaw University of Technology, Inst. of Microelect. & Optoelect., 00-662 Warszawa, Koszykowa Str. 75 Poland

Starting from binary eutectic Sn-Ag and close to ternary eutectic Sn-Ag-Cu alloys we have investigated the additions of Sb to quaternary tin based Sn-Ag-Cu-Bi alloys. Main aim is to obtain alloys with wettability and T_m closer to 183°C characteristic for traditional Sn-Pb eutectic solders. In this study we use database for Pb-free solders ADAMIS developed in Tohoku University in Japan, to calculate the phase diagram of the Sn-Ag-Cu-Bi-Sb quinary system by means of Calphad method and the melting phenomena, to simulate solidification using Scheil's model and to calculate surface tensions from thermodynamic parameters of the liquid phase by the Butler's model. The resulting calculated surface tensions will be verified with experimental data from maximum bubble pressure method (performed within 250°C to temperatures exceeding 900°C) and with interfacial tensions from meniscographic studies made at 250°C, finally to draw the conclusions important from the point of view of practical application analyzing wettability, mechanical and electrical properties.

4:20 PM

Wettability of Electroplating Ni-P in Under Bump Metallurgy with Sn-Ag-Cu Solder: *Yung-Chi Lin*[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan

The nickel plating has been used as the under bump metallurgy (UBM) in the microelectronic industry. In this study, the electroplating process was demonstrated to be a good alternative approach to produce the Ni-P layer as UBM. The wettability of several lead-free solders, such as Sn-3.5Ag, and Sn-3.5Ag-xCu (x=0.2, 0.5, and 1.0) solder, on electroplating Ni-P with various phosphorous contents (7wt%, 10wt% and 13wt%) was investigated. The role of phosphorus in the wettability was probed. The surface morphology and surface roughness in electroplating Ni-P was observed with the aid of both field emission scanning electronic microscope and atomic force microscope. The correlation between wettability and phosphorus contents in electroplating Ni-P was evaluated. As the phosphorous contents increased, the nodule size of the Ni-P deposit reduced and surface roughness of Ni-P became smaller. The improvement of surface morphology and surface roughness enhanced the wettability of electroplating Ni-P. The interfacial reaction between lead free solder and electroplating Ni-P UBM was also investigated.

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Interfacial Reactions in the Sn/(Ni,V) Couples and Phase Equilibria of the Sn-Rich Sn-Ni-V System: *Sinn-wen Chen*[1]; Chih-chi Chen[1]; Ching-ya Kao[1]; [1]National Tsing-Hua University, Chem. Engrg. Dept., #101, Sec. 2, Kuang-Fu Rd., Hsin-Chu 300 Taiwan

In the Cu/Ni(V)/Al UBM, 7wt% vanadium is added to overcome the magnetic interference of nickel and to enhance the Nickel sputtering.

The interfacial reactions between various solders with the nickel substrate have been investigated extensively; however, the effects of vanadium addition upon the solder/(Ni,V) interfacial reactions have not been investigated. This study examined the interfacial reactions of pure Sn with the pure vanadium and (Ni,V) substrates of various vanadium contents. It is found that besides the Ni3Sn4 phase, a new ternary phase is formed. The phase equilibria of the Sn-rich Sn-Ni-V are studied. The reaction paths are determined based on the determined isothermal section and the reaction couple analysis.

Phase Transformations Within Small-Size Systems: Magnetic and Structural Transformations

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee, EMPMD-Nanomaterials Committee

Program Organizers: Vijay K. Vasudevan, University of Cincinnati, Department of Chemical and Materials Engineering, Cincinnati, OH 45221-0012 USA; Robert D. Shull, National Institute of Standards and Testing, Metallurgy Division, Gaithersburg, MD 20899-8552 USA; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375-5000 USA; Xinghang Zhang, Texas A&M University, Department of Mechanical Engineering, College Station, TX 77843-3123 USA

Tuesday PM Room: 3002
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Ramamoorthy Ramesh, University of California, Matls. Sci. & Engrg., Berkeley, CA 94720 USA; Marc DeGraef, Carnegie Mellon University, Matls. Sci. & Engrg., Pittsburgh, PA 15229-3180 USA

2:00 PM Invited
The Phase Diagram of Magnetic Nano-Rings: *Marc De Graef*[1]; Marco Beleggia[2]; June Lau[2]; Marvin Schofield[2]; Shakul Tandon[1]; Yimei Zhu[2]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15229-3180 USA; [2]Brookhaven National Laboratory, Ctr. for Functional Nano-Matls., Building 480, PO Box 5000, Upton, NY 11973 USA

Magnetic nano-rings, circular structures with rectangular cross-section, form the basic building blocks of magnetic random access memory (MRAM). There are three basic magnetization states for such rings when they are sufficiently small: uniform axial, uniform in-plane, and vortex. Most studies of magnetic nano-rings use a micromagnetic computational approach to determine the magnetization state for a given set of shape and materials parameters. In this contribution we present an analytical approach to the complete magnetic phase diagram of such rings. We derive the magnetostatic energy, including demagnetization factors, the magnetocrystalline anisotropy and exchange energy terms, and derive from them a four-dimensional phase diagram. The magnetization state is determined by two shape parameters, (ratio of inner to outer radius and aspect ratio), and two material parameters (ratio of anisotropy to magnetostatic energy density and magnetic exchange length). We will present the detailed phase diagram, as well as confirmation of the diagram by means of micromagnetic simulations and electron holography observations.

2:35 PM Invited
Microstructural Evolution and Interfaces in Ferromagnetic Semiconductor Films Based on p-Type Ge-X (X=Mn,Fe,Cr) and n-Type CdCr2Se4: *Ramasis Goswami*[1]; G. Kioseoglou[2]; A. T. Hanbicki[2]; B. T. Jonker[2]; G. Spanos[2]; [1]Geo-Centers Inc., Naval Rsch. Lab., 4555 Overlook Ave., Metall., Code 6320, Washington, DC 20375 USA; [2]Naval Research Laboratory, Code 6300, 4555 Overlook Ave., Washington, DC 20375 USA

Considerable efforts have been made at the Naval Research Laboratory to understand the basic mechanism(s) of ferromagnetic order in Ge-X semiconductors (X = Mn, Fe, Cr), and in hetero-epitaxial thin films based on CdCr2Se4/ZnSe/AlGaAs. It is observed that the Ge transition metal films are not single crystalline and consist of transition metal rich particles in a Ge matrix; small amounts of transition metal are often still retained in solution in the Ge matrix between the particles. Likewise, relatively complex microstructural evolution has been observed in the hetero-epitaxial thin film systems. It is therefore essential to understand the phase evolution and characterize the microstructure and interfaces in these films in order to put forward successful theoretical models which explain the underlying mechanism(s) of ferromagnetic order. Phase transformations, defect evolution, and interfaces have thus been studied in detail in Ge-X (X= Mn, Fe, Cr), CdCr2Se4/ZnSe/AlGaAs/GaAs(LED), and CdCr2Se4/AlGaAs/GaAs(LED) films prepared by molecular beam epitaxy at different substrate temperatures. The microstructural evolution in all three Ge-based systems will be discussed first, and correlated with the magnetic properties. The effect of substrate temperature on the microstructure and interfaces of CdCr2Se4/ZnSe and CdCr2Se4/AlGaAs will be shown next, and related to the observed spin polarized electron injection into the AlGaAs/GaAs LED structures.

3:10 PM
Cu-Fe Interdiffusion During Severe Plastic Deformation of a Nanoscaled Composite: *X. Sauvage*[1]; P. Pareige[1]; [1]Université de Rouen, Groupe de Physique des Matériaux - UMR CNRS 6634, 76801 Saint-Etienne-du-Rouvray France

As reported in the literature, the ball milling of Fe-Cu powder mixtures usually leads to the formation of supersaturated solid solutions. The aim of this work was to induce such a mechanical mixing in a bulk Cu-Fe nanocomposite by continuous severe plastic deformation. The nanostructure of a Cu-Fe(10%vol.) composite processed by High Pressure Torsion (HPT) was investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), and 3D atom probe analysis (3D-AP). This latter technique gives the evidence of Fe-Cu that the smaller the Fe nanoclusters the stronger the interdiffusion. Thus, the driving force for the mechanical mixing is thought to be the result of a high capillary energy. The mechanism of Fe-Cu enhanced diffusion during the HPT process is then discussed. The formation of excess vacancies would dramatically increase the atomic mobility.

3:35 PM Break

3:50 PM Invited
Self-Assembled Single Crystal Ferromagnetic Iron Nanowires formed by Decomposition: *Ramamoorthy Ramesh*[1]; Ladan Mohaddes-Ardabili[1]; [1]University of California, Hearst Memorial Mining Bldg., Berkeley, CA 94720 USA

We report a novel and simple approach to create self-assembled ferromagnetic α-Fe nanowires, which involves spontaneous phase decomposition of a single-phase perovskite oxide during film growth. The growth of $La_{0.5}Sr_{0.5}FeO_{3-x}$ films by pulsed laser deposition under reducing oxygen conditions leads to spontaneous formation of an array of single crystalline α-Fe nanowires embedded in an antiferromagnetic $LaSrFeO_4$ matrix, which grow perpendicular to the substrate and span the entire film thickness. The diameter, shape, density and spacing of these α-Fe nanowires depend systematically on growth conditions. At high growth temperature, square shaped α-Fe pillars with a lateral width of 40-50 nm are formed. As the deposition temperature is reduced the shape evolves progressively into octahedral and then circular section. The diameter of the nanowires reduces to 4-6 nm for the growth at 560ºC. The large remanence and sizable coercivity of the nanowires make them desirable for high-density data storage and other magnetic device applications.

4:25 PM Invited
Self-Assembled Growth of DySi2 Nanowires and Nanostructures on Si(100): Gangfeng Ye[1]; Jun Nogami[1]; *Martin A. Crimp*[1]; [1]Michigan State University, Dept. of Cheml. Engrg. & Matls. Sci., E. Lansing, MI 48823-1226 USA

Dysprosium silicide nanostructures have been grown by metal deposition on to Si(100) substrates at 600°C with coverages ranging from 0.3 to 3.0 monolayers. The resulting DySi2 nanostructures have been characterized using scanning tunneling microscopy (STM) and high resolution transmission electron microscopy (HRTEM). STM has revealed two general types of structures. Large aspect ratio nanowires, with heights up to 0.7 nm and widths up to 6 nm were found to grow in the <110> Si directions. Larger rectangular 3-D islands (nanostructures) with heights ranging from 1.2 to 7.5 nm (depending on growth condition) and widths up to 50 nm were found interspersed amongst the nanowires. HRTEM and associated fast Fourier transform (FFT) analysis indicates that in addition to the expected hexagonal form (AlB2 type) of the DySi2, orthorhombic (GdSi2 type) and/or tetragonal (ThSi2 type) co-exist on the Si(100) substrates. Cross-sectional HRTEM reveals a number of different interfacial structures, with varying degrees of strain. The observed structures will be discussed in terms of the mismatch between the different observed phases and the Si(100) substrate. This work has been supported by the National Science Foundation grant number DMR-0305472.

5:00 PM
Phase Stability in $Ce_{1-x}Zr_xO_{2-y}$ Nanocrystals: Feng Zhang[1]; Chih-Hao Chen[1]; Jonathan C. Hanson[2]; Caliebe Wolfgang[2]; Richard D. Robinson[1]; Irving P. Herman[1]; *Siu-Wai Chan*[1]; [1]Columbia University, Matls. Sci. & Engrg. Dept. of Applied Physics & Applied Math., 1136 Mudd Bldg., MC 4701, 500 W. 120th St., New York, NY 10027 USA; [2]Brookhaven National Laborataory, Upton, NY USA

Cubic $Ce_{1-x}Zr_xO_{2-y}$ is the oxygen storage constituent in three-way catalyst and will loose its functionalities when it transforms to either the tetragonal or the monoclinic phase. It is therefore important to know the stability range of the cubic phase (c') of $Ce_{1-x}Zr_xO_{2-y}$. We employed x-ray diffraction (XRD), time-resolved high temperature XRD, transmission electron microscopy, Raman spectroscopy, and X-ray absorption near edge spectroscopy (XANES) for the investigation. With decreasing particle size, the c'-tetragonal phase boundary shifts to a higher zirconium concentration. A clear relationship between the phase stability of c' phase with Ce3+ concentration has been found from Ce LIII edge in XANES. We will report different phase stability ranges for nanoparticles in different redox environments as well as the differences from those predicted by traditional bulk phase diagrams.

Refractory Metals in Electronic Applications: Joint Session with Texture and Microstructure in Thin Films and Coatings: Texture and Thin Films

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee, EMPMD-Thin Films & Interfaces Committee, SMD-Refractory Metals Committee, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Gary A. Rozak, Fabricated Products, Cleveland, OH 44117 USA; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102 USA

Tuesday PM Room: 3010
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Christopher Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA

2:00 PM
Development of Texture in Asymmetric Processing of Tantalum Plate: *David P. Field*[1]; Jeff Yanke[1]; Eva V. McGowan[1]; Christopher A. Michaluk[2]; [1]Washington State University, Mechl. & Matls. Engrg., Box 642920, Pullman, WA 99164-2920 USA; [2]Williams Advanced Materials, 2306 Cassard Cir., Gilbertsville, PA 19525 USA

Sputtered tantalum and TaN are employed as barrier layers in modern integrated circuits to enable reliable use of Cu as an interconnect material. The directional properties of sputtering Ta can result in non-uniform film thicknesses (from heavily textured plate) and unpredictable sputtering rates (from plates with through thickness texture gradients). This results in film thicknesses larger than necessary because of the sputtering being unpredictable. This presentation reports on an effort to increase textural and grain size uniformity in Ta by deforming the plate under conditions that simulate asymmetric rolling. This is accomplished by using a channel die configuration with uneven friction on the top and bottom platens so a strong shear component is added to the plane strain condition enforced by the channel dies. Results indicate a trend towards less severe texture banding and more uniform structure in the plate processed by asymmetric friction conditions.

2:25 PM
Control of Texture in Plates for Sputtering: *Peter R. Jepson*[1]; [1]H.C. Starck Inc, Fabricated Products, 45 Industrial Pl., Newton, MA 02461 USA

For some applications, achievement of excellent uniformity of thickness of a sputtered film is important: for example, tantalum when used as a barrier layer under copper interconnects in integrated circuits. The crystallographic texture of the target plate affects sputter yield and must therefore be carefully controlled, if excellent uniformity is to be achieved. H.C. Starck Inc. has worked since 2000 to establish itself as the technology leader in the supply of plate for this application, and in so doing has studied the effects of target texture on the thickness of the thin film, and also developed manufacturing processes which consistently produce top-quality product at a competitive price. Innovative R&D techniques (Finite Element Modelling, and Electron Back-Scattering Diffraction) will be described, as well as the important features of the products which have been launched since 2002, including crystallographic texture control.

2:50 PM
Tantalum Angular Sputter Emission Distributions: *Charles E. Wickersham*[1]; Zhiguo Zhang[1]; [1]Cabot Corporation, Thin Films, 1275 Kinnear Rd., Columbus, OH 43212 USA

Sputtering target crystallographic orientation affects the film deposition uniformity by controlling the emission trajectories of the sputtered atoms. This effect is well known having been first reported by Wehner et al. in 1956. More recently, this effect has been observed to affect film uniformity for magnetron sputtered aluminum alloy films. Understanding the relationship between the crystallographic orientation of the sputtering target and the emission trajectory for the sputtered atoms is important for being able to accurately model film thickness uniformity and step coverage. In this paper we report measurement of the relationship between the crystallographic orientation of tantalum single crystal sputtering targets and the tantalum atom sputter trajectory. We use a quartz crystal monitor to detect sputter atom flux as a function of angle. The intensity of the sputtered atom flux as a function of angle show that the bcc tantalum single crystals strongly emits sputtered atoms along the <111> close packed direction as well as the <100> direction. Emission from the <110> direction is significantly less than that from the <111> direction. This fundamental property of tantalum sputtering is then used to accurately model thin film uniformity produced by magnetron sputtering from polycrystalline tantalum sputtering targets with strong (110), (111) and (100) orientations.

3:15 PM
Effect of Tungsten Sputtering Target Density on PVD Thin Film Properties: *Chi-Fung Lo*[1]; Peter McDonald[1]; Darryl Draper[1]; Paul Gilman[1]; [1]Praxair Electronics, Deposition Matls., 542 Rte. 303, Orangeburg, NY 10962 USA

Tungsten is commonly used for diffusion barrier, via and gate materials in semiconductor devices. Using physical vapor deposition (PVD), tungsten is deposited on to the silicon substrate. In order to achieve the desired film properties and minimize particle generation, control of the sputter target properties, which include density, purity, grain size and orientation, are essential. This study focused on the effect of sputtering target density on the thin film properties and defect generation. By controlling the powder sintering process, three sputtering targets from 80% to 100% of theoretical density were prepared. Using a sputtering tool, thin films were deposited on to the silicon wafers. The deposited films were then evaluated by four-point probe, XRD and SEM. In addition to film properties, the re-deposition behavior of the test targets during sputtering was also investigated by SEM. The re-deposited structures are known to be a potential particle source from the target. By monitoring the formatio n of re-deposition structure on the sputtered surface as function of target life, a correlation between target density and re-deposition behavior was established.

3:40 PM
MOCVD Ultra Thin Tungsten Nitride Films as Barrier Metal for Interconnect Applications: *Wei Pan*[1]; Robert Barrowcliff[1]; David Russell Evans[1]; Sheng-Teng Hsu[1]; [1]Sharp Labs of America, Inc., IC Process Tech., 5700 NW Pacific Rim Blvd., Camas, WA 98607 USA

This paper discusses the MOCVD processes, thin film properties, and barrier metal application for interconnects of the ultra thin W2N films deposited from a solid source W(CO)6 along with the gaseous NH3 through thermal decomposition. W(CO)6 vapor was carried into the deposition chamber by hydrogen to mix with NH3. The wafer was heated to the temperature between 350¢XC and 450¢XC, where the precursor vapor decomposed and reacted with NH3 to form W2N films. The as-deposited ultra thin W2N films, with thickness around 8nm, had resistivity as low as 275?Ýohm-cm and density as high as 17g/cm3. X-ray diffraction and X-ray reflectivity were used to characterize phase, density, and thickness. Growth kinetics was also studied. Standard bias-temperature-stress method was used to evaluate the barrier properties. Furthermore, the contact resistance of CVD Cu/CVD W2N/CVD Cu was measured on a Kelvin structure. Results indicate that CVD W2N has a great potential for barrier applications.

4:05 PM
The Effect of Texture of TiN Diffusion Barrier for Cu Metallization: Insoo Kim[1]; Dong Young Sung[1]; Min Gu Lee[1]; *No Jin Park*[1]; Beelyong Yang[1]; Jun Mo Yang[2]; Jung Kyu Ko[2]; [1]Kumoh National Institute of Technology, Sch. of Advd. Matls. & Sys. Engrg., 188 Shin Pyung Dong, Kumi, Kyung Buk 730-701 Korea; [2]Hynix Semiconductor Inc., Memory R&D Div., San 136-1, Ami-ri, Bubal-eub, Ichon-si, Kyoungki-do 467-701 Korea

TiN coated films have a good mechanical and conductivity properties, high thermal properties, and strong erosion and corrosion resistance. Therefore, TiN coated films have been used as a surface modification method in parts and as a diffusion barrier in semiconductors. The uniform and dense structure of thin films is influenced by the texture of films. It was good to have uniform and dense structure and bad to have an open columnar structure in TiN thin films. Therefore, the property of diffusion barrier of the TiN films in semiconductor also is related to the texture and microstructure of TiN coated layers. In this study, the relationships between the textures and the properties of TiN diffusion barrier for Cu metallization on SiO2/Si wafer semiconductor were investigated under different processing methods (PVD and MOCVD). The property of diffusion barrier for Cu metallization of RF sputtered (PVD) TiN is better than that of metal organic chemical vapor deposited (MOCVD) TiN thin films. Also the property of diffusion barrier for Cu metallization of PVD (111) textured TiN is better than that of PVD (100) textured TiN thin films on SiO2/Si wafer.

