2008 Nanomaterials: Fabrication, Properties, and Applications: Device

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee
Program Organizers: Wonbong Choi, Florida International University; Seong Jin Koh, University of Texas at Arlington; Donna Senft, US Air Force; Ganapathiraman Ramanath, Rensselaer Polytechnic Institute; Seung Kang, Qualcomm Inc

Tuesday AM Room: 273
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Donna Senft, US Air Force; Seong Jin Koh, University of Texas at Arlington

8:30 AM Invited
From Nanogenerators to Nano-Piezotronics: *Zhong Lin (Z.L.) Wang*[1]; [1]Georgia Tech

Developing novel technologies for wireless nanodevices and nanosystems are of critical importance for in-situ, real-time and implantable biosensing. It is essential to explore innovative nanotechnologies for converting mechanical energy (such as body movement, muscle stretching), vibration energy (such as acoustic/ultrasonic wave), and hydraulic energy (such as body fluid and blood flow) into electric energy that will be used to power nanodevices without using battery. We have demonstrated an innovative approach for converting nano-scale mechanical energy into electric energy by piezoelectric zinc oxide nanowire (NW) arrays.[1,2] We have recently developed DC nanogenerator driven by ultrasonic wave, which is a gigantic step towards application in practice. A field of nano-piezotronics has been proposed.[3] [1]Z.L. Wang and J.H. Song, Science, 312 (2006) 242-246. [2]X.D. Wang, J.H. Song J. Liu, and Z.L. Wang, Science, 316 (2007) 102-105. [3]Z.L. Wang "Nano-piezotronics", Adv. Mater., 19 (2007) 889.

8:55 AM Invited
Nanomaterials for Energy Conversion and Storage: *Ryne Raffaelle*[1]; [1]Rochester Institute of Technology

We will review some of the exciting new developments in the use of nanomaterials in energy conversion and storage for space power applications. In particular, we will present results on synthesis, purification, and characterization of single- and multi-wall carbon nanotubes and their use in lithium ion batteries, as well as other potential space power applications. We will also discuss how these materials are being used in concert with a variety of other nanomaterials to further improve device efficiencies and performance. Finally, we will also review some of the work we have been engaged with recently concerning the use of epitaxially grown InAs quantum dots on GaAs for use in multi-junction space solar cells. A particular focus in this talk will be given to the potential improvements to specific power and ancillary behavior of power devices which is beneficial to space application, such as thermal stability and radiation tolerance, afforded through the use of nanomaterials.

9:20 AM
Hyperbranched Nanowires of PbS and PbSe and Their Applications in Photovoltaics: *Song Jin*[1]; [1]University of Wisconsin-Madison

The discovery of multiple exciton generation in nanocrystals of semiconducting PbS and PbSe promises a 800% quantum efficiency limit and 65% theoretical photovoltaic conversion efficiency. We report a chemical vapor deposition synthesis of hyperbranched single-crystal nanowires of PbS and PbSe. Multiple levels of nanowires nucleate perpendicularly from the previous generation of nanowires in an epitaxial fashion to produce a dense cluster structure of a complex nanowire network. No intentional catalyst was employed for the nanowire synthesis, but it is suggested that lead itself might serve as a vapor-liquid-solid (VLS) catalysts for the anisotropic growth of PbS/PbSe. The flow rate and duration of a hydrogen co-flow are found to have significant effect on the PbS/PbSe nanowire nucleation and growth. We will explain the formation mechanism of these intricate network structures and other complex exotic PbS/PbSe nanostructures, and explore these nanostructures in high performance photovoltaic applications.

9:35 AM Invited
Monodisperse Anatase TiO_2 Nanotubes: Template-Directed Synthesis and Applications for High - Efficient Solar Energy Conversion: Changdeuck Bae[1]; Hyunjun Yoo[1]; Sihyeong Kim[1]; Kyungeun Lee[1]; *Hyunjung Shin*[1]; [1]Kookmin University, National Research Laboratory for Nanotubular Structures of Oxides and School of Advanced Materials Engineering and Center for Materials and Processes of Self-Assembly