4:30 PM
Factors Affecting the Sputtering Performance of Magnetic Materials: *Christopher A. Michaluk*[1]; Henry L. Grohman[2]; David P. Field[3]; [1]Williams Advanced Materials, 2306 Cassard Cir., Gilbertsville, PA 19525 USA; [2]Williams Advanced Materials, 42 Mt. Ebo Rd. S., PO Box 1950, Brewster, NY 10509-8950 USA; [3]Washington State University, Dept. Mechl./Matls. Engrg., 239C Dana Hall, PO Box 642920, Pullman, WA 99164-2920 USA

D.C. magnetron sputtering relies on a magnetic field usually imparted by a fixed or rotating permanent magnet located behind the sputtering target, to trap free electrons within the plasma. The spiral trajectory of the contained electrons acts to increases the frequency of collisions with gaseous atoms, subsequently increasing the density of the plasma and the sputtering efficiency of the system. However, the magnetron sputtering of magnetic metals and alloys present unique challenges; the target's low magnetic permeability significantly hinders the magnetic flux from the permanent magnet to the plasma. This paper reviews how a combination of texture control and innovative target design is employed to enhance the sputtering performance of magnetic materials. Recently developed data revealing the correlation between global texture, characterized by Electron Backscatter Diffraction (EBSD), and the Pass-Through Flux (PTF) of a Co-4.5Zr-4.5Ta (CZT) alloy is also presented.

4:55 PM
Structure and Soft Magnetic Properties of Fe-Co-Ni-Based Multi-Component Thin Film: *Hung-Kai Chen*[1]; Shih-Hai Li[1]; Jenq-Gong Duh[2]; [1]National Tsing Hua University, Dept. of Engrg. & Sys. Sci., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan; [2]National Tsing Hua University, Dept. of Matl. Sci. & Engrg., 101 Sec.2 Kuang-Fu Rd., Hsinchu 300 Taiwan

Soft magnetic with suitable uniaxial anisotropy and high saturation magnetization are required for the high frequency application. Multi-components of Fe-Co-Ni-based soft magnetic thin films were deposited on the Si substrate by RF magnetron sputtering with different Ar/N_2 ratios at room temperature. The composition, crystal structure and surface morphology were analyzed by using electron probe microanalyzer (EPMA), X-ray diffraction (XRD), atomic force microscope (AFM) and magnetic force microscope (MFM). Without nitrogen doping, the domain distribution of the magnetic thin film arranged orderly and the domain thickness was about 1μm. The effect of N_2 content in the thin film on the magnetic properties was further discussed. Magnetic properties, such as saturation magnetization and coercivity, were determined with a vibrating sample magnetometer (VSM) and Magneto-Optics Kerr Effect (MOKE). The saturation magnetization (M_s) of the un-doped magnetic thin film is around 1.3T. It is expected that the derived magnetic thin film is a promising candidate for using in high frequency inductor.

5:20 PM
Growth Processes, Structure Peculiarities and Intergranular Magnetic Interaction in Electrodeposited Magnetic Nanostructures: *Vladimir Grigor'evich Shadrow*[1]; Anatolii Vasil'evich Boltushkin[1]; Ludmila Vas'vna Nemtsevich[1]; [1]Academy of Science, Inst. Solid State Physics, P. Brovki,17, Minsk, Belrus 220072 Belarus

Growth processes, structure peculiarities and magnetic properties of electrodeposited magnetic nanostructures have been investigated by means of EM,XRD,AFM,VSM and AGFM. Mechanisms of columnar and fine grained structure formation, regularities of content modulated FeCu and CoCu nanophase particles formation in the pores of aluminium anodic oxide are proposed, which accounts for the observed structure peculiarities.Intergranular magnetic interaction and magnetization reversal processes are investigated through the remanence and delta M curves analysis, magnetic viscosity measurements as well as rotational hysteresis loss and angular variations of hysteresis parameters measurements. The influence of the intergranular interaction on magnetic characteristics of the above model structures is discussed as well as evaluation of structural and magnetic films inhomogenities on the basis of the above techniques.

Shape Casting — The John Campbell Symposium: Structure and Properties

Sponsored by: Light Metals Division, LMD-Aluminum Committee, MPMD-Solidification Committee
Program Organizers: Murat Tiryakioglu, Robert Morris University, Moon Township, PA 15108 USA; Paul N. Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday PM Room: 2008
February 15, 2005 Location: Moscone West Convention Center

Session Chair: Qigui G. Wang, General Motors, Advd. Matls. Engrg., Pontiac, MI 48340 USA

2:00 PM
Effect of Oxide Films and Other Defects on the Fatigue Properties of Aluminium Castings: *Qigui G. Wang*[1]; Paul N. Crepeau[1]; John R. Griffiths[2]; Cameron J. Davidson[2]; [1]General Motors, Matls. Engrg., Powertrain, M/C 483-710-251, 895 Joslyn Ave., Pontiac, MI 48340 USA; [2]CSIRO Manufacturing Science and Technology, PO Box 883, Kenmore, QLD 4069 Australia

The fatigue properties of aluminum castings strongly depend on casting defects including oxide films and other defects. In this paper, the effect of oxide films on the fatigue properties of the most commonly used sand-cast, lost-foam-cast, squeeze-cast, and semi-solid-cast aluminum alloys are reviewed and also compared with the effect of porosity in these castings. In the presence of defects, the fatigue life of these alloys is dominated by defect sizes and the effect of other microstructural features becomes negligible on crack initiation. In comparison with pores of same size, oxide films have a less adverse effect on fatigue life.

2:25 PM
Estimating Ideal Tensile Properties of Cast Al-7%Si-0.6%Mg Alloys via Hybrid Mechanical Testing: *Murat Tiryakioglu*[1]; [1]Robert Morris University, Engrg., 6001 Univ. Blvd., Moon Twp., PA 15108 USA

Cast Al-Si-Mg alloys are in-situ metal matrix composites used in a variety of applications. During tensile deformation, cracks are initiated at major structural defects, such as porosity and bifilms. In the absence of major structural defects, however, Si particles in these alloys crack during tensile deformation. When damage to Si particles reach a critical value, fracture occurs. It has been suggested n the literature that ideal properties of these alloys are obtained when there is no damage to Si particles, as in compression. A new hybrid testing method built on the Kocks-Mecking work hardening model, that uses the entire tensile curve and Rockwell B hardness number will be introduced. The effect of Si particle shape and size on the ideal properties obtained by the new method will be discussed.

2:50 PM
The Influence of Strontium and TiB2 Addition on the Formation of Porosity in Aluminum Castings and its Effect on Final Fatigue Properties: *Peter D. Lee*[1]; Trevor C. Lindley[1]; [1]Imperial College London, Matls., Prince Consort Rd., London SW7 2BP UK

The influence of altering both the processing conditions and minor alloying additions upon the formation of microporosity in aluminium castings was investigated using traditional metallographic and microtomographic techniques. The confounding effects of eutectic modifiers (Sr), grain refiners (TiB2) and solidification rate upon the size, distribution and complex three dimensional shape of the pores formed

was characterised. For each condition tensile and S-N fatigue properties were measured in the heat treated condition. Using scanning electron microscopy, fractographic examination was performed, revealing that the fatigue cracks initiated at pores in almost all cases. The final fatigue performance was correlated to changes in microporosity and hence to casting conditions. The results of the study illustrate that the interaction of strontium and TiB2 is complex, with the as-cast pore morphology changing during heat treatment.

3:15 PM
A Study of Crack Initiation Sites in High Cycle Fatigue of 319 Aluminum Alloy Castings: *Glenn E. Byczynski*[1]; [1]Nemak, Canadian Ops., 4655 G N Booth Dr., Windsor, ON N9C 4G5 Canada

High cycle fatigue performance of aluminum castings is of high importance for the casting designer. Several recent fatigue studies in Al-Si-Mg alloys have found that in addition to porosity, oxide films are viable fatigue crack initiators. The current study analyzed the high cycle fatigue performance of 319 (Al-Si-Cu-Mg) alloy test bars produced in filtered and unfiltered gravity poured sand moulds. The fatigue life was found to be related to the size of defect acting as the crack initiation site. Scanning electron microscopy and energy dispersive spectroscopy were used to identify the nature of crack initiation sites. A linear elastic fracture mechanics crack growth model was found to be particularly successful at predicting the life of fatigue samples that initiated at oxide films. Having crack-like geometry, and a minute crack tip radius, oxide films effectively acted as preformed cracks. Consequently there was an absence of crack nucleation time, explaining the correlation of predicted propagation life to fatigue life.

3:40 PM Break

3:50 PM
The Effect of Si Content on the Ductility of Al-Si-Cu-Mg-(Fe,Mn) Casting Alloys: *Carlos H. Caceres*[1]; John A. Taylor[1]; [1]University of Queensland, Co-op. Rsch. Ctr. for Cast Metals Mfg., Sch. of Engrg., Brisbane, QLD 4072 Australia

The strength-ductility behaviour of a number of experimental Al-Si-Cu-Mg-(Fe,Mn) alloys has been compared to assess the effect of different levels of Si, Cu and Fe/Mn. Increased Si content improves the ductility of most alloys. This is particularly significant when the Fe content is high, but the effect is also important when either Cu alone or both Cu and Fe are present at high levels. A study of the intermetallics formed at different Si levels shows that the length of b-Al5FeSi platelets is reduced at high levels of Si. Similarly, Cu-rich intermetallics, which form extensive, interconnected particle clusters at lower Si content, appear as smaller, more isolated clusters at higher Si levels. It is thought that this refining of intermetallics leads to the observed increase in ductility at high Si content. Possible mechanisms for the refinement of the intermetallics during the solidification of high Si alloys are discussed.

4:10 PM
Hard Spot Defects in Al-Si Cast Alloys: *Xinjin Cao*[1]; John Campbell[2]; [1]Institute for Aerospace Research, Aeros. Mfg. Tech., 5145 Decelles Ave., Montreal, Quebec H3T 2B2 Canada; [2]University of Birmingham, Sch. of Metall. & Matls., Edgbaston, Birmingham B15 2TT England

Hard spot inclusions are one of the main machining defects encountered in cast aluminium alloys. The term "hard spot" designates an inclusion, generally of high hardness and may cause great trouble in machining operations. Basically there are mainly four types of "hard spot" inclusions in Al-Si-Mg cast alloys: oxides, intermetallics, refractory particles and cold drops. The oxides in Al-Si-0.4Mg cast alloys include Al2MgO4, Al2O3 and MgO. SiO2 inclusions are entrained sand or refractory material. Iron-rich inclusions are perhaps the most common of intermetallic hard spots in Al-Si alloys containing Fe and Mn. Other intermetallic hard spot inclusions are composed of TiAl3 and Ti(AlSi)2 in Al-Si-0.4Mg alloy containing Ti, and Al2Si2Sr or Al4Si2Sr in Al-Si-0.4Mg alloy containing Sr. Some intermetallic compounds may originate from the use or presence of grain refiners in the charges (added titanium boride/titanium carbide particles), or other nitrides, carbides, borides, etc. Cold drops are the small droplets of cast alloy themselves formed during excessively violent pouring.

4:30 PM
High Pressure Die Casting of Aluminium and Magnesium Alloys - Comparison of Microstructure Formation: *Hans I. Laukli*[1]; Otto Lohne[2]; Lars Arnberg[2]; [1]Hydro Aluminium, R&D Matls. Tech., Sunndalsøra 6600 Norway

Cold chamber high pressure die casting, (HPDC), is an important commercial process for the production of complex near net shape aluminium and magnesium alloy castings. The solidification characteristics related to the process and the alloys control the formation of grains and defects. This again has a significant impact on the mechanical properties of the castings. Significant amounts of pre-solidified metal flow into the die cavity during cold chamber HPDC of aluminium and magnesium alloys. The solid fraction commonly consists of single crystals which are termed externally solidified crystals (ESCs). The semi-solid metal fills the die cavity and, eventually, the microstructure consists of coarse ESCs and fine grains solidified in-situ. The prevalence of ESCs in the castings takes a variety of distributions and morphologies. Additionally, banded defects commonly form in the final stages of die filling. The nature of the bands depends on different parameters but tend to consist of segregation in Al die castings and porosity (and segregation) in Mg die castings. In the present work the microstructure formation in HPDC of aluminium (A356) is compared with magnesium (AM60B). The major similarities and differences are discussed with particular focus on the physical properties and solidification behaviour of the two alloys.

4:50 PM
Influence of Ca, Be and Mn on the Structure and Properties of 356 Alloy: *Sreeja Kumari*[1]; Raman Marimuthu Pillai[1]; Ballembettu Chadrasekhar Pai[1]; [1]Regional Research Laboratory (CSIR), Metals Procg. Div., Industl. Est. PO, Pappanamcode, Trivandrum, Kerala 695 019 India

Iron, the most harmful impurity in cast aluminium alloys, forms platelet iron intermetallic phase (β), which is detrimental to the mechanical properties and fracture toughness. Among the various methods available to neutralize its ill-effects, trace elements addition is commonly practiced. Effects of Ca, Be and Mn additions on the microstructure and properties of 356 alloy containing 0.6% Fe have been investigated. The alloy without Ca, Be and Mn additions exhibits poor mechanical properties due to the presence of long intercepting platelet iron intermetallics. Addition of Ca (300 ppm) has modified the eutectic Si structure and reduced the size of platelike Fe-intermetallic phases, while Be (0.2%) and Mn (0.3%) have changed the platelet morphology to Chinese script form. Mechanical properties have been improved with Ca, Be and Mn additions. The mechanisms for the microstructural changes have also been discussed.

5:10 PM
Fluidized Bed Heat Treatment of Cast Al Alloys: *Sujoy Chaudhury*[1]; Diran Apelian[1]; [1]MPI - WPI, Matls. Sci. and Engrg., 100, Inst. Rd., Worcester, MA 01609 USA

Improved mechanical properties in cast Al alloys are often achieved through heat treatment comprising of solution heat treatment, quenching, and ageing successively. With the impetus to be cost effective, it is imperative to reduce the long heat-treating times needed (i.e., up to 15-20 hours) without any reduction in performance. Fluidized beds provide an attractive heat treating technology for cast components with more efficient energy transfer and thereby reducing the net heat treatment time and enhance productivity. However, typical fluidized bed units are batch systems; therefore, further reductions in cycle time may be possible by using in-line continuous fluidized beds. In this paper, we will review the fundamentals of fluidized beds, highlight their advantages and examine applications of fluidized beds to heat treat cast Al-Si-Mg, and Al-Si-Cu-Mg alloys. Mechanical properties data along with the resultant microstructure as a result of extensive trials with fluidized bed technology will be reviewed and discussed.

Superalloys and Coatings for High Temperature Applications: Ceramic Materials for TBCs

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), High Temperature Materials Committee of IoM3

Program Organizers: Roger C. Reed, University of British Columbia, Department of Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Qiang (Charles) Feng, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Tim Gabb, NASA Glenn Research Center, Cleveland, OH 44135 USA; John Nicholls, Cranfield University, Bedfordshire MK43 OAL UK; Bruce A. Pint, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Tuesday PM — Room: Nob Hill A/B
February 15, 2005 — Location: San Francisco Marriott

Session Chairs: Sammy Tin, University of Cambridge, Dept. Matls. Sci. & Metall., Cambridge CB2 3Q UK; Roger C. Reed, University of British Columbia, Dept. of Metals & Matls. Engrg., Vancouver, BC V6T 1Z4 Canada

2:00 PM Invited
Stability Issues in Current and Emerging TBC Materials: Stephan Kraemer[1]; Ashutosh S. Gandhi[1]; Noemi R. Rebollo[1]; Rafael M. Leckie[1]; Felicia M. Pitek[1]; *Carlos G. Levi*[1]; [1]University of California, Matls. Dept., 1355 Engrg. II, Santa Barbara, CA 93106-5050 USA

Insulating layers for present thermal barrier systems comprise a metastable t' structure with composition ZrO_2-7.6%$YO_{1.5}$ (7YSZ) which exhibits remarkable stability under current operating conditions, but are deemed inadequate to satisfy the demands of upcoming engine designs. The current 7YSZ can also be severely degraded by chemically aggressive environments, e.g. those associated with molten silicate and/or sulfate/vanadate deposits. Emerging alternate compositions involve co-doping 7YSZ with RE oxides or its replacement by RE zirconates. Preliminary evidence suggests that both approaches can enhance thermal resistivity as well as the morphological stability of the microstructure at higher temperatures. As new oxides are incorporated, however, concerns arise about the phase stability of the coatings, their response to corrosive environments and their thermo-chemical compatibility with the underlying thermally grown alumina. These issues are examined, highlighting the thermodynamic and kinetic fundamentals determining the long-term stability of the coatings.

2:30 PM
Development of Ion Plasma Deposition Process for Turbine Coating Applications: *Don M. Lipkin*[1]; Joseph Rigney[2]; [1]GE Global Research, Ceram. & Metall. Tech., Bldg. K1, Rm. MB207, One Rsch. Cir., Niskayuna, NY 12309 USA; [2]GE Transportation, Aircraft Engines, 1 Neumann Way, MD M85, Cincinnati, OH 45215 US

The Ion Plasma Deposition process has proven to be a robust, versatile approach to depositing a broad range of coatings for use in high-temperature applications. We describe GE's experiences, including the application of Ion Plasma Deposition to advanced turbine airfoil coatings. The process is shown to exhibit excellent capability to transfer multi-component alloys with reproducible chemistry, coating thickness, adhesion, and microstructural quality. In developing the Ion Plasma Deposition process, GE adapted the cathodic arc process widely used for thin-film tool and decorative coating applications, thus enabling practical deposition of thick, complex-alloy metallurgical coatings. Using coating hardware specifically designed to optimize the uniformity of coverage on complex-geometry components, these modifications have proven key to the demonstration of Ion Plasma Deposition for turbine coating applications. Several examples of coatings deposited using the Ion Plasma Deposition process will be described, with special emphasis on the high-pressure turbine airfoil coatings.

2:55 PM
Cold-Shock Spallation Mechanism in Thermal Barrier Systems Subject to CMAS-Infiltration: *Sabine Faulhaber*[1]; Chris Mercer[1]; Anthony G. Evans[1]; [1]University of California, Matls. Dept., Santa Barbara, CA 93106-5050 USA

Three principle mechanisms govern the spallation of thermal barrier coatings (TBC) - stresses due to the formation of thermally grown oxide (TGO), erosion due to particle impact and foreign object damage (FOD) and infiltration with CMAS (calcium-magnesium-alumino-silicate) at high temperatures. A new mechanism for the spallation of the CMAS infiltrated layers has been identified: it involves cold-shock during shut-down of the engine. Calculations predict a critical penetration thickness that, if exceeded, makes the TBC layer susceptible to cold-shock in the presence of a vertical separation. The model is consistent with the experimental observations of an airfoil removed from service, gained from scanning electron microscopy (SEM) on metallographic cross-sections.

3:20 PM
Non-Destructive and Microstructural Characterization of Thermal Barrier Coatings: *Yongho Sohn*[1]; Balaji Jayaraj[1]; Sankar Laxman[1]; Barb Franke[1]; David Miranda[1]; Jaewon Byeon[1]; Vimal H. Desai[1]; [1]University of Central Florida, AMPAC & MMAE, Box 162455, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA

The durability and reliability of thermal barrier coatings (TBCs) play an important role in the service reliability and maintainability of hot-section components in advanced turbine engines for aero and utility applications. Photostimulated luminescence spectroscopy (PSLS) and electrochemical impedance spectroscopy (EIS) are being concurrently developed as complimentary non-destructive evaluation (NDE) techniques for quality control and life-remain assessment of TBCs. The evolution the residual stress, polymorphic constituents and impedance of TBC constituents with an emphasis on the thermally grown oxide were examined by NDE techniques as a function of thermal cycling. Results from PSLS and EIS were correlated to the microstructural development of TBCs examined by using a variety of microscopic techniques including focused ion beam in-situ lift-out, transmission and scanning transmission electron microscopy (TEM and STEM). TEM/STEM revealed many features of microstructural development that can influence the failure mechanisms of TBCs, and in turn affect the durability and reliability.

3:45 PM Break

4:05 PM
Simulation of the High Temperature Indentation of TBC with Columnar Microstructure: *Tao Xu*[1]; [1]University of California, Matls. Dept., Santa Barbara, CA 93106 USA

The phenomena governing the durability of thermal barrier coatings (TBCs) are affected by their high temperature mechanical properties: especially the mechanisms of material removal upon particle impact. Some high temperature properties can be explored using an impression test. The utility of the test is contingent upon a method for deconvoluting aspects of the stress/strain response from load-displacement measurements. A numerical procedure having this attribute is described, and applied to TBCs with a columnar microstructure. The method elucidates the extent of the plastic deformation and densification as well as the column distortions caused by the impression. It is also capable of exploring the deformation heterogeneities observed experimentally, such as shear bands, by embodying salient constituent properties, such as the column width, contact friction, and inter-columnar porosity. Comparisons with measurements provide some understanding of the plastic response of several thermal barrier systems.

4:30 PM Invited
Thermal Barrier Coatings Under In-Service TMF Conditions: *Jeffery W. Brooks*[1]; Bernd Vermeulen[1]; [1]QinetiQ Ltd., Rm. 2008, Bldg. A7, Cody Tech. Park, Ively Rd., Farnborough, Hampshire GU14 0LX UK

Thermal Barrier Coatings (TBC) are fast becoming mainstream technology for use in gas turbine engines. The work to be presented assesses the effects of thermo-mechanical fatigue (TMF) on the degradation of the TBC. The inherent problem, when using standard induction heating during TMF tests, is that the specimen is heated from the inside and hence the temperature profile seen is the reverse of that experienced in service. An alternative heating system is described, which achieves temperature distributions close to in-service conditions. The test cycle profiles were derived from finite element analyses of a typical gas turbine blade using an anisotropic deformation code to compute the substrate strain field. The TMF tests were conducted with single crystal CMSX4 specimens coated using plasma vapour deposition (PVD) with an MCrAlY bond coat and a zirconia based TBC. The degradation and adhesion of the TBC, assessed using microscopy, will be compared with results from TMF tests performed using standard induction heating.

4:55 PM Invited
Thermal Barrier Effect and Thermal Cyclic Behavior of YSZ Coating with Rare Earth Oxide Addition: *Huibin Xu*[1]; Shengkai Gong[1]; [1]Beijing University of Aeronautics and Astronautics, Sch. of Matls. Sci. & Engrg., Xueyuan Rd. 37, Beijing 100083 China

The present TBC system with Y2O3 stabilized ZrO2 (YSZ) top coat exhibits an excellent high temperature performance, however, its thermal barrier effect has been greatly limited due to the thermal conductivity of YSZ top coat prepared by EB-PVD. Low conductivity thermal barrier coatings have been increasingly demanded to obtain thin coating configurations which still could achieve sufficient temperature reductions at higher engine operating temperatures. In this study, lattice distortion has been calculated by ab initio method on the system of YSZ added by various rare earth oxides to study the change of atomic oscillation frequency and thermal conductivity as well. Furthermore, some of YSZ with rare earth oxides have been deposited onto MCrAlY bond coat by EB-PVD method. The thermal barrier effect and thermal cyclic behaviors have been investigated as a function of the amount of the different kinds of rare earth oxides.

5:20 PM
Nondestructive Evaluation of Thermal Barrier Coatings Thermal Properties: *Ted D. Bennett*[1]; Fengling Yu[1]; [1]University of California, Mechl. & Environml. Engrg., Santa Barbara, CA 93106 USA

A nondestructive technique for determining thermal properties of thermal barrier coatings is developed. The measurement reveals both the thru-plane thermal diffusivity and the effusivity contrast between the film and substrate. From these measurements both the thermal conductivity and volumetric specific heat of the film may be determined. Because the measurement is nondestructive, this technique may be applied to evaluate film properties over the lifetime of a serviceable part. The measurement can be used to resolve the effects of structural variability in the film, high temperature sintering of pores, and localized delamination of the film from the substrate.

5:40 PM
Enhancing the Environmental Stability of Thermal Barrier Coatings: *Felicia Marie Pitek*[1]; Stephan Kraemer[1]; Carlos G. Levi[1]; [1]University of California, Matls. Dept., Santa Barbara, CA 93106-5050 USA

The hot corrosion of yttria stabilized zirconia (7YSZ) thermal barrier coatings by molten sulfate/vanadate deposits is discussed. The ensuing reaction of V_2O_5 with Y leads to the depletion of stabilizer from the initially "non-transformable" t' phase and rendering it susceptible to the deleterious monoclinic transformation upon cooling. The presence of a stable non-transformable tetragonal region in the ZrO_2-$YO_{1.5}$-$TaO_{2.5}$ phase diagram provides an opportunity to enhance the corrosion resistance of the coating. In principle, the addition of Ta reduces the activity of Y in the solid solution and presumably its susceptibility to reaction. Moreover, coating compositions can be selected to be tolerant to depletion without de-stabilization. Selected Y and Y+Ta doped compositions were made by precursor methods, consolidated into pellets and tested at 900°C using a Na_2SO_4-30mole%$NaVO_3$ corrodent. The resulting changes in microstructure and composition were characterized by XRD, SEM and TEM to compare the relative stabilities and differences in corrosion mechanisms.

Surface Engineering in Materials Science - III: Thin Films

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Craig Blue, Oak Ridge National Laboratory, Materials Processing Group, Metals and Ceramic Division, Oak Ridge, TN 37831 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Tuesday PM Room: 2022
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sudipta Seal, University of Central Florida, Advd. Matls. Procg. & Analy. Ctr. & Mechl., Oviedo, FL 32765-7962 USA; Yip-Wah Chung, National Science Foundation, Civil & Mechl. Sys. Div., Arlington, VA 22230 USA

2:00 PM Invited
Stable Four Terminal Solar Cells Using Thin Film Silicon Technology: *Arun Madan*[1]; [1]MVSystems, Inc., 17301 W. Colfax Ave., Ste. 305, Golden, CO USA

For photovoltaics (PV) to be competitive with other forms of energy generation, a consensus is that thin film technologies could provide the route to lower costs. One of the most likely contender is thin film Silicon which has been commercialized (with efficiency,h, of 6-8%) by several companies. However, the intrinsic light induced degradation in amorphous Silicon (a-Si:H), remains an impediment. The degradation is strongly dependant on the thickness of the device used and can be reduced by the use of multi-junction (MJ) at the expense of complexity in fabrication. MJ type devices use several cells stacked on top of each other with differing band gaps (and thickness) to absorb a wider portion of the solar spectrum (e.g. a-SiH/a-SiGeH/a-SiGeH). As this two terminal (2-T) MJ device structure requires the same current from each constituent cell, it necessitates the use of relatively thick a-SiH junctions (~2000A) and the device generally degrades by ~20%. Further, the fabrication of a-SiGe:H requires GeH4 gas which is prohibitively expensive and since the gas utilization during production is normally <10%, the cost reduction of such PV panels may be difficult to realize. Hence the use of 2-T MJ solar cell with stable micro-(or rather nano-) crystalline Si (nc-SiH) as the bottom cell and a-Si:H as the top cell is attracting attention (termed as "micro-morph"). Such MJ (or tandem) solar cells can produce an initial h of ~14.5% in a small area (3cm2) and ~12% in large area modules. However, this structure also contains a thick (~ 4000A) a-Si:H junction (due to the current matching) and as a result majority of the power (~ 70%) emanates from the unstable thick a-Si:H portion with an inevitable degradation under light. We have developed a simpler 4 terminal (4-T) thin film Si based MJ solar cell configuration in which the current matching constraint is released from each constituent cell, e.g. two cells (a-SiH and stable low band gap material, such as nc-SiH)) are separated via an insulator. This allows the use of ultra-thin (<1000A) a-Si:H solar cell where instability is no longer an issue. This stable solar 4-T MJ design, has the potential to attain h >16%. We discuss the steps that are needed to go from the current efficiency of ~10% to >16%. We discuss the use of a modified pulsed PECVD (plasma enhanced chemical vapor deposition) technique, which provides a powerful way to improve the nc-SiH materials by altering the growth via a layer-by-layer technique. With an improvement in grain size to > 1000-2000A, as in fine grained polycrystalline-Si, that higher carrier mobility's (especially for holes) could be attained leading to longer minority carrier diffusion length and to a substantial increase in the open circuit voltage (from 480 mV to >650mV) thus opening the route to high efficiency (>16%) low cost stable thin film Si solar cells.

2:25 PM Invited
Coatings for Extreme-Condition Applications: *Yip-Wah Chung*[1]; [1]National Science Foundation, Civil & Mechl. Sys. Div., 4201 Wilson Blvd., Rm. 545.25, Arlington, VA 22230 USA

Coatings used in certain mechanical components operate under extremely demanding conditions. For example, coatings used in protecting cutting tools have to endure high contact stresses and tempera-

tures; nanometer-thick overcoats in computer hard disk drives have to provide the necessary wear and corrosion protection for extended periods; bearings and gears operate under high cyclic stresses, and the applied coatings must be fatigue-resistant and provide low friction/traction performance. This talk presents selected examples to illustrate some general principles in the selection and synthesis of such protective coatings.

2:50 PM
Enhanced Adhesion of Polyimide with Sputtered Copper by Argon Plasmas for Microelectronic Flex Substrates: *Yung-Sen Lin*[1]; Huang-Ming Liu[1]; Hsuan-Ta Chen[1]; [1]Feng Chia University, Dept. of Chem. Engrg., No. 100, Wenhwa Rd. Seatwen Dist., Taichung, Taiwan 407 China

Enhanced adhesion of polyimide films such as Kapton E(N) and Upilex S with sputtered copper by argon plasmas were investigated. Peel tests demonstrate this improvement. The enhanced adhesive strengths of sputtered coppers to polyimide films by argon plasmas were strongly affected by surface morphology and surface energy of polyimide films. The surface morphology of polyimide film was investigated by atomic force microscopy (AFM) and the surface energy of the polyimide film was calculated by the sessile drop method that indicated the surface roughness and the surface energy of polyimide films were much increased by argon plasmas that results in much increased peel strengths of sputtered coppers to polyimide films. Electron spectroscopy for chemical analysis (ESCA) observed the increased surface energy on polyimide films by argon plasmas were due to the increased surface compositions of O and N and the increased chemical bonds of C-O, C=O and C-N.

3:05 PM
Magnetic Semiconductor Thin Films - Magnetic and Transport Properties: *Lidia Jadwiga Maksymowicz*[1]; Maria Lubecka[1]; Beata Teresa Ciêciwa[1]; Zbigniew Stanislaw Sobkow[1]; Rita Szymczak[2]; Marek Sikora[3]; Czeslaw Kapusta[3]; [1]University of Science & Technology, Dept. of Elect., al. Mickiewicza 30, Kraków 30-059 Poland; [2]Polish Academy of Sciences, Inst. of Physics, al.Lotników 32/46, Warszawa 02-668 Poland; [3]University of Science & Technology, Dept. Solid State Physics, Reymonta 19, Kraków 30-059 Poland

Thin films of magnetic semiconductor (Cd1-yIny)[Cr2-2xIn2x]Se4 have been investigated as the perspective element of near-infrared detector. It is soft magnetic material, the energetic structure is modyfied by In dilution level and magnetic interaction. Also the increase of lattice parameter and changes of the local atomic environments with increasing the amount of In is detected. For CdCr2Se4, the magnetic state with reentrant transition (REE) is achieved. When In substitites Cr or Cd, we have the spin glass state (SG). The more complex type of magnetic ordering, the randomly canted (RC) was found when In substitutes Cr and Cd. The basic photoconductivity parameters as spectral voltage responsivity and detectivity were determined by lock-in measurement technique. It was found that for thin films in the state with REE the value of detectivity is the same order as for semiconductor materials.

3:20 PM
Surface Modification Through Inter Electrode Mass Transfer - A Phenomenon Intrinsic to Electro Discharge Machining: *T. A.J. Reddy*[1]; S. R.M. Voleti[1]; N. N. Ramesh[1]; [1]Osmania University, Mechl. Engrg., Coll. of Engrg., Hyderabad, Andra Pradesh 500 007 India

A significant observation in electro discharge machining is the occurrence of inter electrode material transfer. This phenomenon can be exploited for surface modification by suitable selection of process parameters, which can promote significant diffusion of selected electrode material into the machined surface for improved tribological properties. Microscopic analysis of such processed surface exhibits the presence of hard resolidified stratum with significant diffusion of electrode material revealed from Energy Dispersive X-ray Analysis. Consequently the micro hardness of resultant surface also increases significantly. Shaped electrodes fabricated by powder metallurgy technique and wire electrode with coating of selected materials are more effective in such mass transfer along with positive and negative polarity respectively. This increases electrode erosion and higher diffusion of electrode material into the processed surface. A major attraction of this approach is the simultaneous occurrence of machining and surface modification.

3:35 PM
Mechanically Induced Modification of an Al Surface and the Synthesis of Ceramic Coatings by Mixing in SiO_2 Particles: *Aghasi R. Torosyan*[1]; Laszlo Takacs[2]; Nshan H. Zulumyan[1]; Ashot A. Tataryan[1]; [1]National Academy of Sciences, Inst. of Gen. & Inorganic Chmst, 2-tup., Argutyan St. 10, Yerevan, Yerevan 375051 Armenia; [2]University of Maryland, Dept. of Physics, 1000 Hilltop Cir., Baltimore, MD 21250 USA

Surface modification of an Al plate and its interaction with amorphous and crystalline SiO2 powders during mechanical processing has been investigated. The Al plate was placed into a vibratory ball mill where it was exposed to the impact of the balls and mechanochemical interaction with SiO_2 powder for intervals of 20, 40, 60 and 120 min. Heat treatment at temperatures from 300°C to 600°C was applied to induce or continue the chemical reaction between the Al substrate and the SiO_2 particles forced into its surface layer. The mixing of the SiO_2 powder particles into the substrate, the evolution of the microstructure and the progress of the mechanochemical reaction between Al plate and SiO_2 were investigated by XRD and SEM. It was established that the products depend on the structural modification of the used SiO_2.

3:50 PM Break

4:00 PM Invited
Expansive Breadth Surface Engineering to Revolutionize the Energy Industry: *J. DeBarro*[1]; K. E. Rea[2]; [1]Mitsubishi Power Systems, Orlando Service Ctr., 2287 Premier Row, Orlando, FL 32809 USA; [2]University of Central Florida, AMPAC, MMAE, 4000 Central Florida Blvd., Orlando, FL 32816 USA

The materials required for the energy systems industry has and will be dominated with the modification of surfaces or surface science engineering. With the principal requirements of the materials' properties constantly shifting to resist higher temperature regimes and more corrosive environments to augment energy generation and efficiency, the surfaces of the materials become the essential building block of the entire industry. Enabling the manipulation and control of the materials surface through the processes of plasma forming, heat treating, and combinatorial alloy materials is paramount for development and progression of the energy systems and turbine repair manufacturing. Plasma spray and its relative process High Velocity Oxygen Fuel (HVOF) have lead the way in surface engineering breakthroughs in the recent decades and are becoming increasingly automated for manufacturing. New techniques of process and manufacturing evaluation are essential for overall control of strenuous requirements with a broad capacity of revolutionizing the industry.

4:25 PM
Texture and Microstructure Characteristics of Aluminium Thin Film on Stainless Steel: *Zhongliang Shi*[1]; *Hualong Li*[1]; Reza Bateni[1]; *Jerzy A. Szpunar*[1]; [1]McGill University, Dept. of Mining, Metals & Matls. Engrg., Wong Bldg., 3610 Univ., Montreal, Quebec H3A 2B2 Canada

Aluminium thin film is ductile with good electrical and thermal conductivities. Particularly, it can be oxidized easily by artificial oxidation or anodizing process to provide an advantageous combination of a good conducting layer and a completely insulating layer which can be applied in integrated circuit technology, micromachining, optical devices and for the development of a rechargeable aluminium battery system. There are several processes available for coating aluminium on work pieces such as (1) thermal spraying coating; (2) hot dipping or galvano-forming; (3) roll binding; (4) PVD/CVD process, for example, sputter and plasma processes and (4) electrodeposition of aluminium. Except for electrodeposition process, any other techniques are rather expensive or run at high temperature or are limited by the arbitrary shape of work pieces. Aluminium coating obtained by electrodeposition has very good quality. However, the electrodeposition process of aluminium is much more complicated than many other metals from aqueous electrolyte as it is less noble than hydrogen, so it cannot be deposited from aqueous solution. In this paper, an organic nonaqueous electrolyte based on tetrahydrofuran with AlCl3 and LiAlH4 dissolved in is employed for electrodeposition of aluminium thin film on stainless steel at room temperature and its texture and microstructure on stainless steel substrate is characterized in details. The measured texture results show that the aluminium thin film has weak (210) and (111) fibre textures and their intensity maximum is 1.7. The observation of the microstructures under different current densities and coated time illustrates that nano-size aluminium particles (10~50nm) are packed together after initial nucleation and the obtained homogenous aluminium thin film is attached to the stainless steel substrate well and no cracks were observed in the film.

4:40 PM
Formation and Characteristics of Oxidation Film Produced by Heat Treatment of Ti and Ti Alloys: *Kimihiro Ozaki*[1]; [1]Advanced Industrial Science and Technology, Matl. Rsch. Inst. for Sustainable

Dvlp., Anagahora 2266-98, Shimoshidami, Moriyama, Nagoya 463-8560 Japan

An oxidation layer of a pure titanium and Ti alloys has interference colors. Especially, it is famous to make a design by an anodic oxidation method. The anodic oxidation layer has various colors, but the layer has disadvantage in erosion resistance and in heat resistance. An oxide film by heat treatment in the atmosphere has higher erosion resistance and heat resistance than that by anodic oxidation. However, the former has fewer kinds of color and worse controllability of the thickness than the latter. Then in this, the formation of oxide films produced by heat-treating Ti and Ti-Si alloys and characteristics of interference color of the film were investigated for obtaining stability and variety of color.

4:55 PM
Microstructures and Ferroelectric Properties of Barium Strontium Titanate Thin Films: Chen Hong Wei[1]; [1]University of Electronic Science & Technology of China, Inst. of Microelect. & Solid State Elect., Chengdu, Sichuan 610054 China

Barium strontium titanate (BST) thin films were prepared by RF magnetron sputtering. The microstructures and ferroelectric properties of Ba0.6Sr0.4TiO3 films were investigated. The composition of BST thin film with tetragonal perovskite structure was near to BST ceramic target. Nanometer-sized domains were observed by piezo-response force microscopy (PFM). The critical size is found to be between 28nm to 33nm from a multi-domain to a mono-domain. The dielectric constant-electric field curves and polarization hysteresis loops of BST films have been measured. At 1kHz the dielectric constant, tunability and dielectric loss of the Ba0.6Sr0.4TiO3 film with thickness 280nm are 562,25.6% and 0.016, respectively. The remanent polarization (Pr) and the coercive electric field (Ec) of the BST film are 1.2iC/cm2 and 42.9kV/cm at room temperature, respectively. The år-V and P-V curves show a voltage shift toward the negative side and asymmetry, which may be caused by asymmetric potential barriers at the upper and bottom interfaces.