Template-directed synthesis strategy is an ideal tool to fabricate oxide nanotubes in that their physical dimensions can be precisely controlled and monodisperse samples can be harvested in large quantity. Wall thickness of the oxide nanotubes is controllable with a high precision by varying the deposition cycles of atomic layer deposition (ALD), and the length and diameter can be tailored in accordance with the templates used. A wealth of functional oxide materials with the controlled polymorphs can be deposited to be nanotubular structures by various synthesis methods. Here we present our recent progress made in the template synthesis and applications of anatase TiO2 nanotubes. (i) We first discuss the template synthesis strategy that combines porous membrane templates and ALD techniques, addressing the processing issues associated with coating inside nanoscale pores, selectively etching of oxide nanotubes from the templates, and dispersion against the formation of nanotubes' bundle-up. (ii) We then report on bamboo-like self-organization of the anatase grains along the axis during the amorphous-anatase transitions at 400-500°C being responsible for their transport properties. (iii) We also studied electrical transport properties of both individual and bundle anatase nanotubes on the basis of conventional thermionic emission model and found that they have a superior conductivity (over three orders of magnitude) to that of bulk. (iv) Toward solar energy conversion applications, we explored their surface defect structures, which are strongly related to harvesting efficacy of photo-oxidized electrons, based on photoluminescence measurements. (v) Finally, we constructed dye-sensitized solar cells (DSSCs) devices consisting of our anatase TiO_2 nanotubes and preliminarily came by high conversion efficiency (~ 8%) of the devices. (vi) Their potential for other applications such as drug delivery systems, (bio)sensors, and photocatalysts are introduced based on our preliminary results.

10:00 AM Invited
Nanomaterials in Space Power Generation: *John Merrill*[1]; [1]Air Force Research Laboratory

Photovoltaics continue to be the primary source for electric power for space missions. The need for ever higher power, specific power, areal power density, and radiation resistance continues to push development of novel solar cell technologies. To meet present and future space power requirements, conventional crystalline multijunction solar cells, next generation thin-film solar cells, and novel technologies are being pursued. In the near term, III-V based multijunction solar cell efficiencies are being increased through incorporation of new materials and metamorphic structures. These efforts are expected to result in AM0 solar cell efficiencies of 33-35%, however, theoretical efficiency limits for such structures are typically less than 40%. For significantly increased efficiency and radiation hardness, so-called "third generation" technologies are being pursued by many. These approaches have theoretical efficiencies around 70%; practical efficiencies well in excess of 40% are possible. Third generation concepts include intermediate band structures, hot carriers, multi-exciton, and nanocomposite approaches. These technologies will be discussed in relation to space military and commercial needs as well as the unique requirements attributable to the harsh space environment.

10:25 AM
The Usage of Nano-Structure for Direct Harvesting of the Nuclear Particles Energy as Electricity: Liviu Popa-Simil[1]; *Claudiu Muntele*[2]; [1]LAVM LLC.; [2]AAMU

Most of the exothermic nuclear reactions transfer the mass defect or binding and surplus energy into kinetic energy of the resulting particles. These particles are traveling through material lattices, interacting by ionization and nuclear collisions. Placing an assembly of conductive-insulating layers in the path of such radiation, the ionization energy is transformed into charge accumulation by polarization. The result is a super-capacitor charged by the moving particles and discharged electrically. Another more promising solution is to use bi-material nanoparticles organized such as to act like a serial connection and add the voltage. A spherical symmetry fission products source coated in several nanolayers is desired for such structures. The system may operate as dry or liquid-immersed battery, removing the fission products from the fissile material. There is a tremendous advantage over the current heat flow based thermal stabilization system allowing a power density up to 1000 times higher.

10:40 AM
Continuous Manufacturing and Novel Coating Technologies of Photocatalytic Nanoparticles: *Christian Wögerer*[1]; Nils Stelzer[2]; Humbert Noll[3]; [1]Profactor Research and Solutions GmbH; [2]Austrian Research Center Seibersdorf - ARC; [3]University for Applied Science Wiener Neustadt

A continuous manufacturing technology by high pressure mixing reactors, the HPMN reactor, is a production method for Nanoparticles with the Possibility of functionalsazion and aftertreatment in one step and he will open the door for the Industries to a cost efficient mass production of functionalized Nanoparticles and Nanoproducts. The HPMN-reactor combines the flexibility of precipitation with the possibilities of controlling the particle characteristics and the ability of continuous processing. Combined with new photocatalytic particles and coating technologies in the same plant it is possible to realize the production from materials to a ready product with new photocatalytic properties in only one equipment. In a two year common research project 5 Austrian research institutes and 9 companies from different markets develop new photocatalytic materials, production equipment and photocatalytic applications.

10:55 AM Break

11:10 AM Invited
Functional Wide Bandgap Semiconductor Nanowire Devices: *Stephen Pearton*[1]; D. Norton[1]; B. Kang[1]; F. Ren[1]; H. Wang[1]; L. Tien[1]; J. Lin[1]; [1]University of Florida

We have fabricated many types of ZnO and GaN nanowire devices, including MOSFETs, diodes, UV sensors and pH sensors. ZnO has been effectively used as a gas sensor material based on the near-surface modification of charge distribution with surface-absorbed species. The large surface area of the nanorods makes them attractive for gas and chemical sensing, and the ability to control their nucleation sites makes them candidates for high density sensor arrays.