5:10 PM
A Model for Dielectrics Nonlinearity of Ferroelectric Thin Films: Fu Chun Lin[1]; [1]University of Electronic Science & Technology of China, Inst. of Microelec. & Solid State Elect., Chengdu, Sichuan 610054 China

Dielectric nonlinearity is an important characteristic of ferroelectric thin films. Based on the characteristics of hysteresis loops and å-E curves in thin films, a novel model for dielectric nonlinearity is established. Many parameters, intensively influencing the ferroelectric properties of thin films, such as grain size, film thickness and the barrier height at the interface are involved. In this model, the barrier layers were modeled in terms of p-n junction equivalent circuits. The accuracy of the model predictions is quantitatively verified with the data from reference.

Texture and Microstructure in Thin Films and Coatings: Joint Session with Refractory Metals in Electronic Applications: Texture and Thin Films

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Texture & Anisotropy Committee
Program Organizers: David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Chris A. Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA; John E. Sanchez, Advanced Micro Devices, Sunnyvale, CA 94088 USA; J. A. Szpunar, McGill University, Department of Metallurgical Engineering, Montreal, Quebec H3A 2A7 Canada

Tuesday PM Room: 3010
February 15, 2005 Location: The Moscone Center

Session Chair: Christopher Michaluk, Williams Advanced Materials, Gilbertsville, PA 19525 USA

See Refractory Metals in Electronic Applications Symposium on page 214 for schedule.

The Armen G. Khachaturyan Symposium on Phase Transformation and Microstructural Evolution in Crystalline Solids: Session IV

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; John William Morris, University of California, Department of Materials Science and Engineering, Berkeley, CA 94720 USA

Tuesday PM Room: 3003
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Ken R. Elder, Oakland University, Physics, Rochester, MI 48309 USA; Jong K. Lee, Michigan Tech, MSE, Houghton, MI 49931 USA

2:00 PM Opening Remarks

2:05 PM Invited
Phase Transformations in Ferroic Materials: *David E. Laughlin*[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Ferroic materials are those materials which on cooling, transform from a high symmetry phase into a lower symmetry phase without any reconstruction of the underlying crystal structure. The low symmetry phase is composed of two or more orientation variants, or domains. An important additional feature of ferroic materials is that their domains can be transformed into each other by the application of an appropriate external field. Ferroic materials are being used extensively as sensors, actuators, shape memory alloys and in other "smart material" applications. In this paper an overview will be presented of the various phase transformations and symmetry changes that are involved in selected ferroic materials. A general classification of ferroic phase transformations in terms of symmetry changes will also be given. This research has been supported in part by the Data Storage Systems Center at CMU.

2:30 PM Invited
Thermodynamics and Evolution of Ferroic Domain Structures: J. X. Zhang[1]; Y. L. Li[1]; S. Choudhary[1]; *L. Q. Chen*[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 102 Steidle Bldg., Univ. Park, PA 16802 USA

One of Khachaturyan's seminal contributions to theoretical materials science is his mesoscopic elasticity theory developed about 35 years ago. It has been the basis for the prediction and simulation of precipitate morphologies and microstructures during various phase transformations. In this presentation, the applications of Khachatuyran's elasticity theory and phase-field approach to ferroic domain structures will be discussed. Emphasis will be on systems that are simultaneously ferroelectric and ferroelastic or ferromagnetic and ferroelastic. Examples to be discussed include PbZrxTi1-xO3 (PZT) piezoelectric thin films and the ferromagnetic shape memory alloy Ni2MnGa. While PZT is a primary ferroelectric and secondary ferroelastic, Ni2MnGa is both a primary ferromagnetic and a primary ferroelastic. Similar approach can be applied to domain structures of ferromagnetoelectric multiferroics that are simultaneously ferromagnetic and ferroelectric.

2:55 PM
The Strain-Induced Paramagnetic to Ferromagnetic in Intermetallic Compounds: *Ian Baker*[1]; Markus W. Wittmann[1]; [1]Dartmouth College, Thayer Sch. of Engrg., 8000 Cummings Hall, Hanover, NH 03755 USA

A strain-induced paramagnetic to ferromagnetism transition has been observed in a number of intermetallic compounds containing magnetic moment-bearing elements. This phenomenon has been quantitatively modeled in FeAl, using the local environment theory, based on the idea that the ferromagnetism arises from APB tubes, where Fe atoms can have >=3 like nearest neighbors. It is shown that the saturation magnetization, Ms, depends on both the Fe:Al ratio, the ternary atoms present and on the degree of deformation. The behavior of both

ternary FeAl-based alloys and of the L21-structured intermetallic compound Fe2AlMn will be explained by considering the site preferences of ternary atoms on the Fe and Al sublattices. TEM observations of APB tubes in lightly-strained single crystals of FeAl and Fe2AlMn will be presented. The effects of plastic strain on the magnetic properties of L12-structured Co3Ti are also outlined. Research sponsored by NSF grant DMR 9973977 and NIST grant 60NANB2D0120.

3:10 PM
Effects of Si Addition on the Microstructure and Magnetic Properties of Permalloys Fabricated by Melt Drag Casting: *Kyoung-Mook Lim*[1]; Chan-Gyung Park[1]; Jung Namkung[2]; Mun-Chul Kim[2]; [1]Pohang University of Science & Technology, Matls. Sci. & Engrg., San 31, Hyojadong, Namgu, Pohang, Kyungbuk 790-784 Korea; [2]Research Institute for Science and Technology, Casting Process Rsch. Team, San 31, Hyojadong, Namgu, Pohang, Kyungbuk 790-785 Korea

Effects of Si addition on the microstructure and magnetic properties of 79Ni-Fe based permalloys, fabricated by melt drag casting, has been investigated. The Permalloy strips of 50mm width obtaining various Si contents were successfully prepared by melt drag casting, which was newly applied to fabrication of the permalloy strips in this study. In order to understand the relationship between magnetic properties and Si contents, microstructure and texture were extensively analyzed by TEM and EBSD. Increasing Si contents improved permeability of the permalloys in low Si content region below 2% due to the enlarged grain size. In high Si content region more than 2%, the formation of Ni3Fe ordered phase was revealed remarkably, which caused the drastic reduction of permeability. In addition, the formation of Ni3Fe was promoted by higher temperature annealing at fixed Si content. From these results, the ordering kinetics of Ni3Fe in Si added permalloys will be discussed.

3:25 PM
Effect of High Magnetic Field on Microstructural Evolution in Fe-1%Si: *T. A. Bennett*[1]; R. A. Jaramillo[2]; J. B. Wilgren[2]; R. Kisner[2]; G. Mackiewicz-Ludtka[2]; G. M. Ludtka[2]; P. N. Kalu[3]; A. D. Rollett[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 3325 Wean Hall, Pittsburgh, PA 15217 USA; [2]Oak Ridge National Laboratory, Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6065 USA; [3]FAMU-FSU College of Engineering, Mechl. Engrg. Dept., 2525 Pottsdamer St., Tallahassee, FL 32310-6046 USA

Microstructural and texture evolution and the change in grain boundary character distribution have been studied in Fe-1%Si as a function of field strength. Magnetic fields varying from 1.5T to 30T have been applied parallel to the sample rolling direction at 600°C and 787°C, i.e. below and above the Curie temperature. When annealed above the Curie temperature for 1 hour at 1.5T, samples experienced a drastic increase in average grain size from 50 microns to 220 microns. No significant texturing occurred but there was an increase in the cube texture component. The cube component persisted up to 30T but its volume fraction decreased relative to that observed for lower fields. The grain boundary character distribution also exhibited a higher percentage of low angle boundaries with increasing field strength; hence, a correlation was observed between the magnetic field strength and the microstructural changes in Fe-1%Si.

3:40 PM Break

4:05 PM Invited
Applications of Numerical Simulations of Displacive and Diffusional Transformations: *Greg Olson*[1]; [1]Northwestern University/ QuesTek, Matls. Sci. & Engrg., 2225 N. Campus Dr., Evanston, IL 60015 USA

Application of a numerical "phase-field" approach based on rigorous Landau-Ginzburg models of martensitic transformations has quantified the boundary between classical and Cahn-Hilliard nonclassical behavior in both heterogeneous and homogeneous nucleation. This has allowed confident predictive application of classical approximations in computational materials design of steels and shape-memory alloys. Application of precipitation simulation to the accelerated development and qualification of new alloys requires higher computational efficiency provided by sharp-interface models. The new PrecipiCalc code grounded in multicomponent thermodynamics and diffusion has demonstrated an optimal combination of fidelity and efficiency for both process optimization at the component level and prediction of part-to-part variation in manufacturing.

4:30 PM Invited
Quantitative Phase Field Modeling: *Alphonse Finel*[1]; Yann Le Bouar[1]; Quentin Bronchart[1]; Guillaume Boussinot[1]; [1]ONERA-CNRS, LEM, BP 72, 29 Ave. Div. Leclerc, Châtilllon 92322 France

Phase Field methods are intensively used to predict the mesoscopic behavior of complex materials and its range of applicability has been extended to a wide range of physical phenomena. One of the directions which are today the subject of an intensive activity is its ability to be quantitative for a specific system. We will address this last aspect, and discuss different points of view for devising a quantitative Phase Field modeling.

4:55 PM Invited
A Comparison of the Phase Field and Monte Carlo Simulations of the Interaction Between a Grain Boundary and a Coarse Particle: *Bala Radhakrishnan*[1]; Gorti Sarma[1]; Yunzhi Wang[2]; [1]Oak Ridge National Laboratory, Computer Sci. & Math., Bldg. 5600, MS 6008, Oak Ridge, TN 37831-6008 USA; [2]Ohio State University, Matls. Sci. & Engrg., 2041 College Rd., Columbus, OH 43210 USA

The paper presents large scale, three-dimensional, Potts model simulations of the interaction between an initially straight grain boundary driven by a bulk stored energy difference across the boundary and a coarse grain boundary particle. The Potts model simulations show that the interaction energy is significantly affected by the choice of the lattice temperature and the driving force. The boundary velocity responds appropriately to changes in the magnitude and direction of the driving force only at high lattice temperatures or high driving forces where the boundary is flexible enough to bend locally near the particle. At lower lattice temperatures or low driving forces, the boundary response is "stiff" and this leads to an increase in the initial energy before it starts to decrease as the boundary approaches the particle. The Potts model simulations are compared with the predictions of the phase field model under identical initial conditions.

5:20 PM
Study on the Abilities of the Phase Field Method in Simulating Grain Growth in Materials Containing Second-Phase Particles: *Nele Moelans*[1]; Bart Blanpain[1]; Patrick Wollants[1]; [1]K. U. Leuven, Dept. of Metall. & Matls. Engrg., Kasteelpark Arenberg 44, B-3001 Heverlee Belgium

A Phase Field model, together with a convenient implementation technique, has been worked out for grain growth in materials containing inert, immobile particles. Simulations were performed for several sizes and volume fractions of the second-phase particles. Properties frequently measured in grain growth experiments were calculated for the simulated systems and compared with experimental values. Two main difficulties arose. (1) The essential microstructural features, viz. grains, second-phase particles and grain boundaries, range in size over very different scales. This leads to long computational times and huge computer memory requirements. (2) It is difficult to reproduce grain boundary energy and thickness, which are important parameters determining the interaction between grain boundary and particle. Both difficulties are general drawbacks of the Phase Field method in its current state of development. This work can bring new insights in grain growth in multi-phase materials and contributes to a further development of the Phase Field simulation technique.

The Langdon Symposium: Flow and Forming of Crystalline Materials: Equal Channel Angular Pressing

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; P. B. Berbon, Rockwell Scientific Company, Thousand Oaks, CA 91360 USA; Atul H. Chokshi, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India; Z. Horita, Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-8581 Japan; Sai V. Raj, NASA Glenn Research Center, Materials Division, Cleveland, OH 44135 USA; K. Xia, University of Melbourne, Department of Mechanical and Manufacturing Engineering, Victoria 3010 Australia

Tuesday PM Room: 3024
February 15, 2005 Location: Moscone West Convention Center

Session Chairs: Sai V. Raj, NASA Glenn Research Center, Matls., Cleveland, OH 44135 USA; Marco J. Starink, University of Southampton, Matls. Rsch. Grp. Sch. of Engrg. Sci., Southampton SO17 1BJ UK; Patrick B. Berbon, Rockwell Scientific, Thousand Oaks, CA 91360 USA; Kenji Higashi, Osaka Prefecture University, Dept. of Metall. & Matls. Sci., Sakai, Osaka 599-8531 Japan

2:00 PM
Interactions Between Microstructure and Texture Evolution in the Early Stages of ECAE: *Irene J. Beyerlein*[1]; David J. Alexander[1]; Saiyi Li[1]; Carl T. Necker[1]; Qing Xue[1]; [1]Los Alamos National Laboratory, Theoretical Div., MS B216, Los ALamos, NM 87545 USA

Through severe plastic deformation and strain path changes, ECAE imparts substantial microstructural changes in the material at multiple length scales. Our pursuit involves developing a multi-scale model that couples the mechanics starting at the subgrain level, increasing to the grain and aggregate levels and eventually ending at the macroscopic length scale of the sample. Understanding the impact of the strain path changes associated with various ECAE routes requires appreciation of the evolution of microstructure at these length scales in each pass. Combining multi-scale modeling (substructure evolution, polycrystal models, and finite element simulation) and experimental techniques (TEM, OIM, neutron diffraction, and mechanical testing), we investigate the relationship between substructure and texture evolution in the early stages of the ECAE process for routes Bc and C. We present our findings on how the grain morphology, substructure, and texture developed in the new strain path (either a mechanical test or subsequent ECAE pass) interact with those generated in the previous pass.

2:15 PM
Anisotropy in Mechanical Properties of High-Purity Copper Processed by Equal Channel Angular Extrusion: *David J. Alexander*[1]; Irene J. Beyerlein[2]; [1]Los Alamos National Laboratory, MST-6, G770, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, T-3, B-216, Los Alamos, NM 87545 USA

High-purity oxygen-free electronic copper has been processed by equal channel angular extrusion for up to 4 passes at room temperature, using 90° tooling, by either route Bc or C. Small tensile specimens, as well as compression cubes, were sectioned along the length of the billet by electrodischarge machining, and tested in the as-processed condition. The ECAE-processed material was anisotropic, with the strength in the direction perpendicular to the plane formed by the inlet and outlet channels being greater than that in the other directions. Also, the yield strength in compression was greater than the yield strength in tension. The results for routes Bc and C will be compared, and viscoplastic self-consistent modeling will be used to help interpret these results.

2:30 PM
Texture Development During the Equal Channel Angular Pressing of Magnesium Alloys AZ31, AZ80, Mg-4wt%Li, WE43, and ZK60: *Sean R. Agnew*[1]; P. Merhotra[1]; T. M. Lillo[2]; G. M. Stoica[3]; P. K. Liaw[3]; [1]University of Virginia, Matls. Sci. & Engrg., Charlottesville, VA 22904 USA; [2]Idaho National Engineering and Environmental Laboratory, Idaho Falls, ID USA; [3]University of Tennessee, Matls. Sci. & Engrg., Knoxsville, TN USA

In recent years, Terry Langdon has contributed to the literature of severe plastic deformation more than anyone. One notable contribution has been his group's (and collaborators') exploration of the effect of various processing routes possible via equal channel angular pressing (ECAP), i.e. A, Ba, Bc, C, etc., on the microstructure and properties of many metals and alloys. Defining and describing these various strain paths has helped to clarify many findings in recent years. The effect of ECAP processing on the properties of magnesium alloys originally focused on the potential for superplastic behavior. However, a report of tremendously improved room temperature ductility stressed the possible importance of the texture developed through ECAP. As a result, the texture development of 5 magnesium alloys representing 4 major classes of alloys (Mg-Al, Mg-Li, Mg-Rare Earth, and Mg-Zn based) has been explored. Each class exhibits a distinct texture evolution, which may be linked to distinctions in the activities of the various underlying deformation mechanisms. These connections may be made more quantitatively through the use of computer simulation of the texture using polycrystal plasticity models. The implications for properties will be discussed and demonstrated, in select cases.

2:45 PM
Creep Processes in Pure Aluminium Processed by ECAP Technique: *Vaclav Sklenicka*[1]; Jiri Dvorak[1]; Petr Kral[1]; Milan Svoboda[1]; [1]Academy of Sciences of the Czech Republic, Inst. of Physics of Matls., Zizkova 22, Brno CZ-616 62 Czech Republic

Creep tests were conducted on pure aluminium processed by ECAP at temperatures 423-523K and a 10-25MPa. Specimens were examined by transmission and scanning electron microscopy equiped with an EBSD facility. The amount of grain boundary sliding was experimentally determined. Based on the results, it is suggested that creep of the ECAPed aluminium occurs by diffusion-controlled movement of dislocations and by grain boundary sliding. The coexistence of a dislocation climbing process and grain boundary sliding may explain the observed decrease of the creep resistance with increasing number of ECAP passes. Since high-angle grain boundaries are necessary in order to achieve grain boundary sliding, an increase in the fraction of high-angle boundaries with increasing number of ECAP passes will essentially lead to an increasing contribution of sliding to the total creep strain as it was observed experimentally.

3:00 PM
Ultrafine Grained Steels Processed by ECAP: *Kyung-Tae Park*[1]; Young-Kook Lee[2]; Dong Hyuk Shin[3]; [1]Hanbat National University, Div. of Advd. Matls. Sci. & Engrg., Taejon 305-719 S. Korea; [2]Yonsei University, Dept. of Metall. Engrg., Seoul 120-749 S. Korea; [3]Hanyang University, Dept. of Metall. & Matls. Sci., Ansan, Kyunggi-Do 425-791 S. Korea

ECAP is very useful to modify the microstructure of ferrite-pearlite steels, resulting in considerable enhancement of their mechanical properties. The microstucture and mechanical properties of ECAPed ferrite-pearlite steels are reviewed, specifically focusing on fabrication of ultrafine grained (UFG) dual phase steels and their mechanical properties. In addition to ultrahigh strength, the UFG dual phase steels fabricated by ECAP exhibited extensive strain hardening unlike other UFG metallic materials.

3:15 PM
A Quantitative Study of Cavity Development in the Tensile Testing of an Aluminum Metal Matrix Composite Processed by ECAP: *Megumi Kawasaki*[1]; Yi Huang[1]; Cheng Xu[1]; Minoru Furukawa[2]; Zenji Horita[3]; Terence G. Langdon[1]; [1]University of Southern California, Dept. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA; [2]Fukuoka University of Education, Dept. of Tech., Munakata, Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan

An Al-6061 metal matrix composite, reinforced with 10 vol. % of fine Al_2O_3 particulates, was subjected to equal-channel angular processing (ECAP) through one pass at 298 K and an additional 11 passes at a temperature of 473 K. The mechanical properties were investigated through tensile testing of both as-received and as-pressed materials over a range of strain rates at 873 K. The results show that higher elongations are achieved in both materials at the faster strain rates. A detailed examination was conducted to analyze the extent of the internal cavitation in these materials after tensile testing to failure. This paper compares the development of cavities in the composite material in both the as-received and as-pressed conditions.

3:30 PM Break

3:45 PM
An Investigation of the Deformation Process During Equal-Channel Angular Pressing of an Aluminum Single Crystal:

Minoru Furukawa[1]; Yoshiyasu Kawasaki[2]; Yuichi Miyahara[2]; Zenji Horita[2]; Terence G. Langdon[3]; [1]Fukuoka University of Education, Dept. of Tech., 1-1 Akama-Bunkyomachi, Munakata, Fukuoka 811-4192 Japan; [2]Kyushu University, Dept. of Matls. Sci. & Engrg., Fukuoka 812-8581 Japan; [3]University of Southern California, Depts. of Aeros. & Mechl. Engrg., Los Angeles, CA 90089-1453 USA

This investigation examines the deformation process when an aluminum single crystal is subjected to equal-channel angular pressing (ECAP) using a die having a channel angle of 90°. The crystal within the entrance channel was oriented so that the (111) slip plane and the [110] slip direction were inclined by 20?a in a counter-clockwise direction from the theoretical shear plane and direction, respectively. The crystal was pressed through one pass at room temperature and examined by orientation imaging microscopy. After passing through the shear plane, the initial orientation remained in the upper portion within the exit channel but there was a rotation by ~60?a in a counter-clockwise direction near the lower die wall. These observations show pure shear occurs in the upper portion of the specimen but there is a rotation in the lower portion due to the curvature at the point of intersection of the two channels.

4:00 PM

Effect of Acute Tool-Angles on Equal Channel Angular Extrusion/Pressing: *Anumalasetty Venkata Nagasekhar*[1]; Yip Tick-Hon[1]; Li Suixiang Sean[1]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Singapore 639 798 Singapore

Materials processed by equal channel angular extrusion/pressing (ECAE/ECAP) are influenced by factors like amount of strain induced, and uniformity of strain distribution. These factors in turn depend on parameters like tool angles (channel angle and outer corner angle), material properties, and friction between the die and the sample. Acute tool-angles can increase the strain induced in the material for minimum number of passes. Such increased strains can yield ultrafine grains and high fraction of high angle grain boundaries, which enhance mechanical and superplastic properties. However, with acute tool angles the deformation and punch pressure requirements will be more stringent. Hence, to study the effect of the parameters, finite element simulations of ECAE were carried for channel angles of 60°, 75° and 90°, and with various outer corner angles by using Abaqus/Explicit. Factual phenomena like strain hardening of material and friction were considered to obtain realistic deformation behavior.

4:15 PM

Influence of Severe Plastic Deformation on Aging of Al-Mg-Si Alloys: *Emanuela Cerri*[1]; Leo Paola[1]; [1]University of Lecce, Dept. Ingegneria dell'Innovazione, via Arnesano, Lecce 73100 Italy

The influence of SPD induced by ECAP on microstructural modifications and aging effect was studied in two different Al-Mg-Si alloys. The microstructure of both alloys in different heat treated and deformed state was characterized by X-Ray diffraction, SEM and optical microscopy. The effect of artificial aging was studied after SPD in the as received state and after SPD performed in the solution condition. The aging effect was followed by microhardness and electrical conductivity measurements. At higher aging temperature (i.e 170°C and 190°C) the alloys showed an increasing softening with time and number of pressing due to recovery or/and grain coarsening effect. At low temperature of aging (110°C) only the solutioned alloy exhibited an hardness increasing with time due to an enhanced precipitation, induced by an higher dislocation density present at low temperature, and a reduced recovery phenomena.

4:30 PM

Creep and Superplasticity in a Spray-Cast Aluminium Alloy Processed by Equal-Channel Angular Pressing: *Cheng Xu*[1]; Terence G. Langdon[1]; [1]University of Southern California, Dept. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

A commercial spray-cast aluminium alloy having a composition of Al-11.5% Zn-2.5% Mg-0.9% Cu-0.2% Zr was processed by equal channel angular pressing (ECAP) at a temperature of 473 K. The alloy achieved a grain size reduction from ~2.1 μm to ~0.3 μm and it was observed that there is a breaking of precipitates during ECAP. The ultrafine-grained structure of the as-pressed alloy was reasonably stable up to temperatures of the order of ~670 K. Tensile and creep tests were conducted using the alloy in both the unpressed and the as-pressed conditions. The results show that the alloy subjected to ECAP achieved superplastic elongation to failure of >1000% at high stain rates of $\geq 10^{-2}$ s^{-1} when testing at 673 K. Also, faster creep rates were observed in the as-pressed material when testing at the same applied stress and testing temperature.

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Microstructural Evolution in a Spray-Cast Aluminium Alloy During ECA Pressing and in Subsequent Heat Treatment: Nong Gao[1]; *Marco Jan Starink*[1]; Minoru Furukawa[2]; Zenji Horita[3]; Cheng Xu[4]; Terence G. Langdon[4]; [1]University of Southampton, Matls. Rsch. Grp., Sch. of Engrg. Scis., Highfield, Southampton SO17 1BJ UK; [2]Fukuoka University of Education, Dept. of Tech., Munakata, Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-4192 Japan; [4]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

The microstructures of a spray-cast Al-7034 (Al-Zn-Mg-Cu) alloy after processing through equal-channel angular pressing (ECAP) were studied using electron back-scatter diffraction (EBSD) and differential scanning calorimetry (DSC). The ageing response and recrystallisation softening post ECAP were studied by hardness testing and DSC. The results demonstrate there is a relatively rapid increase in the fraction of high-angle boundaries during the initial ECAP passes and a subsequent more gradual increase in further passes. The crystallographic textures introduced by ECAP are analysed. For the Al-7034 alloy, the DSC analysis identifies the occurrence of several thermal effects during heating involving the formation, coarsening, dissolution and melting of the eta phase.

5:00 PM

Scaling Up of Equal Channel Angular Pressing (ECAP) and its Effect on Mechanical Properties, Microstructure, and Hot Workability of AA 6061: *Prabir K. Chuadhury*[1]; Raghavan Srinivasan[2]; [1]Materials and Processing Resources, Inc., 4120 Foxtail Ln., Plano, TX 75024 USA; [2]Wright State University, Mechl. & Matls. Engrg. Dept., Dayton, OH 45435 USA

Equal Channel Angular Pressing (ECAP) at various cross sectional areas were performed on aluminum alloy 6061 to study the effect of scaling up on the mechanical properties, microstructure, and the hot workability of the alloy. In this study, annealed AA 6061 was subjected to severe plastic deformation at room temperature by ECAP (Route BC), producing 12.5 mm (0.5 inch), 50 mm (2.0 inch), and 100 mm (4.0 inch) square billets. The mechanical properties and microstructure of as pressed alloy were examined as a function of the extent of deformation and the cross sectional area of the billets. Also hot workability was determined by forging industrial parts at various temperatures. Results indicate that the average grain size after ECAP is of the order of 0.5 mm. This grain refinement resulted in enhanced hot workability at 315 C (600 F), indicating applicability of ECAP processed material in Forging Industry.

The Langdon Symposium: Flow and Forming of Crystalline Materials: Poster Session

Sponsored by: Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

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Tuesday, 5:15-7:00pm Room: 3024
February 15, 2005 Location: Moscone West Convention Center

Intermediate-Temperature Equal Channel Angular Extrusion of Electron-Beam Melted Crystal Bar Zirconium: *David J. Alexander*[1]; [1]Los Alamos National Laboratory, MST-6, G770, Los Alamos, NM 87545 USA

Electron-bean melted crystal bar zirconium was processed by equal channel angular extrusion at temperatures from 200 to 450°C, for 4 passes with tooling with a 105° angle. The deformed material showed evidence of deformation by twinning, and was finely subdivided. Annealing was performed at temperatures from 400 to 550°C. Full recrystallization occurred at annealing temperatures of 500°C or higher; only partial recrystallization occurred below this temperature. The recrystallized grain size increased as the annealing temperature in-

creased. The effect of the deformation temperature on the resultant microstructure and texture will be discussed.

Processing of Tantalum by Equal Channel Angular Extrusion with the Aid of Electroplated Surface Layers: *David J. Alexander*[1]; [1]Los Alamos National Laboratory, MST-6, G770, Los Alamos, NM 87545 USA

Tantalum is difficult to process by conventional extrusion as it has a marked tendency to gall on the tooling. Equal channel angular extrusion of Ta was successfully performed at room temperature by using thick electroplated layers of Cu, Ni, or Ni + Cu to prevent contact between the Ta billet and the tooling. The electroplated layers successfully withstood 4 passes for tooling with a 105° angle between the channels. The processed material was annealed in vacuum at temperatures ranging from 800 to 1200°C. The recrystallized grain size increased as the annealing temperature increased.

An Upper Bound Analysis in ECAE: *Burhanettin Semsi Altan*[1]; [1]Michigan Technological University, Mechl. Engrg. & Engrg. Mech., 1400 Townsend Dr., Houghton, MI 49931-1299 USA

An upper-bound analysis has been carried out to investigate the plastic deformation behavior of the material during the 90-ECAE process. Material is assumed to be rigid-plastic Von Mises material. The deformation model proposed in this study allows plastic deformation to occur in a region symmetric with respect to plane formed by the intersection of the two extrusion channels. Formation of the plastic deformation region is investigated as a function of three parameters: the radius of the outer corner of the die (actually the interface between the dead metal zone and the deformation zone), the radius of the inner corner of the die, and the friction on the die walls. For simplicity, the inner and outer corners of the die are taken to be concentric circles. The relationship between the radius of the inner corner and the friction coefficient has been developed which governs the size of the deformation region.

Analytical Electron Microscopy of Reaction Ball-Milled Y-Ni-O Nanostructures: *James Bentley*[1]; David T. Hoelzer[1]; Laurent Chaffron[2]; Dorothy W. Coffey[1]; Kathy A. Yarborough[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA; [2]CEA Saclay, Service de Reserches de Métallurgie Physique, Gif sur Yvette 91191 France

Powders of NiO and Ni2Y were ball-milled under vacuum at 30 and 100°C for 144h in an instrumented mill at CEA, Saclay to induce the reaction 3 NiO + 2 Ni_2Y —> 7 Ni + Y_2O_3. Quantitative compositional mapping at ~1 nm resolution has been performed at ORNL on the ~10-nm-scale microstructures by energy-filtered transmission electron microscopy (EFTEM) and spectrum imaging techniques. Electron microscopy specimens were made from as-milled powders by focused ion beam (FIB) milling. The atomic diffusion necessary to accomplish the almost complete reduction of NiO and form the observed nanostructures cannot occur at the 30 or 100°C. Temperature spikes, from either localized exothermic chemical reactions or the conversion of ball kinetic energy upon impact, and the role of vacancies injected during the severe plastic deformation (the required activation energy is then just the vacancy migration energy, not migration plus formation energies) are considered as enabling mechanisms.

Applicability of the Groove Pressing Technique for Grain Refinement in Commercial Purity Copper: *Krishnaiah Arkanti*[1]; *Uday Chakkingal*[1]; P. Venugopal[1]; [1]Indian Institute of Technology, Metal Forming Lab., Metallurgl. & Matls. Engrg., Madras, Chennai, Tamil Ladu 600 036 India

There is currently a lot of interest in producing bulk ultra-fine grained materials by imposing severe plastic deformation. This interest arises because a reduction in grain size leads to increases in the strength and toughness of the material at ambient temperatures. In the current study, specimens of commercial purity copper were subjected to large strains using the groove pressing technique. Dies were designed and fabricated with the groove angle (q) of 45° so that a single pressing yields a shear strain of 1, at deformed region. This is equivalent to an effective true strain of 0.58 and each series of four pressings yields a homogeneous effective strain of 1.16 throughout the sample. Copper specimens were subjected to several passes using these techniques at room temperature and at cryogenic temperature. The mechanical properties and microstructure were studied as a function of the number of passes. The Vickers hardness values increased from 44 VHN to 98 VHN. The microstructure of the copper samples reduced to 32 mm from 87 mm as the starting grain size after 3 passes. Cell sizes approximately 500 nm in size were obtained.

Creep Behavior in a Cryomilled Ultrafine-Grained Al 5083 Alloy: *Manish Chauhan*[1]; Indranil Roy[1]; Farghalli A. Mohamed[1]; [1]University of California, Chem. Engrg. & Matls. Sci., 916 Engrg. Tower, Irvine, CA 92696-2575 USA

The creep behavior of an ultrafine-grained (UFG) Al 5083 alloy, prepared by a cryomilling with an average grain size of 300 nm, was investigated at three temperatures: 573, 623 and 673 K. The results show the presence of three creep regions: Low-stress region that exhibit a low stress exponent of less than 10, intermediate-stress region that is characterized by a high stress exponent of approximately 47, and a high-stress region that is characterized by an intermediate stress exponent of approximately 17. An analysis of the data of the low-stress region suggests the presence of a threshold stress, which strongly depends on the temperature. Possible creep mechanisms are discussed in cryomilled Al 5083 alloy and compared with those proposed for dispersion strengthened alloys.

Mechanical Behavior of a 6061 Al Alloy and Al2O3/6061 Al Composite Processed by ECAP: *Lijia Chen*[1]; Chunyan Ma[2]; *G. M. Stoica*[1]; *Peter K. Liaw*[1]; *Cheng Xu*[3]; *Terence G. Langdon*[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., Rm. 427-B, Dougherty Engrg. Bldg., Knoxville, TN 37996-2200 USA; [2]Shenyang University of Technology, Sch. of Matls. Sci. & Engrg., 58 Xinghua S. St., Tiexi Dist. Shenyang 110023 China; [3]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Olin Hall of Engrg. 430, Los Angeles, CA 90089-1453 USA

A commercial 6061 Al alloy and an Al2O3/6061 Al composite were subjected to 8 passes of equal-channel angular pressing (ECAP) using the strain path of route BC. The tension and stress-controlled fatigue behavior of the alloy and composite at room temperature were investigated and compared. For the commercial 6061 Al alloy, it was found that after ECAP both the tensile and fatigue strengths of the alloy decreased while the elongation to failure showed a minor reduction. However, the tensile and fatigue strengths of the Al2O3/6061 Al composite subjected to ECAP were significantly although the elongation to failure of the composite was lower than before ECAP. In addition, the fracture surfaces were observed using scanning electron microscopy to determine the fracture mode.

A Comparison of the Properties of SPD Processed AA-6061 by ECAP, Multi-Axial Compressions/Forgings (MAC/F) and Accumulative Roll Bonding (ARB): *Balakrishna Cherukuri*[1]; Teodora S. Nedkova[1]; Raghavan Srinivasan[1]; [1]Wright State University, Mechl. & Matls. Engrg. Dept., 3640 Col. Glenn Hwy., Dayton, OH 45435 USA

Commercially available AA-6061 in the annealed condition was subject to Severe Plastic Deformation (SPD) processing by Equal Channel Angular Pressing (ECAP), Multi Axial Compression/Forgings (MAC/F) and Accumulative Roll Bonding (ARB) at room temperature to approximately the same accumulated strain (~4). Room temperature and elevated temperature tensile and compression tests were carried out to evaluate the flow behavior of the as-SPD materials. In comparison to the as received material, the SPD processed materials show high strain rate sensitivity, and hence potentially enhanced formability in the temperature range of 300C-350C. Results presented will include the as-processed microstructure, the stability of the microstructure at elevated temperatures, microhardness and the flow behavior of the various materials at different strain rates and temperatures. This work was supported by the US Department of Energy through Grant No. DE-FC36-01ID14022.

Superplastic Behavior of Coarse-Grained Aluminum Alloys: *A. R. Chezan*[1]; J. Th.M. De Hosson[1]; [1]University of Groningen and Netherlands Institute for Metals Research, Dept. of Applied Physics, Nijenborgh 4, Groningen 9747AG The Netherlands

We report on the superplastic behavior and the microstructural evolution of two coarse-grained Al alloys: Al-4.4w/oMg and Al-4.4w/oMg-0.4w/oCu. The maximum tensile elongations obtained by deformation at 450°C and at a strain rate of 10^{-2}/s were 260 and 315%, respectively. The value for the strain rate sensitivity index of 0.3 and a sharp peak stress at small plastic strain suggest that solute drag on dislocation motion is an important phenomenon in these materials. Orientation imaging microscopy (EBSP) observations show a decrease of the average grain size with increasing plastic strain. The deformed microstructure is characterized by an increased density of low angle grain boundaries and by a pronounced texture as compared to the initial grain structure. These microstructural changes trigger instabilities in the plastic flow resulting in necking followed by fracture and act as limiting factors of the superplastic performance in coarse-grained aluminum alloys.

Creep Behavior of Extruded Al-6Mg-1Sc-1Zr-10SiC Alloy: *S. P. Deshmukh*[1]; R. S. Mishra[1]; K. L. Kendig[2]; [1]University of Missouri, Dept. of Metallurgl. Engrg., 1870 Miner Cir., Rolla, MO 65409 USA;

[2]Air Force Research Laboratory, Matls. & Mfg. Direct., Wright Patterson AFB, OH 45433 USA

Creep tests were performed on fine grained Al-6Mg-1Sc-1Zr-matrix alloy with and without SiC particulates in the temperature range of 150 to 260°C. The composite was fabricated by direct extrusion of billets made from helium atomized alloy powder and SiC particulate. The composite exhibits high value of apparent stress exponent and apparent activation energy for creep at low temperatures. The flow behavior of these composite is characterized by the presence of a threshold stress. Incorporation of threshold stress into analysis reduces the high values of apparent stress exponent and activation energy to those anticipated form the creep of solid solution alloys. Results shows increased creep resistance in composite compared to unreinforced alloy at higher temperatures.

Tensile and Compressive Creep Behaviour of Al2O3 (Saffil)-Short Fiber Reinforced Magnesium Alloy AE42: *Hajo Dieringa*[1]; Yuanding Huang[1]; Petra Maier[1]; Norbert Hort[1]; *Karl Ulrich Kainer*[1]; [1]GKSS Research Center, Ctr. for Mg. Tech., Max-Planck-Str. 1, Geesthacht 21502 Germany

Development of metal matrix composites (MMCs) is a possibility to overcome the disadvantages of poor high temperature creep proiperties of common magnesium alloys. It is well known that short fiber reinforcement improves the high temperature creep resistance of magnesium alloys. Tensile and compressive creep properties of magnesium alloy AE42 (4 wt.-% aluminum and 2 wt.-% rare earths) reinforced with 20 vol.-% alumina (Saffil)-fibres were investigated in the temperature range of 175-300°C and stress range of 40-140 MPa. The MMC was manufactured by direct squeeze casting. The materials investigated show different behaviour depending on type of loading. From the stress dependence of the minimum creep rate stress exponents were calculated in order to find indications for the possible mechanisms of deformation. The need of correcting the stress dependence of minimum creep rate by a threshold stress was found. Electron microscopy was performed to investigate the surface of fracture of a tensile creep specimen.

The Effect of Testing Temperature on Stress-Strain Relationships in Pure Aluminum Over a Wide Range of Strain: *Nguyen Quang Chinh*[1]; Zenji Horita[2]; Terence G. Langdon[3]; [1]Eötvös University, Dept. of Gen. Physics, 1117 Budapest, Pázmány P. sétány 1/A., Budapest Hungary; [2]Kyushu University, Dept. of Matls. Sci. & Engrg., Faculty of Engrg., Fukuoka 812-8581 Japan; [3]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

The stress-strain relationships of high purity aluminum were investigated over a wide range of strain by combining data obtained in conventional testing of annealed samples with data obtained after processing by equal-channel angular pressing (ECAP) to high imposed strains. The results show the nature of the macroscopic stress-strain relationship changes characteristically in different temperature regions. In the low temperature region the macroscopic stress-strain behavior exhibits a monotonously increasing tendency over the entire range of strain whereas in high temperature testing the flow stress increases only to a critical strain. It is demonstrated that the stress-strain relationship in the positive strain-hardening region may be described by an exponential-power law constitutive equation which reduces to the conventional Hollomon power-law relationship at low strains and to the Voce exponential relationship at high strains. On the basis of this new equation, it is possible to define low and high temperature deformation regions.