11:40 AM
Large-Scale Fabrication of Single Electron Devices: *Vishva Ray*[1]; Ramkumar Subramanian[1]; Pradeep Bhadrachalam[1]; Seong Jin Koh[1]; [1]University of Texas at Arlington

Fabrication of single electron devices requires defining source-drain electrode gap in the range of 10 nm as well as precise positioning of Coulomb island between the electrode gap. Achieving these nanoscale geometrical requirements over a large area for large-scale device fabrication is a formidable task. Here we present new single electron device architecture and its large-scale fabrication using CMOS technology. This has been possible by employing vertical electrode configuration as well as spontaneous and precise positioning of Coulomb islands between vertically placed electrodes. We demonstrate Coulomb staircases at room temperature using 10 and 20 nm Au nanoparticles as Coulomb islands. The voltage intervals of the Coulomb staircases were ~55 and ~25 mV for 10 and 20 nm Au nanoparticles, respectively, which is in good agreement with calculated single electron charging energies. The current demonstration implies that practical fabrication of integrated systems of single electron devices is now quite feasible.

11:55 AM
Electron-Phonon Interaction in Nanodevices: *Karel Kral*[1]; [1]Institute of Physics, Academy of Science of Czech Republic

Theory of the nanotransistor will be presented with the effect of the electronic multiple scattering on LO phonons included. The electronic transport part of the theory will be based on the well-known simple model, in which the quantum dot-contact coupling will be expressed with help of the electronic energy level broadening in a zero-dimensional nanostructure. The effect of the up-conversion of electronic level occupation will be included in the transport equations.

12:10 PM Invited
Nanoscale Heterojunction Engineering and Selective Growth of High-Quality Ge on Si by Molecular Beam Epitaxy: *Sang Han*[1]; [1]University of New Mexico

Growing a lattice-mismatched, dislocation-free epitaxial film on Si has been a challenge for many years. Herein, we exploit nanoscale heterojunction engineering to grow high-quality Ge epilayer on Si. A 1.2-nm-thick chemical SiO_2 film is produced on Si in a H_2O_2 and H_2SO_4 solution. When the chemically oxidized Si substrate is exposed to Ge molecular beam, relatively uniform nanoscale seed pads form in the oxide layer and "touchdown" on the underlying Si substrate. Upon continued exposure, Ge selectively grows on the seed pads rather than on SiO_2, and the seeds coalesce to form an epilayer. The resulting density of threading dislocations is ≤ 1E5 per square cm. In this presentation, we will discuss the reasons behind the selective growth as well as reduction in strain density near the Ge-Si heterojunction, leading to high quality Ge epilayer.

12:40 PM
Microstructure and Magnetic Properties of Nickel Nanorod Arrays on Silicon Substrate: *Chien-Ming Liu*[1]; Chih Chen[1]; [1]National Chiao Tung University

In this work, Ni nanorod arrays were grown on Si substrate using anodized aluminum oxide (AAO) as a template. The Ni nanorods were formed in the nanopores of the AAO film by electroless plating. The microstructure and magnetic property of Ni nanorod arrays on Si were examined by field emission scanning electron microscopy, transmission electron microscopy (TEM), and superconducting quantum interference device. The results show that the Ni nanorod is nanocrystalline, about 70 nm in diameter. It is found that these Ni nanorods appear to be superparamagnetic after they were deposited. However, after annealing at 400°C for few minutes, their magnetic properties changes from superparamagnetism to ferromagnetism. TEM results shows that grain growth occurred during the annealing process and thus change their magnetic properties.

3-Dimensional Materials Science: Large Datasets and Microstructure Representation II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee
Program Organizers: Michael Uchic, US Air Force; Eric Taleff, University of Texas; Alexis Lewis, Naval Research Laboratory; Jeff Simmons, US Air Force; Marc DeGraef, Carnegie Mellon University

Tuesday AM — Room: 286
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: David Rowenhorst, Naval Research Laboratory; Anthony Rollett, Carnegie Mellon University

8:30 AM Invited
Design and Implementation of an Interactive Repository for Materials Microstructural Data: Paul Dawson[1]; *Donald Boyce*[1]; [1]Cornell University

We discuss the concepts underlying the design of a repository for data related to the structure and properties of structural alloys. The design follows from the intended functionality of the repository, allowing for access by parties that store data in the repository and by parties that view or work with the data. The organization of data mimics the nature of the material, beginning with its constituent elements to define an alloy and branching to domains that reflect its state in terms of the attributes of its microstructure. The design anticipates that new motivations for accessing the data will emerge once the system is implemented and can facilitate a wide array of applications via an interface for application plug-ins. We will discuss the software for organizing the data (a relational database) and for storing the data (the repository) as well as the graphical user interfaces for communicating with these.