Synthesis of Bulk Nanostructured Materials by Repeated Cold-Rolling: *Guru Prasad Dinda*[1]; Harald Roesner[1]; Gerhard Wilde[1]; [1]Forschungszentrum Karlsruhe, Inst. of Nanotech., Eggenstein-Leopoldshafen, Karlsruhe 76021 Germany

Recently, bulk nanostructured materials synthesized by severe plastic deformation (SPD) have attracted increased attention because of their improved and rather unusual mechanical properties. Repeated cold-rolling with intermediate folding represents an alternative SPD-technique to produce extremely fine nanocrystalline materials at ambient temperature. In the present work, massive nanocrystalline samples of Ti and Zr with average grain sizes below 100 nm and Ni with grain size less than 10 nm have been synthesized at ambient temperature by repeated cold-rolling. The evolution of the microstructure and texture at different stages of plastic deformation was investigated by X-ray diffraction, scanning and transmission electron microscopy. Mean grain sizes less than 10 nm in pure Ni have not been obtained by any SPD techniques. The main key to produce such fine nanocrystalline structure seems to be the requirement of very large plastic strain but not necessarily a high imposed pressure in the range of several GPa.

Finite Element Modeling of Equal Channel Angular Pressing: Effect of Material Properties, Friction and Die Geometry: Stephane Dumoulin[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., Trondheim 7491 Norway

Equal channel angular pressing (ECAP) is an efficient process for obtaining ultra-fine grained materials. To be industrially applicable, it is of great importance to create a homogeneous microstructure in order to have constant material properties in the whole work-piece i.e. to have homogeneous strains during the process. However, the strain distribution developed during pressing strongly depends on material properties (yield strength, strain hardening, strain rate sensitivity), die geometry (channel angle, die corner angle) and friction. Therefore, finite element modeling of one pass ECAP at room temperature of an aluminum alloy is performed in order to investigate and characterize the effects of these parameters on strain distribution and some selected parameters of the process such as the work-piece shape in the die angle region. Simulations are performed using the commercial finite element code DEFORM. The results are validated by comparison with experimental results.

Improving the Superplastic Properties of a Two-Phase Mg-8% Li Alloy Through Processing by ECAP: *Mitsuaki Furui*[1]; Cheng Xu[2]; Tetsuo Aida[3]; Makoto Inoue[4]; Hiroshi Anada[1]; Terence G. Langdon[2]; [1]Toyama University, Dept. of Sys. Engrg. of Matls. & Life Sci., Faculty of Engrg., 3190 Gofuku, Toyama 930-8555 Japan; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA; [3]Toyama University, Dept. of Mechl. & Intelligent Sys. Engrg., Faculty of Engrg., Toyama 930-8555 Japan; [4]Toyama National College of Technology, Dept. of Ecomatls. Engrg., Toyama 939-8630 Japan

Grain refinement was achieved in a cast Mg-8mass% Li alloy through processing by equal-channel angular pressing (ECAP) using a die having an internal channel angle of 110° and a pressing temperature of 473 K. Following extrusion and subsequent ECAP through 2 passes, the alloy exhibited superplasticity over the temperature range from 423 to 473 K. A maximum fracture elongation of >950% was attained at 473 K with an initial strain rate of 1×10^{-4} s^{-1} and the strain rate sensitivity was of the order of 0.6. This elongation is high by comparison with other Mg alloys tested in tension at the same temperature and strain rate. The activation energy for superplastic flow in this alloy was essentially equal to the value for grain boundary diffusion of Mg. The use of ECAP gave a significant improvement in strength and ductility at room temperature.

An Investigation of Crack Growth Behaviour by Reverse Bending Cycling Under Creep-Fatigue Condition: *Nong Gao*[1]; [1]University of Southampton, Matls. Rsch. Grp., Southampton SO17 1BJ UK

An investigation of short crack growth behaviour of AISI type 316 stainless steel under creep-fatigue conditions at 550 within high strain ranges of 0.9-2.5% and 60 minutes hold time was undertaken on a high temperature reverse bending rig. Throughout the tests, surface crack initiation and growth on both tensile and compressive sides were monitored by means of a plastic replication technique. These analyses revealed that the behaviour of individual initiation and growth of many minor cracks in Stage I, and their subsequent coalescence in Stage II are the dominant characteristics for the failure of the specimens. Increasing the strain range causes an increasing number of minor cracks and a promotion of the process of minor crack coalescence. Predominantly intergranular long cracks on the tensile side and transgranular short cracks on the compressive side are the prominent feature. The different crack morphology and crack length on the two sides of a specimen indicates that a compression-only dwell is much less dangerous than a tension-only dwell.

Microstructure and Mechanical Properties of Highly Deformed Ti-6Al-4V: *Mehmet N. Gungor*[1]; Lawrence S. Kramer[1]; Ibrahim Ucok[1]; Hao Dong[1]; Nicholas R. Martin[1]; Wm. Troy Tack[1]; [1]Concurrent Technologies Corporation, 100 CTC Dr., Johnstown, PA 15904 USA

Various deformation processes, including beta extrusion, alpha + beta extrusion, rotary piercing and flowforming, were utilized to produce seamless Ti-6Al-4V tubular structures. Microstructures and mechanical properties of deformed Ti-6Al-4V tubes were studied. Microstructure development in tubes was investigated utilizing metallography, texture analysis, and scanning electron microscopy techniques. While extruded and rotary pierced tubes were annealed, flow formed tubes were stress relieved prior to mechanical testing. The mechanical testing matrix included tensile and fatigue tests. In this paper, the study results for different tube manufacturing methods are presented, compared and discussed. This work was prepared by the National Center for Excellence in Metalworking Technology, operated by Concur-

rent Technologies Corporation (CTC), under Contract No. N00014-00-C-0544 to the Office of Naval Research as part of the Navy Manufacturing Technology Program.

Improvement of High-Temperature Behavior in a Mg-0.55% Zr Alloy Through Equal-Channel Angular Pressing: *Bing Q. Han*[1]; Terence G. Langdon[2]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Equal-channel angular pressing was performed on a Mg-0.55% Zr alloy at a temperature of 513 K. The grain size was refined from 75 mm to 8.6 mm by ECAP. Tensile tests were performed at temperatures of 473 to 773 K and at strain rates from 10-5 to 10-1 s-1. It was found that the tensile elongation in the as-pressed state was higher than in the as-received state. A largest elongation of 384 % was observed at a temperature of 773 K using a strain rate of 4 x 10-5 s-1. The stress exponent at all temperatures was estimated to be about 0.17. The activation energy of deformation of the pressed Mg alloy was estimated to be close to that of grain boundary diffusion while the activation energy of the as-received Mg alloy was close to that of lattice self-diffusion. The possible deformation mechanisms at elevated temperatures were discussed.

Superplasticity of a Commercial 6061 Al Composite Processed by Severe Plastic Deformation: *Bing Q. Han*[1]; Terence G. Langdon[2]; [1]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Davis, CA 95616 USA; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Severe plastic deformation of a commercial 6061 Al composite was performed through equal-channel angular pressing at a temperature of 533 K. A microstructure with ultrafine grains was present after 8 passes. Superplasticity was observed in the ultrafine-grained 6061 Al composite at a temperature of 853 K and a strain rate of 2.5×10-4 s-1 due to this grain refinement. The effects of severe plastic deformation on the microstructure and superplasticity of 6061 Al composites are discussed in the present study.

New Developments in Geometric Dynamic Recrystallization: *M. E. Kassner*[1]; [1]University of Southern California, Aeros. & Mechl. Engrg., OHE 430, Los Angeles, CA 90089-1453 USA

The concept of geometric dynamic recrystallization (GDX) originated in 1985 with work on elevated-temperature deformation pure aluminum to large strains. In this case, substantial grain refinement occurs through a process of grain elongation and thinning leading to a dramatic increase in grain boundary area. The grain boundaries become serrated as a result of subgrain (low angle) boundary formation. Pinching off and annihilation of high-angle grain boundaries occurs as the original grains thin to about twice the subgrain diameter to and a ?steady-state structure. About 1/3-1/2 the subgrain facets remaining high angle boundaries, with this geometric dynamic recyrstallization. This concept has since been carefully verified in pure Al, as well as Al-Mg alloys deforming in the 3-power regime. Large strain deformation of Al single crystals are consistent with the concept. Recent experiments on alpha zirconium are show that GDX applies to this low SFE hcp metal. Also, data in the literature on large strain deformation of a bcc iron alloy is consistent with GDX. Thus, it appears that GDX is a general phenomena that can lead to grain refinement in the absence of any discontinuous dynamic recrystallization (DRX) or continuous dynamic recrystallization (CDX).

Microstructure and Anisotropic Mechanical Properties of Pure Ti Produced by Equal Channel Angular Pressing: *Ho-Kyung Kim*[1]; Woo-Jin Kim[2]; [1]Seoul National University of Technology, Dept. of Auto. Engrg., 172 Kongnung-dong, Nowon-gu, Seoul 139-743 S. Korea; [2]Hong-Ik University, Dept. of Matls. Sci. & Engrg., 72-1 Sangsu-dong, Mapo-gu, Seoul 121-791 Korea

Microstructure, texture analysis and anisotropic tensile properties of the 1, 2 pass ECAPed and unECAPed pure Ti were investigated. The unECAPed coarse microstructure became refined to finer grains after ECAP. Tensile properties in the transverse and longitudinal directions of Ti billets processed by route Bc were investigated. Work hardening was observed in all the samples. After ECAP, yield strength increased by 141, 103, 123, 129% for 1 passed transverse and longitudinal and 2 passed transverse and longitudinal direction samples, respectively. However, tensile ductility decreased by 68, 34, 37, 38%, respectively. The strength differential between the samples pulled along the transverse and longitudinal directions and its dependence on pass number is believed to be related to texture modification during ECAP. The microstructure and texture development in the Ti during ECAP process, and their relation with the strength are discussed.

Substructural and Phase Transformations During Plastic Deformation of Materials Obtained by Severe Plastic Deformation: *Nina A. Koneva*[1]; Eduard V. Kozlov[1]; [1]Tomsk State University of Architecture and Building, Dept. of Physics, Solyanaya sq. 2, Tomsk 634003 Russia

The paper summarizes the results of the studies of structural transformations that take place in UFG metals and alloys during deformation in tension or compression at room temperature. The studies of UFG Cu and its alloys are performed using TEM methods. Quantitative analysis of changes in (1) grain structure (grain size, density and types of boundaries), (2) dislocation structure (substructural transformations, scalar and excess dislocation density), (3) phase content (decomposition of the solid solution, deformational dissolution of small particles) and (4) the spectrum of stress fields from various sources is performed. The role of dislocational mass transfer, migration of grain boundaries and movement of triple junctions is emphasized. Contributions from thermally activated processes at moderate temperatures are considered.

High Strength and Ductility of UFG Metals and Alloys, Subjected to Severe Plastic Deformation: Nikolay Krasilnikov[1]; Alfred Sharafutdinov[2]; Witold Lojkowski[3]; Ruslan Z. Valiev[2]; [1]Ulyanovsk State University, 42, L. Tolstoy str., Ulyanovsk 432700 Russia; [2]Institute of Physics of Advanced Materials, UGATU, Ufa 450000 Russia; [3]Polish Academy of Sciences, High Pressure Rsch. Ctr., Warsaw Poland

The Ni and Al-based alloy 2024 after different methods of severe plastic deformation (SPD) are investigated. The equal channel angular pressing (ECAP) and cold rolling nickel, subjected also to high-pressure torsion (HPT), is characterized of large strain degree, homogeneous structure with grain size 120 nm and shows at room temperature the record strength with ultimate tensile stress (UTS) (1270 MPa), but limited plasticity (6%). The annealing at 200°C of Ni after ECAP and rolling leads to UTS about 900 MPa and ductility 12%. Al-based alloy 2024 with grain size 70 nm was obtained using HPT at room temperature. The nanostructured alloy at room temperature demonstrated record UTS above 1100 MPa, and superplastic behavior at temperature higher then 300°Ñ. The microhardness of nanostructured alloy after superplastic deformation (1.5 GPa) was more than after standard treatment of coarse-grained alloy (1.2 GPa). Opportunity of achievement in metals and alloys of combination high strength and good ductility opens perspectives of its application in industry, particularly, for mirco-systems and for details with complex geometry, obtained due to superplastic forming.

Significance of Grain Boundary Sliding in the Zn-22% Al Alloy After Processing by Equal-Channel Angular Pressing: *Praveen Kumar*[1]; Cheng Xu[1]; Terence G. Langdon[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Specimens of the Zn-22% Al eutectoid alloy were processed by equal-channel angular pressing (ECAP) through a total of 8 passes at 473 K using processing route BC. Following ECAP, the grain size was measured as ~1.0 μm. Tensile specimens, having gauge lengths of 4 mm, were cut from the as-pressed billets and tested at strain rates from 10^{-2} to 10^{-1} s^{-1}. Marker lines were placed on the specimens prior to testing and these markers were used to take measurements of the grain boundary sliding offsets at an elongation of 30%. Similar sets of measurements were also taken at a total elongation of 230%. Separate sliding measurements were taken for the three types of interfaces: Zn-Zn, Zn-Al and Al-Al. This paper describes the results obtained in this investigation and estimates the contributions of grain boundary sliding to the total strain.

Bulge Forming Characteristics of AZ 31 Sheet at Elevated Temperature: *Yong-Nam Kwon*[1]; Y.-S. Lee[1]; J.-H. Lee[1]; [1]KIMM, Matls. Procg. Dept., Sangnam 66, Changwon 641-010 Korea

Magnesium alloys have a huge potential for the structural applications due to the light weight and high specific strength. Until recently, die casting has been considered as the best way to fabricate components in practice since magnesium has such a low plastic formability. For more wide application of magnesium alloys, the plastic forming needs to be developed to ensure the high productivity with the reliability of the products. In the present study, superplastic behavior of AZ31 was studied on the respect of temperature and strain rate. Blow forming used for the investigation of the forming characteristics of AZ31 alloy with uni-axial tensile test. Formability was observed to enhance greatly with temperature increment. Strain rate sensitivity became over 0.5 over 400C below the strain rate of 10-3/s. Deformation assisted grain growth occurred in the superplastic deformation condition, while grains got refined below 250C due to dynamic recrystallization.

Development of Particle Free Zone During Superplastic Deformation in 7075 Al Alloy: *Yong-Nam Kwon*[1]; Y.-S. Lee[1]; J.-H. Lee[1]; Y.-W. Chang[2]; [1]KIMM, Matls. Procg. Dept., Sangnam 66, Changwon 641-010 Korea; [2]POSTECH, CAAM, Hyoja, San 31, Pohang 790-784 Korea

Particle free zones(PFZs) were observed to occur during the high temperature creep in many alloys containing second phase particles and often considered as a direct evidence for diffusional creep. These PFZs were also reported to develop during superplastic deformation of Al alloys. Therefore PFZ formation was suggested as a clear evidence of diffusional creep for superplastic deformation. However, it is still unclear how diffusion takes a role during superplastic deformation of Al alloys. The major features of the present study are to explain on the physical mechanism of a superplastic deformation and its corresponding PFZ development. The flow curves were separated into grain boundary sliding and accommodating parts. Deformed specimens on several deformation conditions were observed to investigate the evolutions of PFZ formation. Some new insights are presented on the development of PFZ during the superplastic deformation.

Cavitation and Failure in a Fine-Grained Superplastic Inconel 718 Sheet: Yi Huang[1]; *Terence G. Langdon*[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Olin Hall of Engrg., Rm. 430G, 3650 McClintock Ave., Los Angeles, CA 90089-1453 USA

Tensile tests were carried out at 950C over a range of strain rates to failure using the superplastic IN718 sheet. Cavity morphology observations and quantitative analysis (area fraction and average cavity size) were determined. The results are discussed including the possible mechanism of cavity development and the factors contributing to failure.

Structure and Mechanical Properties of Long-Sized Titanium Rods Processed by Severe Plastic Deformation: *Vladimir Vladimirovich Latysh*[1]; Irina P. Semenova[2]; Gulnaz Sadikova[2]; Ruslan Z. Valiev[2]; [1]Scientific Design Technical Office "Iskra", 81 Pushkina Str., Ufa, Republic Bashkortostan 450025 Russia; [2]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., 12 K. Marx Str., Ufa, Republic Bashkortostan 450000 Russia

Recently we have developed the technology consisting of equal-channel angular pressing (ECAP) and thermomechanical treatment (TMT) for fabrication of nanostructural Ti rods 6-12 mm in diameter and up to 1.5 meter long. The present work considers the homogeneity of microstructure and mechanical properties distribution along the length of CP Ti (Grade 2) rods 8 mm in diameter and up to 1 meter long, processed by the described technique. It was established that the scatter of mechanical properties along the length of a rod subjected to the treatment was within ±5%. It is shown that the processed long-sized Ti billets possess a homogeneous ultrafine-grained structure with a- grain size of 150 nm and highly homogeneous mechanical properties with yield stress value exceeding 900 MPa, which is almost two times as much as the same value of the initial annealed alloy with ductility constituting d•10.

Investigation of Precipitation and Deformation in Cryomilled Bulk Nanocrystalline Al-Mg Alloys Using Transmission Electron Microscope: *Zonghoon Lee*[1]; Enrique J. Lavernia[2]; Steven R. Nutt[1]; [1]University of Southern California, Matls. Sci., 3651 Watt Way, VHE-602, Los Angeles, CA 90089-0241 USA; [2]University of California, Dept. of Cheml. Engrg. & Matls. Sci., Bainer Hall, Davis 95616-5294 USA

The precipitation and deformation mechanism in cryomilled bulk nanocrystalline Al-Mg alloys were investigated using analytical and high resolution transmission electron microscope. Grain refinement was achieved by cryomilling of atomized Al-Mg powders, and then cryomilled nanocrystalline powders were consolidated by hot isostatic pressing followed by extrusion to produce bulk nanocrystalline alloys, resulting in high strength. The precipitations such as aluminum nitride and oxide were not easily resolvable in microscope because those precipitations were pretty small and often not fully crystallized. Thus the grown crystalline precipitations were investigated using both analytical and high resolution transmission electron microscope in this study. The enhanced strength of Al-Mg alloy might arise from the combination of ultrafine grain size and the existence of precipitates. The oxide and nitride content is also responsible for the unusual thermal stability and resistance to grain growth. This process demonstrates a novel approach to designing and producing bulk nanocrystalline metals that exhibit exceptionally high strength and acceptable toughness.

Microstructural Influence on Low-Temperature Superplasticity of UFG Ti-6Al-4V Alloy: *Young Gun Ko*[1]; Chong Soo Lee[1]; Dong Hyuk Shin[2]; [1]Pohang University of Science and Technology (POSTECH), Dept. of Matls. Sci. & Engrg., San 31, Hyoja-dong, Nam-gu, Pohang, Gyeongbuk 790-784 S. Korea; [2]Hanyang University, Dept. of Metall. & Matls. Sci., Ansan, Gyunggi-do 425-791 S. Korea

Microstructural influence on low-temperature superplastic behavior of ultra-fine grained Ti-6Al-4V alloy fabricated by ECA pressing was investigated in this study. For this purpose, a series of tensile tests was carried out on samples with equiaxed and lamellar microstructures at temperature range of 873K - 973K and at strain rates of 10-4/s - 10-2/s. After 4 ECA pressings, lamellar microstructures exhibited higher elongation than that of equiaxed microstructures at the same temperature and strain rate, which was attributed to the finer grain sizes of alpha and beta phases resulted from fragmentation of alpha/beta lamellae. When increasing ECAP strain from 4 to 8, elongation was significantly increased, which was associated with higher potion of high-angle grain boundary and lower activation energy. Finally, deformation mechanisms of ultra-fine grained microstructure were analyzed and discussed in the context of inelastic deformation theory.