9:00 AM
Development of a Methodology for the Prediction of 3D Grain Structure from 2D Measurements: *Michael Groeber*[1]; Michael Uchic[1]; Dennis Dimiduk[1]; Somnath Ghosh[2]; [1]Air Force Research Laboratory; [2]Ohio State University

Currently, there remains a disconnect between the ability to investigate microstructure in 3D and its widespread use. Though many tools have matured to allow for robust data collection, availability continues to be limited to much of the community. Thus, it is still a reality to investigate materials in 2D and infer 3D characteristics. Stereological relationships have provided 3D information with limited data collection. However, there are microstructural descriptors that cannot be inferred from 2D measurements. In addition, many stereological relationships yield only average quantities to describe features. These limitations provide areas for improvement in predicting 3D structure from 2D measurements. Advances in collection of 3D microstructural data offer insight into the quality of stereological methods. True 3D microstructures serve as an answer key to stereological approaches, highlighting shortcomings in various techniques. This presentation will develop new, more robust, prediction tools using the true 3D structure as a validation tool.

9:20 AM
Reconstructing 3D Polycrystals: *Anthony Rollett*[1]; S. Lee[1]; S. Sintay[1]; [1]Carnegie Mellon University

Three-dimensional polycrystals can be assembled or reconstructed from statistical information on grain shape by packing ellipsoids into a representative volume element (RVE). A specified distribution of size and shape of ellipsoids can be used as input, from which the packing algorithm selects a subset according to an objective function that minimizes gaps and overlaps. The set of packed ellipsoids is converted to a space filling polycrystal by operating a cellular automaton in which each grain is grown from the ellipsoid center using its axes to specify (anisotropic) growth rates. The resulting polycrystal typically has a slightly different size distribution than the input and the junctions between boundaries do not conform to local equilibrium. Relaxation of the microstructures with a Monte Carlo grain growth model is used to examine the potential for generating realistic polycrystals with specified size distributions.

9:40 AM Break

10:00 AM Invited
A Data Driven Approach for Generating Reduced-Order Stochastic Models of Random Heterogeneous Media: *Nicholas Zabaras*[1]; Baskar Ganapathysubramanian[1]; [1]Cornell University

Stochastic analysis of random heterogeneous media provides information of significance only if realistic input models of the property variations are used. We will review a framework for constructing such input stochastic models using a data-driven strategy. This problem is analogous to the problem of manifold learning encountered in image processing and psychology. We showcase the methodology by constructing low-dimensional input stochastic models to represent property variations in two-phase and polycrystalline 3D microstructures. We will show how this model is used to model physical processes on the random microstructure. This framework has direct applicability to problems where working in high-dimensional spaces is computationally intractable, for instance, in visualization of property evolution, extracting process-property maps in low-dimensional spaces, among others. Furthermore, the generation of a low-dimensional surrogate space has major ramifications in the optimization of properties-processes and structures, making complicated operations like searching, contouring and sorting computationally feasible.

10:30 AM
Vectorized 3D Microstructure for Finite Element Simulations: *Asim Tewari*[1]; Vijayalakshmi Swamydhas[1]; Pinaki Biswas[1]; Raja Mishra[2]; Sooho Kim[2]; Anil Sachdev[2]; [1]General Motors Research and Development, India Science Laboratory; [2]General Motors Research and Development, Materials and Processes Laboratory

A technique to perform finite element analysis on a vectorized 3D microstructure is developed and applied to intermetallic particles found in continuous cast (CC) AA5754 and AA3005 sheets. CC sheets have particles of several phases (Al(FeMnSi), Al_6MnFe, etc) in different shapes and sizes (from 0.2µm to 10 µm) and heterogeneously distributed in stringers and centerline segregations. The technique consists of 3D reconstruction of the true microstructure through serial-sectioning and conversion of the 3D raster image to vector image. The vector image is constructed by union set operation of closed quadratic surfaces of tri-axial ellipsoids capable of approximating a wide range of shapes. This results in a 3D mesh generated automatically using Unigraphics and Hypermesh from real microstructural measurements, which can be imported to any FE code. Extension of this approach to create a composite input file combining particle distribution with EBSD measurement of grain orientations and morphologies will be discussed.

10:50 AM
Some Implications of Microstructure Representation below the Homogenization Limit: *Jeff Simmons*[1]; Dennis Dimiduk[1]; Marc De Graef[2]; [1]Air Force Research Laboratory; [2]Carnegie Mellon University

Statistical representation of microstructural information is becoming an important area of research, particularly with exponentially increasing volumes of experimental information becoming available. Synthetic microstructures are often generated with statistical properties consistent with the representation and used for physics-based simulations. This allows property scatter to be estimated by using multiple synthetic inputs. Local features such as particles generally are not symmetrical, but in the infinite limit, must be distributed to reproduce the macroscopic symmetry. Arbitrarily chosen statistical functions can be combined to reproduce the macroscopic symmetry, but will generally result in excessive variability at the local scale, resulting in an over-estimate of the property scatter. Recent work towards developing unbiased microstructure feature representations that bridge the homogeneous and local scales will be presented. Unbiased representations developed from Principal Component Analysis of sets of particle and particle neighborhood features will be presented, along with progress towards correctly matching the local scale variability.

11:10 AM
Mean Width Evaluation of Digital Microstructures in Relation to 3-D Grain Growth Theory: *Fatma Uyar*[1]; Seth Wilson[1]; Jason Gruber[1]; Anthony Rollett[1]; [1]Carnegie Mellon University

Mean width is an important measure for computational materials science, not only for grain shape representation but also for verification of 3-D von Neumann grain growth relation. Two methods have been employed for calculation of mean width of digital microstructures in a finite element mesh. Grains are composed of tetrahedral volume elements and grain boundaries are represented by interface triangles. The first method for mean width calculation uses the additivity rule on volume elements. The second method uses the flat surface rule. Measurements of net mean width and volume change rate are then compared to predictions of the theory of MacPherson and Srolovitz [Nature 446, 1053–1055(2007)] in a moving finite elements model of isotropic grain growth.

9th Global Innovations Symposium: Trends in Integrated Computational Materials Engineering for Materials Processing and Manufacturing: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: Global Innovations Committee, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Phase Transformations Committee, TMS: Powder Materials Committee, TMS: Process Technology and Modeling Committee, TMS: Shaping and Forming Committee, TMS: Solidification Committee, TMS: Surface Engineering Committee

Program Organizers: Corbett Battaile, Sandia National Laboratories; Amit Misra, Los Alamos National Laboratory; Joy Hines, Ford Motor Company; James Sears, South Dakota School of Mines and Technology

Tuesday AM — Room: 281
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Joy Hines, Ford Motor Company

8:30 AM
Processing and Mechanical Behavior of Nanoporous Platinum Films: *Antonia Antoniou*[1]; Nathan A. Mara[1]; Dhriti Bhattacharyya[1]; Elshan A. Akhadov[1]; S. Tom Picraux[1]; Amit Misra[1]; [1]Center for Intergrated Nanotechnologies, Los Alamos National Laboratory

We have synthesized nanoporous platinum films by electrochemical dealloying of amorphous PtxSi1-x films that were electron beam co-evaporated on a silicon substrate. Varying the Pt to Si ratio in the evaporated films and annealing the films after dealloying produced a range of ligament and pore sizes. For 20 at.% Pt-80 at.% Si films, the average pore and ligament sizes were 20 nm and 15 nm respectively. The Pt ligaments in these nanoporous films were polycrystalline with a grain size on the order of 5-10 nm. The mechanical properties of nanoporous platinum films are investigated through nanoindentation, including compression testing of focused-ion-beam machined micro-pillars, and compared to the properties of fully dense platinum as well as macro-scale metallic foams. The dependence of strength is studied as a function of film density as well as ligament size to gain insight on the length scale effects. We observe ultra-high strengths in nanoporous platinum films that are not explained by the scaling laws for the strength of bulk metallic foams as a function of foam density. The mechanical behavior of nanoporous metallic films is discussed in terms of deformation mechanisms involving nucleation of plasticity events at free surfaces. This research is supported by DOE, Office of Science, Office of Basic Energy Sciences.

8:55 AM
Application of a Multi-Physics Computational Model to the Casting of Actinide Metal: *Paula Crawford*[1]; Deniece Korzekwa[1]; Franz Freibert[1]; Tarik Saleh[1]; [1]Los Alamos National Laboratory

The casting of high purity plutonium metal is a process that is not well understood even after several years of study. The application of modern computer modeling and simulation techniques to materials processes can aid in providing valuable insight into the effects of process variables and mold design variations on the final casting product. Following this concept, a plutonium casting was prepared to produce parts of various thicknesses. The plutonium parts were examined and characterized in terms of microstructure, density, experimental cooling rates, etc. A computer model was developed to simulate the casting process using a multi-physics computational modeling package. Combining the results of the simulation with the experimental casting results should provide a better understanding of the effect of process variables on the casting of plutonium metal.