A Crystal Plasticity Finite Element Analysis of Texture Evolution in Equal Channel Angular Extrusion: *Saiyi Li*[1]; Surya R. Kalidindi[2]; Irene J. Beyerlein[3]; Mark A.M. Bourke[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS H805, Los Alamos, NM 87545 USA; [2]Drexel University, Dept. of Matls. Sci. & Engrg., Philadelphia, PA 19014 USA; [3]Los Alamos National Laboratory, Theoretl. Div., Los Alamos, NM 87545 USA

A 3-D crystal plasticity finite element (CPFE) approach [Kalidindi et al., J. Mech. Phys. Solids, 40 (1992) 537] is applied to simulate the texture development during equal channel angular extrusion (ECAE) of metallic materials. With this approach, polycrystal plasticity constitutive equations are incorporated in a FE code. The material is represented by thousands of grains with each grain represented by one or several FEs, and each grain is assigned an orientation randomly selected from a set of orientations discretized from the initial texture. The performance of this approach is evaluated through quantitative comparison of the simulated textures with experimental results for a number of materials (aluminum, copper and interstitial-free steel) reported earlier. They are also compared to those predicted by the full constraints Taylor and viscoplastic self-consistent models. From the CPFE simulations, information about the local variations of deformation as a result of grain interactions is extracted from the deformation gradient components and discussed in relation to the differences in simulated textures compared to the other models.

Enhancing Ductility of AL6061+10wt% B4C Through ECAE Processing: *Thomas Martin Lillo*[1]; [1]Idaho National Engineering and Environmental Laboratory, Matls., MS 2218, PO Box 1625, Idaho Falls, ID 83415-2218 USA

Additions of high modulus particles to aluminum alloys offer the potential developing a high stiffness, lightweight composite. Powder metallurgy was used to create a metal matrix composite of aluminum alloy, 6061, and 10 wt% boron carbide particles. Characterization of the as-fabricated material showed the presence of agglomerates of B4C particles and some residual porosity. Evaluation of the mechanical properties showed little improvement to the elastic modulus, low tensile strength and no significant amount of ductility. The material was ECAE processed using a variety of parameters, e.g. temperature, number of passes through the die, route, intermediates anneals, etc. Subsequent microstructural characterization revealed redistribution of the B4C particles with evidence of B4C particle size reduction. ECAE processing also eliminated residual porosity and improved the elastic modulus. The biggest improvement was observed in the elongation to fracture which increased to >10%.

Cyclic Stress-Strain Response and Low-Cycle Fatigue Damage in Ultrafine Grained Copper: *Hans J. Maier*[1]; Pawel Gabor[1]; Ibrahim Karaman[2]; [1]University of Paderborn, Lehrstuhl f. Werkstoffkunde, Pohlweg 47-49, Paderborn 33098 Germany; [2]Texas A&M University, Dept. Mech. Engrg., 326 Engrg. Bldg., Coll. Sta., TX 77843-3123 USA

We report on the fatigue behavior of ultrafine grained copper obtained by equal channel angular extrusion (ECAE). Cyclic stress-strain response and fatigue life data were determined in low-cycle fatigue (LCF) tests. The early stages of the fatigue process were examined in a scanning electron microscope equipped with a small-scale load frame that allowed for in-situ fatigue observations. The actual ECAE route employed had a substantial effect on macroscopic cyclic stress-strain response. Despite these differences, all ECAEed samples displayed deviation from Masing behavior in LCF tests, but showed nearly perfect Masing behavior in incremental step tests. As expected, fatigue life was observed to depend on the actual ECAE process conditions. The in-situ studies helped to rationalize the effect of the microstruc-

ture on LCF life data, and it is concluded that a microstructure that leads to a more tortuous crack path will decrease small crack growth rate, which in turn increases fatigue life. The effects of the actual ECAE process routes on fatigue performance will be discussed, and the ramifications of the results obtained with respect to optimization of the microstructure will be addressed.

Ultra Fine Grain Interstitial Free Steels: *Joel Malaplate*[1]; Sandrine Guérin[1]; Jean-Pierre Chevalier[1]; [1]CECM-CNRS, 15, rue Georges Urbain, Vitry Cedex 94407 France

The development of steels with high specific strength is of major interest for the automotive industry both for energy saving. A much studied method of increasing yield stress is to reduce the grain size and rely on the Hall-Petch relation. For example, reducing the grain size of an interstitial free (IF) steel from 15 to 1 micron increases the yield stress by a factor of 3 to 4. Furthermore, since the steel composition is unchanged, recyclability is maintained. Here we present results on ultrafine grain IF steels produced by severe plastic deformation using equal channel angular extrusion and controlled recrystallisation. Special attention has been paid to the recrystallisation conditions in order to yield fine grain sizes, leading to high yield stress, together with acceptable work hardening and ductility. The microstructures obtained (grain size, dislocation structures) before and after deformation, as well as after recrystallisation have been examined by electron microscopy.

Unified Terminology for Strain Induced Boundaries: *Hugh J. McQueen*[1]; Enrico Evangelista[2]; Marcello Cabibbo[2]; [1]Concordia University, Mechl. Engrg., 1455 Misonneuve W., Montreal, QC H3G 1M8 Canada; [2]University Polytechnica della Marche, Mech., Via Brecce Bianche, Ancona I-60131 Italy

The terminology of strain ε induced or altered boundaries should reflect the mechanisms that create them and the function performed; both are related to the regions they surround or separate. Dislocation glide is the primary mechanism in high σ creep and hot, warm and cold working, being less influenced by recovery (dynamic DRV, static SRV) as temperature T falls and ε rate rises. Dependent on σ, boundaries contain dislocations that are geometrically necessary for the misorientations Ψ as well as dipoles that are more dense in cold working; cell walls are rather incidental whereas higher angle block walls enclose cell clusters having different slip systems. After SRV, or DRV in hot deformation, polygonized walls consist almost entirely of regularly arrayed, low-energy, geometrically-needed dislocations; such subgrain boundaries (SGB) continually rearrange during steady-state ε in a stable, σ-defined substructure. Due to Taylor-defined multiple-slip in poly-crystals, grains divide into deformation bands slipping on different systems and rotating into different texture components. The transition boundaries between bands are permanent and increase in Ψ with strain to become indistinguishable from grain boundaries (GB). Original GB extended with εare disturbed by lattice dislocations; at high T, they migrate locally at SGB to become serrated. Equilibrium GB, possibly from high Ψ dislocation walls, are able to migrate causing recrystallization (SRX or DRX).

Severe Plastic Deformation of Copper, Aluminum and Titanium Alloys: *Anuj Mishra*[1]; Marc A. Meyers[1]; Bimal Kad[2]; Robert Asaro[2]; Franck Grignon[1]; [1]University of California, Dept. of Mechl. & Aeros. Engrg., 9500 Gilman Dr., La Jolla, CA 92093-0418 USA; [2]University of California, Dept. of Structural Engrg., 9500 Gilman Dr., La Jolla, CA 92093-0085 USA

Equal Channel Angular Processing (ECAP) is being carried out with two goals: a) to understand the micro/nanostructural evolution during intense plastic deformation b) to produce Ti alloys with reduced grain size and superior mechanical properties. Mechanical test results are presented along with TEM pictures to examine structural evolution.

High Temperature Creep Behavior and Microstructure Analysis of Binary Ti-6Al Alloys with Trace Amounts of Ni: *Jun Ho Moon*[1]; S. Karthikeyan[1]; G. B. Viswanathan[1]; R. W. Hayes[2]; S. P. Fox[3]; M. J. Mills[1]; [1]Ohio State University, Dept. of Matls. Sci. & Engrg., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA; [2]Metals Technology Inc., 19801 Nordhoff, Northridge, CA 91324 USA; [3]Timet Inc., America Techl. Lab., W. Lake Mead & Atlantic Ave., Henderson, NV 89015 USA

High temperature creep behavior of two binary, equiaxed Ti-6Al alloys with varying trace amounts of Ni (<3ppm and 3ppm) was studied. Uniaxial compression tests were performed over temperature range 510 to 593°C at applied stresses ranging 150 to 300MPa. Stress and temperature jump tests were performed to obtain the stress exponents and the creep activation energy. Results show that increased amounts of Ni increased the minimum creep rates at all stress levels. Stress exponent values of ~5.0 were obtained for both samples. Detailed TEM analysis of the deformation structure was performed on samples crept monotonically at 200~300MPa upto 0.2~4.5% plastic strain. Results are explained with reference to the recently reported trends associated with lattice self-diffusion in alpha-titanium in the presence of fast diffusing impurities. From the TEM analysis of the dislocation structures, a modified jogged-screw model has been developed that provides quantitative predictions of the observed creep rates.

Microstructural Evolution During Simple Heavy Warm Compression of a Low Carbon Steel: *Susarla Venkata Surya Narayana Murty*[1]; Shiro Torizuka[1]; Kotobu Nagai[1]; [1]Metallurgical Processing Group, Steel Rsch. Ctr., Natl. Inst. for Matls. Sci., 1-2-1, Sengen, Tsukuba Sci. City, Ibaraki Ken 305-0047 Japan

The microstructure development in a low carbon steel (0.15% C) during heavy warm deformation (HWD) was studied using field emission scanning electron microscopy (FESEM) and electron back scattering diffraction (EBSD). Plane strain compression tests have been conducted in the temperature range of 773-923K at strain rates of 0.01/s and 1/s and the specimens were deformed to 25% of the original thickness. Ultrafine grains were noticed when the strain attained a critical value and varied with the Zener-Hollomon parameter (Z). In order to understand the combined effect of strain rate and temperature on the strain required for the formation of ultrafine ferrite grains, the variation of Z parameter was plotted against compressive strain. The evolved microstructures were classified into three regions based on EBSD data, namely (a) elongated grains; (b) mixture of elongated grains with newly generated grains, and (c) newly generated ultrafine grains. The microstructures in these three regions were further studied by transmission electron microscopy (TEM).

Structural Superplasticity of an Al Alloys in Low Strain Rate Regime - An Internal Variable Approach: *Ji Eon Park*[1]; Young Won Chang[1]; [1]Pohang University of Science and Technology, Matl. Sci. & Engrg., San 31, Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 Korea

On the superplastic deformation behavior the existing researches using the external variables such as total stress and strain have failed to explain the role of grain size and test temperature quantitatively. Recently, however, quantitative analysis for structural superplasticity has been progressed considerably with the use of the concepts of internal deformation variables. Nevertheless the effect of grain size and test temperature on flow characteristics in low strain rate region has not been elucidated precisely and several fundamental characteristics, such as the validity of threshold stresses and the role of grain size, are yet to be resolved or still in controversy in the superplasticity community. In this study, a series of load relaxation and tensile tests has been conducted to obtain the flow curves, which were consequently analyzed based on the internal variable theory for structural superplasticity, focusing especially on the low strain rate region.

Comparison of the Microstructure and Thermal Stability of an AZ31 Alloy Processed by Different Severe Plastic Deformation Processing Routes: Mohamed Eddahbi[1]; *Jorge Antonio del Valle*[1]; María Teresa Pérez-Prado[1]; Oscar Antonio Ruano[1]; [1]CENIM,CSIC, Physl. Metall., Avda. Gregorio del Amo, 8, Madrid, Madrid 28040 Spain

The aim of this work is to compare the microstructure, the texture, as well as the thermal stability of an AZ31 Mg alloy processed via two different severe plastic deformation (SPD) processing techniques, namely large strain hot rolling (LSHR) and equal channel angular pressing (ECAP). The microstructure was characterized by optical microscopy and the texture was measured both by X-ray diffraction (XRD) and electron backscatter diffraction (EBSD). The processing conditions were chosen to achieve similar strain levels using both routes. The microstructure obtained via LSHR is finer, with average grain sizes around 3 mm, but quite heterogeneous. During large strain hot rolling a well define basal texture develops. The sample processed by ECAE is comparatively more homogeneous and slightly coarser, with an average grain size of 7 mm. Simultaneously, a shear-type texture develops. It was found that the sample processed via LSHR was prone to heterogeneous grain growth and secondary recrystallization after annealing at 250°C for 15 h whereas the sample processed via ECAE underwent homogeneous grain growth under the same annealing conditions. The influence of the microstructure on the thermal stability is explored.

Severe Plastic Deformation Associated with [001] Single-Crystal W and W-Ta Alloy Ballistic Rod Penetration into Steel Targets: *C. Pizana*[1]; E. V. Esquivel[1]; L. E. Murr[1]; M. T. Baquera[1]; C. Y. Pina[1]; I. A. Anchondo[1]; L. S. Magness[2]; [1]University of Texas, Metallurgl.

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Examples of severe plastic deformation (SPD) associated with [001] oriented, single-crystal W and W-5%Ta alloy ballistic rod penetration into steel targets were observed in this study. Both zone-melt and CVD grown single-crystal rods were studied. Examination of in-target residual cross-sections of rod heads and rod erosion fragments was performed utilizing scanning and transmission electron microscopy (TEM) as well as optical metallography. Consistent with prior related studies, rod penetration (at 1.2 to 1.4 km/s) was facilitated by SPD induced solid-state flow, cleavage cracking, shear instabilities, and overlapping adiabatic shear bands characterized by dynamic recrystallization (DRX); observed by both optical metallography and TEM. The erosion of the [001] penetrators as a consequence of the solid-state flow creates an erosion tube of primarily DRX penetrator material which flows from the penetrator head and can particulate behind the penetrating rod, within the penetration channel. Some zones within the penetrator cleave and/or flow as large crystalline blocks on thin DRX zones. Microhardness evidence suggests that the penetrator head is heavily deformed by the initial shock compression and penetration, and the penetrator flows by DRX in a surface zone. For example, the initial CVD W [001] rod Vickers microhardness was 417 in contrast to average values of 550 near the embedded rod head center, and values of 450 in the surface-related flow zone. This research was supported by the U.S. Army Research Laboratory (Aberdeen Proving Ground, MD) under contract No. DATM 05-02-C-0046 (TO#6).

Developments in Nanostructured TiNi-Based Shape Memory Alloy: *Vladimir G. Pushin*[1]; Vladimir V. Stolyarov[2]; Ruslan Z. Valiev[2]; Terry C. Lowe[3]; Yuntian T. Zhu[3]; [1]Ural Division of Russian Academy of Sciences, Inst. of Metal Physics, 18 S. Kovalevskaya St., Ekaterinburg 620219 Russia; [2]Ufa State Aviation Technical University, Inst. of Advd. Matls., 12 K. Marx St., Ufa 450000 Russia; [3]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

In this paper, we present the processing, microstructure and properties of nanostructured TiNi-based shape-memory alloys synthesized by severe plastic deformation (SPD) methods, including torsion under high pressure (HPT), equal-channel angular pressing (ECAP), and complex combined deformations (SPD plus rolling or drawing). It is found that the SPD processing altered the phase transformation sequence of the alloy as well as the morphology of the martensite. Also, we found that the mechanical and shape memory properties can be enhanced by forming nanostructures in these alloys. More specifically, SPD processing renders higher reactive stress (up to 1.5 GPa) and reverse deformation (up to 10%) of shape memory, which are desired for various medical and other engineering applications. Several examples of medical and engineering applications of nanostructured shape memory TiNi-based alloys will be presented.

Equal-Channel Angular Pressing in Parallel Channels: *Georgy Iosifovich Raab*[1]; [1]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., 12 K. Marx St., Ufa, Republic Bashkortostan 450000 Russia

The efficiency increase of ultrafine-grained (UFG) structure formation in metallic materials is directly connected with the number of passes, type of the processing route as well as with the level of hydrostatic pressure in deformation zone. In this connection, the scheme of equal-channel angular pressing (ECAP) in parallel channels seems to be the most appropriate one. In this scheme, the material is two times subjected to successive shear localized deformation during one press stroke, which allows to decrease the number of processing cycles, to increase structure homogeneity as well as hydrostatic pressure in deformation zone of the 1st shear process due to thrust deformation, provided by the 2nd shear process. These factors contribute to the formation of more refined and homogeneous structure. The advantages of the processes have been studied on copper M1 subjected to ECAP in parallel channels.

Nanostructure and Thermal Stability of Two AlXSc Alloys Processed by SPD: *Hans J. Roven*[1]; Hakon A. Nesboe[1]; Borge Forbord[2]; Jens C. Werenskiold[1]; [1]Norwegian University of Science and Technology, Matls. Tech., Trondheim N-7491 Norway; [2]SINTEF, Matls. & Chmst., Trondheim N-7491 Norway

Severe plastic deformation (SPD) has been used to create ultra-fine grains in two different AlXSc alloys, i.e. one alloy with a high diffusivity and the other with a low diffusivity ternary element. Both alloys are pressed in the same ECAP tool in two conditions (homogenized and not homogenized). Ultra fine grains are formed after ECAP in both alloys and an even finer distribution of Sc and/or ScZr disperoids are sub-imposed to the grain structure. The dispersoids are effectively locking down the grain boundaries. Also, the nano-structured grains embed some nanodomains with crystallographic twin character. The alloys are subjected to ordinary mechanical testing, superplastic tensile testing, calorimetric investigations and thermal stability annealing. Post ECAP thermal recrystallization is strongly dependent on the solution temperature for disperoids. Finally, the roles of the Sc containing dispersoids in regard to properties and nanostructures are briefly discussed.

Fracture Properties of Nanostructured Materials Processed by Severe Plastic Deformation: *Ilchat Sabirov*[1]; Vladimir V. Stolyarov[2]; Ruslan Z. Valiev[2]; Reinhard Pippan[1]; [1]Erich Schmid Institute of Materials Science, Jahnstrasse, 12, Leoben 8700 Austria; [2]Institute of Physics of Advanced Materials, K. Marx str., 12, Ufa 450000 Russia

In recent years, bulk nanostructured materials processed by methods of severe plastic deformation (SPD) have attracted the growing interest of specialists in materials science. The mechanical properties of these materials have been intensively studied. But in spite of a vast amount of publications devoted to this topic, the fracture properties of nanostructured materials processed by SPD have not been investigated so far. This work deals with the effect of SPD on the fracture behavior of materials. The object of this investigation is a Ti subjected to equal channel angular pressing (ECAP) for 2 and 8 passes. The microstructure of the material in all investigated conditions is studied. Standard mechanical tensile tests are performed to determine the tensile mechanical properties. Disk compact tension specimens are tested to determine the J-Äa curves. A great effect of ECAP on the morphology of the fracture surface is observed. The value of the crack tip opening displacement at the moment of fracture initiation (CODi) is measured by a fracture surface analysis system. The evolution of the fracture properties and fracture behavior, namely, the fracture toughness, the slope of the J-Äa curves dJ/d(Äa), the maximum extension of the stable crack propagation, etc., with the number of ECAP passes is studied and will be discussed.

Analysis of High Strength State of Ultrafine-Grained TI-6AL-4V ELI Alloy Processed by Severe Plastic Deformation: *Irina Petrovna Semenova*[1]; Yuntian Theodore Zhu[2]; Terry C. Lowe[3]; Georgy Iosifovich Raab[1]; Lilia Rashitovna Saitova[1]; Ruslan Zufarovich Valiev[1]; [1]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., 12 K. Marx St., Ufa, Republic Bashkortostan 450000 Russia; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [3]Metallicum, 1207 Callejon Arias, Santa Fe, NM 87501 USA

As we showed earlier, equal-channel angular pressing (ECAP) and further extrusion of two-phase titanium alloy Ti-6Al-4V contributes to a considerable enhancement of strength properties up to UTS [3]1400-1450 MPa as a result of grain refinement. The current work presents the detailed study of the microstructure of medical Ti-6AL-4V ELI alloy subjected to the described treatment. It was established that the morphology of a- and b-phases in the initial state before ECAP had great effect on the microstructure refinement and the alloy mechanical behavior. The influence of various structural parameters (ultrafine grains, disperse phase precipitates, supersaturated solid solution, etc.) on the alloy strengthening was analyzed. It was found that strength and fatigue properties could be enhanced by means of the control of a grain size and phase morphology of the initial structure before ECAP as well as after ECAP processing and further thermal and thermomechanical treatment.

Characteristics of Aluminum 6061-T6 Deformed to Large Strains by Machining: *M. Ravi Shankar*[1]; Srinivasan Chandrasekar[1]; W. Dale Compton[1]; Alexander H. King[2]; [1]Purdue University, Sch. of Industrial Engrg., IE, GRIS, 315 N. Grant St., W. Lafayette, IN 47907-2023 USA; [2]Purdue University, Sch. of Matls. Engrg., 315 N. Grant St., W. Lafayette, IN 47907-2023 USA

A study has been made of large strain deformation characteristics of heat-treatable aluminum alloys (6061-T6) by analyzing chips created by plane strain machining. By varying the geometry of the tool, different levels of strain were imposed in the material chip in a single pass of machining. The chip micro-hardness values were found to be somewhat greater than the hardness values reported for Al 6061-T6 in Equal Channel Angular Pressing (ECAP). Also, chips deformed to larger strains were generally harder than chips deformed to smaller values of strain. Microstructure of the chips was found to be composed of relatively equi-axed grains with mean size in the range of 80-150 nm. The microstructure and hardness were found to be relatively stable even after extended periods of annealing. Small but statistically significant differences were found between the annealing behaviors of the chips deformed to different strains, with chips deformed to larger strains being less thermally stable vis-à-vis their less deformed counterparts.

A Multi-Grain Model for Subgrain Formation and GB Sliding: *A. Simone*[1]; E. van der Giessen[1]; [1]University of Groningen, Dept. of Applied Physics, Micromech., Nyenborgh 4, Groningen 9747 AG The Netherlands

Recent investigations have reported superplastic flow at high temperature and high strain rates in various coarse-grained polycrystalline materials, including some Al-alloys. Although the precise mechanism is not yet well understood, there are many indications that it involves a combination of dislocation creep and grain boundary sliding, facilitated by plasticity-induced grain refinement and subgrain formation. To contribute to a better understanding of the main mechanisms behind superplastic flow in coarse-grained polycrystalline aggregates, we present a multi-grain finite-element model that incorporates these phenomena. The model is used here to study the effect of both grain size, shape and distribution in the initial stages of superplastic flow. Special emphasis is placed on the analysis of cell formation with subsequent cell evolution into subgrains and eventually into real grains. This leads to the breaking down of coarse grains which would allow massive superplastic flow to proceed.

Ti-6Al-4V Alloy Flange Production by Isothermal Roll Forming Process: *Youngil Son*[1]; [1]ADD, R&D Ctr., Yusung PO Box 35-5, Daejeon 305-600 S. Korea

The high cost and poor machinability of Ti alloys makes them prime candidates for net-shape manufacturing techniques. Isothermal forming of Ti alloys enables net-shape forming at comparatively lower loads. This paper will show the manufacturing process and metallurgical evaluation of thin walled Ti-6Al-4V alloy flange by isothermal roll forming process. The process of isothermal roll forming employs rollers to shape a cylindrical workpiece into a complex symmetric shape by simultaneously adjusting the roll shape and by moving the rolls radially outward on the workpiece while it is rotated about its axis of symmetry. Both the workpiece and the rolls are maintained at temperatures close to the beta transus. The evaluations of the flange included microstructure, crystallographic texture, heat treatment response, tensile strength. The flange microstructures were found to be uniform and without any strongly textured colonies. Mechanical properties of the roll formed flanges were compared with those of conventionally forged flanges.

Al-Cu-Mg-Li (Mn, Zr, Sc) Alloys for Age-Forming of Damage Tolerant Curved Structures: *Marco Jan Starink*[1]; Nong Gao[1]; Nicolas Kamp[1]; Ian Sinclair[1]; [1]University of Southampton, Matls. Rsch. Grp., Sch. of Engrg. Sci., Highfield, Southampton SO17 1BJ UK

Age forming has substantial potential cost benefits for the production of curved aluminium structures. This technique is currently applied to production of upper wing skins of commercial aircraft, but is not currently applied for lower wing skins as incumbent lower wing skin alloys lose their damage tolerant properties upon ageing. On the basis of modelling of strength and general metallurgical understanding, a series of Al-Cu-Mg-Li (Mn, Zr, Sc) alloys predicted to provide good proof strength (PS) and a good balance of damage tolerant properties are designed and manufactured through casting and rolling. Extensive experimental work has been conducted on these alloys by metallography, hardness testing, tensile testing, fatigue testing, fracture testing, DSC, SEM/EBSD, TEM and 3DAP analysis. After artificial ageing representative of age-forming several of the newly designed alloys have PS, fatigue crack growth resistance and toughness that are comparable or better than incumbent 2024-T351. Strategies for counteracting the reduction in UTS-PS are discussed.

Analysing the Shear Zone for Metals Deformed by Equal-Channel Angular Processing: *Grigoreta Mihaela Stoica*[1]; Douglas E. Fielden[1]; Robert L. McDaniels[1]; Peter K. Liaw[1]; Cheng Xu[2]; Terence G. Langdon[2]; [1]University of Tennessee, Matl. Sci. & Engrg., 323 Dougherty Engrg. Bldg., Knoxville, TN 37996-2200 USA; [2]University of Southern California, Depts. of Aeros. & Mechl. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

The plastic deformation during the Equal-Channel-Angular Processing (ECAP) of metals concentrates in a shear zone, which is located close to the plane separating the two channels of the die. Scribed split billets were used in order to experimentally observe the flow field in the shear zone for different transient states of the billet subjected to ECAP. Spatial distributions of the strain and strain rate were evaluated using an analytical procedure developed for orthogonal cutting. The experimental results are compared with the theoretical predictions of the shear strain obtained from the slip-line models. An analysis of the shearing nonuniformity across the billet was conducted for dies having both sharp and round corners.

On the Effect of SPD on Recycled Experimental Aluminium Alloys: Nanostructures, Particle Break-Up and Properties: *Przemyslaw Szczygiel*[1]; Hans J. Roven[1]; Oddvin Reiso[2]; [1]Norwegian University of Science and Technology, Matls. Tech., Trondheim N-7491 Norway; [2]Hydro Aluminium AS, R&D Matls. Tech., Sunndalsora 6600 Norway

As a part of on-going research on the effects of recycled alloy chemistries on alloy design, properties and applications, the present work focuses on SPD processing. An ECAP tool is used to process different Al alloys with “recycled alloy chemistry”. A comparison between deformation routes A and Bc is performed to evaluate the effects of deformation path on particle break-up and particle distribution. The particle break-up sequences are followed under intrinsically measured strain conditions and along different flow paths in the ECAP tool. Also, the effects of friction and deformation temperature on particle structures are studied. Phase characterization is performed by microprobe, HR-SEM, TEM and calorimetric investigations. Local deformation zones around particles are studied in order to reveal the local strain gradients in the nano-to micro scale. Finally, both dynamic presipitation during ECAP and post ECAP aging characteristics as influenced by particle structures are qualitatively described.

Superplastic Joining of Y-TZP Enhanced by Titania-Doping in the Insert Material: *Yorinobu Takigawa*[1]; Hiroaki Takadama[2]; Kenji Higashi[1]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., Grad. Sch. of Engrg., 1-1 Gakuen-cyo, Sakai, Osaka 599-8531 Japan; [2]Japan Fine Ceramics Center, Matls. R&D Lab., 2-4-1 Mutsuno Atutaku, Nagoya, Aichi 456-8587 Japan

Superplastic joining of 3mol% Yttria stabilized tetragonal zirconia polycrystal (3Y-TZP) is examined using 5wt%TiO2-doped 3Y-TZP as an insert material, in order to decrease the joining temperature and stress and to realize the local deformation near the joining surface. The joining tests are conducted by uniaxial compression in the temperature range of 1523 to 1723K. The joined specimen is characterized by scanning electron microscopy (SEM), showing a clean interface with no cavitation. Four point bending tests are conducted to evaluate the joining strength, resulting that an average flexural strength of 960MPa is obtained at 1573K compressed by 10MPa for 30 min, which is the 80% of the strength in 3Y-TZP matrix. The temperature of 1573K is 100K lower to obtain similar strength without the insert. Fracture occurs not from joining surface but from matrix in bending teat. Residual stress must be introduced during joining process.

Distinguishing Ambient-Temperature Creep Region in Deformation Mechanism Map of Annealed CP-Ti: *Hisamune Tanaka*[1]; Tomoyasu Yamada[1]; Eiichi Sato[1]; Itaru Jimbo[2]; [1]Institute of Space and Astronautical Science/Japan Aerospace Exploration Agency, Space Struct. & Matls. , 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan; [2]Tokai University, Faculty of Engrg., 1117 Kitakaname, Hiratsuka, Kanagawa 259-1292 Japan

Ambient-temperature creep behavior of typical H.C.P., F.C.C. and B.C.C. metals and alloys of annealed state were investigated at a stress levels below their yield stresses. Metals and alloys having H.C.P. structure showed large creep deformation. Among them, CP-Ti showed significant creep deformation. Therefore, activation energy and stress exponent were measured in order to compare the deformation mechanism between ambient and high-temperature creep. The activation energy of ambient-temperature creep indicated much lower than that of high-temperature creep and the stress exponent of the former indicated much higher than that of latter. Based on those creep parameters, ambient-temperature creep region for annealed CP-Ti was distinguished in the Ashby-type deformation mechanism map.

Effects of Microstructures on the Creep Rupture Properties and Fracture Mechanisms in Austenitic Heat-Resisting Steels: *Manabu Tanaka*[1]; Ryuichi Kato[2]; Junnosuke Taguchi[2]; [1]Akita University, Faculty of Engrg & Resource Sci., Rsch. Inst. of Matls. & Resources, Dept. of Mechl. Engrg., 1-1 Tegatagakuen-cho, Akita, Akita Prefecture 010-8502 Japan; [2]Akita University, Grad. Sch., Dept. of Mechl. Engrg., 1-1 Tegatagakuen-cho, Akita, Akita Prefecture 010-8502 Japan

Effects of microstructures on the creep-rupture properties were investigated using the aged specimens of the SUS304 steel and the 21Cr-4Ni-9Mn (21-4N) steel at 973 K. Size distribution of cracks was also examined during creep. The grain size dependence of creep and creep-rupture properties (especially, creep ductility) was very different in these steels because of difference in the fracture mechanisms. Grain-boundary fracture occurred in the 21-4N steel, and a mixed mode of grain-boundary fracture and transgranular fracture was observed in the SUS304 steel, although grain-boundary sliding controlled the crack initiation at grain boundaries. Cumulative size distribution of creep cracks could be fitted to a power law at the larger crack sizes in the 21-4N steel, while the cumulative size distribution could be ap-

proximated by an exponential law in the SUS304 steel. These experimental results coincided with the results of simulation on the growth and linkage of creep cracks.

Microstructures and Mechanical Properties of Weldments from Extruded and Flowformed Ti-6Al-4V Alloy Tubes: *Ibrahim Ucok*[1]; Lawrence S. Kramer[1]; Mehmet N. Gungor[1]; Philip Wolfe[1]; Hao Dong[1]; Wm. Troy Tack[1]; [1]Concurrent Technologies Corporation, MTEC, 100 CTC Dr., Johnstown, PA 15904 USA

The main objective of this study was to perform weldability studies on seamless Ti-6Al-4V structural tubes manufactured by deformation processes such as extrusion, rotary piercing and flowforming to compare weldment properties. The tubular materials were butt-joined by plasma arc welding. The welded tubes were then subjected to a stress relief treatment prior to extraction of specimens for microstructural examination, tensile testing and fatigue testing. The effect of tube forming methods and heat treatments on microstructure and mechanical properties of the weldments are presented, correlated and discussed. This work was conducted by the National Center for Excellence in Metalworking Technology, operated by Concurrent Technologies Corporation, under Contract No. N00014-00-C-0544 to the Office of Naval Research as part of the Navy Manufacturing Technology Program.

Nanocrystallization of a Nickel Alloy Subjected to Surface Severe Plastic Deformation: *Juan C. Villegas*[1]; Kun Dai[1]; *Leon L. Shaw*[1]; Peter K. Liaw[2]; [1]University of Connecticut, Dept. of Metall. & Matls. Engrg., 97 N. Eagleville Rd., U-3136, Storrs, CT 06269 USA; [2]University of Tennessee, Dept. of Matls. Sci. & Engrg., Rm. 427-B Dougherty Engrg. Bldg., Knoxville, TN 37996-2200 USA

Surface nanocrystallization and hardening (SNH) process is a surface severe-plastic-deformation (SPD) process that has been applied to bulk specimens of Hastelloy C2000®, a nickel-base alloy. A microstructural gradient that is a function of the processing conditions was obtained and analyzed in order to discern the mechanism responsible for the observed grain refinement. Many structural and microstructural defects, such as stacking faults, high dislocation density and deformation twinning, were found to be present in the deformed surface layer. The role of these defects in the surface nanocrystallization and its implications in strengthening are discussed.

On the Influence of Processing Temperature During Severe Plastic Deformation of Iron Based Materials: *Andreas Vorhauer*[1]; [1]Austrian Academy of Sciences, Erich Schmid Inst., Jahnstrasse 12, Leoben 8700 Austria

Armco iron, a ferritic and an austenitic steel were subjected to severe plastic deformation (SPD) in order to achieve a refinement of the initially coarse grained microstructures down to microstructural sizes less than 100nm. A specially designed High Pressure Torsion (HPT) tool provides isothermal (+/-5°C) materials processing in a range of temperature between room temperature and 450°C. The aim of this work is to analyze the influence of the processing temperature on the microstructural refinement during SPD as a function of the applied strain (maximum 32). The morphology and the microtexture of the investigated materials were analyzed in a scanning electron microscope by capturing micrographs with the detector for backscattered electrons and measuring orientation maps with the Electron Back Scattering technique, respectively. In selected cases transmission electron microscopy investigations were performed. The relation between different microstructural features (as microstructural size and microtexture) and mechanical properties obtained from micro tensile tests are discussed.

Micro-Mechanics Modelling on the Toughening of Nano-/Micro-Meter Grained Composite Microstructure: *Jing Tao Wang*[1]; Dun Yan[2]; Zhong Ze Du[2]; [1]Nanjing University of Science and Technology, Sch. of Matls. Sci. & Engrg., No.200 Xiaolingwei, Nanjing 210094 China; [2]Xi'an University of Architecture and Technology, Sch. of Metallurgl. Engrg., Xi'an 710055 China

Markedly toughening effect was observed in advanced materials with a composite microstructure of ductile phase dispersed in high strength/hardness less ductile matrix, in the macroscopically elastic stage of amorphous alloys with ductile dendritic dispersions, and in the crack propagation stage of ceramics with ductile inclusions. Similar toughening effect was observed in the plastic stage in bulk nano-crystalline materials with dispersions of micro-meter grained microstructure constituents. Modelling of this toughening effect in nano-/micrometer grained composite microstructure was carried out in this paper based on micro-mechanics. It is found through modelling that the toughening effect is dependent on microstructure parameters like ductile phase dispersion, volume fraction, dimension and aspect ratio; And the encapsulation of the ductile phase by the surrounding high strength less ductile matrix phase is found to crucial for this toughening effect.

Microstructure and Texture Evolution During ECAP of an AlMgSi Alloy: Observations, Mechanisms and Modeling: *Jens C. Werenskiold*[1]; Hans J. Roven[1]; [1]Norwegian University of Science and Technology, Matls. Tech., Trondheim N-7491 Norway

Equal channel angular pressing has been used to create severe plastic deformation (SPD) nanostructures in a standard aluminium alloy at room temperature. Advanced characterization in a modern FEG-SEM with a state-of-the-art microdiffraction unit has been performed on samples carefully prepared from different positions in the process shear zone. Intrinsic strain measurements are done in parallel in order to describe the actual strain tensor in each position studied. This technique is used to discover actual mechanisms for grain break-up and the formation of nano-sized grains. In addition, HREM-TEM investigations reveals that deformation twins are formed and is therefore one of the operating mechanisms. The texture evolution seems to fit the believed deformation micro-to-nano mechanisms and can also be linked to specific deformation structure elements of certain crystallographic orientations. Based on the observations, a crystallographic model describing the main mechanisms for the creation of ultra-fine grains is proposed and briefly discussed.

Strain Hardening During High Pressure Torsion Deformation: *Florian Wetscher*[1]; [1]Erich Schmid Institute of Material Science, CD Lab. for Lokal Analysis of Deformation & Fracture, Jahnstraße 12, Leoben 8700 Austria

Severe Plastic Deformation (SPD) has been applied to different materials (Copper, Armco-Iron, pearlitic steels) by means of High Pressure Torsion (HPT). In this study the shear stress during deformation was measured in situ under different hydrostatic pressures. By applying a simple model for a strain hardening material, a shear stress - shear strain curve can be fitted and the influence of the hydrostatic pressure can be studied. These results are compared to microhardness measurements, tensile tests and the microstructural evolution during deformation.

Creep Deformation of Ordered Intermetallic Alloys: *K. Xia*[1]; [1]University of Melbourne, Dept. of Mechl. & Mfg. Engrg., Parkville, Victoria 3010 Australia

Many ordered intermetallic alloys possess high heat resistance and low density and are being developed for high temperature structural applications. Consequently, their creep resistance is of great significance. In this review, the general creep behaviour observed in a group of selected ordered intermetallics will be compared to that in disordered alloys. Creep deformation mechanisms will be analysed in view of the unique crystal structures, bonds, defect structures and diffusion processes. Effects of microstructure will then be discussed with a view to developing highly creep resistant intermetallic materials.

Processing of Medium Carbon Steel by Hot Pressing Prior ECAP: *Jozef Zrnik*[1]; *Jaroslav Drnek*[1]; Zbysek Novy[1]; *Libor Kraus*[2]; [1]COMTES FHT Ltd., Borska 47, Pilsen 301 00 Czech Republic; [2]COMTES FHT Ltd., Pilsen 301 00 Czech Republic

Intensive plastic deformation of a number of steel grades in conjunction with controlled thermal process yields very fine preliminary microstructure providing favourable mechanical properties. Medium carbon steel containing 0.45% carbon, prior to inserting severe plastic deformation, was repeatedly hot repeatedly press forged between flat swages. Uniform and fine dynamically recrystallized structure of ferrite-pearlite mixture with grain size of about 2 ìm resulted from this specific thermomechanical treatment. Cementite within nest-like pearlite colonies retained rod-like morphology. The total effective strain of å = 3-5 inserted to specimen was estimated by numerical simulation (FEM). In order to release accumulated stress (material strengthening) and evaluate the stability of obtained structure static annealing treatment was introduced to deformed samples. In dependence on annealing temperature and hold time the cementite colonies decomposed to form more or less spherical cementite precipitates. The further grain refinement mechanism during equal channel angular pressing following processing route B was explored at each pass of repetitive pressing. The steel was subjected to five pressings. Employment of this processing route resulted in further refinement of ferrite grains. The submicrometer order ferrite grains enclosed by serrated and low angle boundaries were formed within the former ones. Transmission electron microscopy examination revealed that these boundaries resulted from slip systems interactions. Cementite particle modified the constitution of newly born substructure.