9:20 AM
Thermo-Mechanical Analysis of Continuous Casting with Reduction by Using Meshless Methods: *Lei Zhang*[1]; Yiming Rong[1]; [1]Worcester Polytechnic Institute

Meshless method can solve large deformation problem without re-meshing. An integrated analysis model of heat transfer and thermal stress based on meshless methods has been developed for continuous casting with mechanical reduction. The Finite Point Method is employed to build a thermal module. Solutions of the non-linear material properties and heat latent are given. The stress-strain analysis module is established based on the meshless Local Petrov-Galerkin method. The large deformation stress-strain is presented by Total Lagrange formulation, simultaneously the non-linear and contact problems are solved. Finally, the model is applied in the simulation of continuous casting slab. Temperature distribution and cooling history of the slab was calculated. Thermal stress evolution in the mold and the mechanical reduction regions are evaluated. The result predicted is consistent with the values obtained by theoretical analysis. Successful solution of large deformation problem also shows the potential benefits of meshless methods to continuous casting simulation.

9:45 AM
Simulation-Based Integration of Rapid Solidification to Fatigue Prognostic Predictions Using Laser-Deposited High Strength Materials as an Example: *Paul Wang*[1]; Haitham El Kadiri[1]; Mark Horstemeyer[1]; Liang Wang[1]; Gabriel Potirniche[2]; Sergio Felicelli[1]; [1]Mississippi State University; [2]University of Idaho

The goal of this research is to develop a set of simulation tools integrating thermal/solidification processes with fatigue prognostic models to demonstrate that predictive plasticity and ductility behavior of metallic materials shall be history dependent of upstream process steps. High strength materials made by laser engineering net-shaped (LENS™) process were used as an example. To understand and be able to virtually predict the service performance of LENS™ components, i.e., ductility, fatigue, and fracture strength, three-dimensional computer-assisted X-ray tomography (CT), nano-indentor, EBSD and estimates of dislocation network become a set of vital characterization tools for measuring initial and as-deformed states of materials. At the macro-scale, an internal state variable constitutive theory representing plasticity and hardening behavior is adopted for its ability of tracing deformation history and incorporating structure-property relationships. Results of integration schemes through rapid thermal/solidification, the resulting microstructure state of phase transformation, and its connection to prognostic modeling are discussed.

10:10 AM Break

10:20 AM
Atomistic Scale Study on Effect of Pressure on Densification during Sintering Nano Scale Tungsten Powder: Amitava Moitra[1]; Sungho Kim[1]; Seong-Gon Kim[1]; *Seong Jin Park*[1]; Randall German[1]; Mark Horstemeyer[1]; [1]Mississippi State University

Pressure assisted sintering is a well-known metrology to enhance densification by increase in the contact pressure of particles and thus the driving force for sintering, compared to pressureless solid state sintering. The effect of pressure is getting more important during sintering of nano scale particles with electrical field. In this study, atomistic simulation is used to gain insight into the fundamental characteristics of atomic movement with and without pressure before sintering and during sintering. The presentation will focus on particle deformation before sintering, material softening at elevated temperatures, temperature and pressure sensitivities, and particle size effect. The findings have a potential for nanoscale processing modeling and its optimization.

10:45 AM
Modeling Methods to Guide Recycling Friendly Alloy Design: The Impact of Compositional Data Structure: *Gabrielle Gaustad*[1]; Ray Peterson[2]; Randolph Kirchain[1]; [1]Massachusetts Institute of Technology; [2]Aleris International, Inc.

Recyclability is a context dependent materials property that has proven challenging to evaluate. However, previous studies by the authors have shown that certain modeling techniques, specifically chance constrained optimization, can provide a quantitative evaluation of recyclability. This information can be used to accelerate the evaluation and design of alloys (as well as products and operations) for improved recycling. One assumption in this work, however, is that the compositional variability of materials would be normally distributed. Previous work would indicate this may not be the case for all recycled materials. Performance, in terms of recyclability, may then be heavily influenced by the structure of the compositional data used. To test this, specific case studies involving several recycled material streams were investigated including both metals and non-metals. Results indicate that the underlying structure/distribution of the compositional data can have a profound effect on alloy design decisions to encourage usage of recycled materials.

11:10 AM
Heat Transfer Coefficient Study for Quenching by Experiment and CFD Modeling: *Gang Wang*[1]; Lei Zhang[1]; Qigui Wang[2]; Bowang Xiao[1]; Yiming Rong[1]; [1]Worcester Polytechnic Institute; [2]Powertrain Engineering

Heat transfer coefficient is a key issue in quenching process, and the HTC values obtained directly from experiments are limited to evaluate/predict the quenching ability of real production due to the complicated flow and workpiece geometry. In this paper, CFD method is employed to simulate the heat transfer and flow in the loaded experimental apparatus, which is used to test the temperature and the heat transfer coefficient. The experimental local HTC values are verified and modified for the whole workpiece by studying the details of the simulated temperature, velocity and pressure from CFD modeling. The HTC database is furthermore generated, and extended to include more values calculated by CFD modeling of quenching experiment design under the adjustable variables, e.g. temperature and velocity, but without experiments. Finally, the HTC database is applied to simulate heat transfer in real quenching production and verified by the measurement.

11:35 AM
A Microstructure-Sensitive Design Approach for Controlling Properties of HCP Materials: *Babak Kouchmeshky*[1]; Nicholas Zabaras[1]; [1]Cornell University

A model reduction technique based on the proper orthogonal decomposition method is presented for modeling the microstructure evolution of HCP polycrystals including slip and twinning. The polycrystal is represented by an orientation distribution function using the Rodrigues parameterization. This model can be used for representation and fast exploration of process/structure/property relations in HCP polycrystals. Using this model, a number of microstructure-sensitive deformation design problems are addressed for the control of texture-dependent properties. A continuum sensitivity-based gradient optimization framework is introduced for computing the variability of the microstructure evolution induced by perturbations on the macroscale velocity gradient. Design of both single and multiple stage processes is considered. Finally, integration of the material point design simulator with a multiscale design framework will be shown demonstrating the design of macro-process parameters for the control of microstructure-sensitive properties.

Alumina and Bauxite: Equipment

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Sringeri Chandrashekar, Rio Tinto Aluminium Limited; Peter McIntosh, Hatch Associates

Tuesday AM — Room: 296
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Gajendra Singh, Alcan Engineering Party Ltd.

8:30 AM Introductory Comments

8:35 AM
Advanced Filtration Methods for Pregnant Liquor Purification: *Reinhard Bott*[1]; Thomas Langeloh[1]; Juergen Hahn[1]; [1]Bokela GmbH

A new generation of backflush filters offers the possibility of improved pregnant liquor filtration. BOKELA have developed a continuous pressure filter of advanced mechanical and process design. The new designed and the specially arranged filter elements provide for high specific performance and large filtration area per vessel volume which exceeds status quo values. The new filter runs completely automated due to automatic and complete cake discharge and compared to the status quo technologies it provides for improved filter performance and operation with higher specific performance, improved filtrate clarity, higher concentrated solids discharge, improved mounting and lifetime of filter bags and lower idle times. Thus, the new filter offers the possibility of pregnant liquor filtration with improved performance, lower invest and operating cost. The lecture highlights modern demands on pregnant liquor filtration and presents design characteristics of the new backflush filter. Furthermore, future options of pregnant liquor filtration are discussed.

9:00 AM
Digestion Energy Efficiency at RTA Yarwun Alumina Refinery: Stephen Austin[1]; *Samantha Parry*[1]; [1]Rio Tinto Aluminum Yarwun

Community and shareholder awareness around environmental sustainability has increased industries accountability to deliver significant improvements in utilising energy more efficiently. The Greenfields development of the RTA Yarwun alumina refinery, commissioned in 2004, provided the opportunity to be world class in the alumina industry with respect to energy consumption through an innovative Digestion design. The Digestion tube design has demonstrated to be the most energy efficient for processing Weipa grade bauxite which is typically high in boehmite. The benefit of reduced water ingress, by eliminating the need for direct steam injection, translates to reduced plant evaporation requirements and boiler makeup water. The use of single stream also has the benefit of achieving a tighter balance between the energy required for slurry heating and energy generated by the flash vapour which sees very little excess steam generated by the blowoff system.

9:25 AM
Operational Experience with a Brownfield Expansion Project in Sayanogorsk, Russia: *Andreas Wolf*[1]; Peter Hilgraf[1]; Arne Hilck[1]; Sergey Marshalko[2]; [1]Claudius Peters Projects GmbH; [2]RUSAL, Moscow

This paper describes the installation, experience and operational improvements made after one year of operation for a full scale installation at an aluminium Brownfield expansion project in a Russian Aluminium Smelter. The operational conditions and challenges on the equipment of the so-called "FLUIDCON" pneumatic conveying system, mass flow: 135 tph, conveying distance: 400m, feeding device: screw feeder, will be described in detail. The alumina pot-feeding system ADS (Aerated Distribution System) is engineered and installed for feeding alumina into two pot rooms with 168 electrolysis cells 300kVA each. This paper concludes technical papers presented on earlier TMS Annual Meetings: [1]New Aerated Distribution (ADS) and Anti Segregation (ASS) Systems for Alumina 2002. [2]FLUIDCON - A New Pneumatic Conveying System for Alumina 2006. [3]A New Alumina Distribution and Feeding System for Aluminium Reduction Cells 2007.

9:50 AM
MAX HT™ Sodalite Scale Inhibitor: Plant Experience and Impact on the Process: *Donald Spitzer*[1]; Owen Chamberlain[1]; Calvin Franz[1]; Morris Lewellyn[1]; Qi Dai[1]; [1]Cytec Industries Inc

Development of a reagent to prevent sodalite scale formation in the Bayer process was presented at the 2005 TMS. Since that time this product has been run on many plants across the globe under various operating conditions and has demonstrated that it completely eliminates sodalite scale. This paper will cover the application experience gained and the benefits derived at the plants.

10:15 AM Break

10:30 AM
How to Achieve High Availability with Large Calciners and Avoid Unforseen Downtime: *Pekka Hiltunen*[1]; Roger Bligh[1]; Cornelis Klett[1]; Michael Missalla[1]; Hans-Werner Schmidt[1]; [1]Outotec GmbH

In the last 15 years, the largest available capacity for a new calciner has significantly increased. Large size calciners are being selected to both increase total refinery output and also to replace rotary kiln capacity. Factors that influence the decision to install these larger size calciners include reduced investment cost, operating costs and plant footprint per tonne of capacity. On the other hand, unforeseen downtime, particularly for large calciners can significantly jeopardize these anticipated savings and result in completely different economic outcomes: operating cost overruns and lost production. Parameters such as availability, operating factor, in relation to calciner capacity together with the hydrate filtration facility are identified. Operating data and the impact on production cost of unforeseen downtime based on existing calcination units are presented. Measures in areas such as design, construction, control, operation and maintenance are described to avoid unforeseen downtime during operation of the calcination facility.

10:55 AM
Customized Descaling Robots – Improving Worker's Health and Safety, Increasing Alumina Production Capacity: Clément Potvin[1]; Antonio Pucci[2]; *Claude Caya*[3]; Don Puxley[2]; Éloïse Harvey[3]; [1]Mecfor Inc.; [2]Alcan International; [3]Mecfor

It is a fact that alumina production capacity decreases as residue builds up on a reservoir's shell; nevertheless, descaling operations are often delayed because of the necessary downtime required to perform this maintenance operation. The purpose of this paper is to highlight the many advantages of descaling a reservoir, such as a bauxite digester, with a remotely controlled descaling robot, rather than by installing scaffolding and having workers manually remove the scale with jackhammers. Through real-life case studies in Alcan's plants, we will establish that a descaling robot dramatically improves the worker's working conditions, considerably reduces the downtime required to perform this critical operation, and greatly improves production capacity. Furthermore, it will be demonstrated that the Alcan/Mecfor descaling robot is highly adaptable to a variety of reservoir configurations and sizes, and is designed to work with many descaling methods.

11:20 AM
Determination of a Suitable Dewatering Technology for Filtration of Bauxite after Pipeline Transport: Adriano Campos[1]; *Reinhard Bott*[2]; Thomas Langeloh[2]; Juergen Hahn[2]; [1]Companhia Vale do Rio Doce; [2]Bokela GmbH

In order to expand alumina production Alunorte has to process bauxite from a new mine near Paragominas which is located in a distance of 244 km from the refinery. When CVRD was designing the mining and beneficiation of the Paragominas mine it was to decide by which means the bauxite should be supplied to the alumina refinery. Beside ship or railroad transport the most preferred option was pipeline transport of the bauxite. Pipeline transport of bulk materials such as kaolin or iron ore is not only a proved but also a more economical and environmental method than ship or railroad transport. Costs for pipeline transport are about 80% (?) below the alternative methods. At the refinery, however, the pumped slurry has to be dewatered and a decisive condition for realizing pipeline pumping of bauxite is to have a feasible filtration technology available which is capable to dewater the arriving slurry to a low residual moisture content in an economical way.

Aluminum Alloys: Fabrication, Characterization and Applications: Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Subodh Das, Secat Inc; Weimin Yin, Williams Advanced Materials

Tuesday AM — Room: 293
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Subodh Das, Secat Inc; Weimin Yin, Williams Advanced Materials; Zhengdong Long, Secat Inc

8:30 AM
Numerical Simulations of Texture Evolution in Polycrystalline Al during Channel Die Compression and Uniaxial Tensile Straining: *Fan Zhang*[1]; Allan Bower[1]; Raj Mishra[2]; [1]Brown University; [2]General Motors Corporation

Finite element simulations are used to study the evolution of local and global texture during uniaxial tensile straining and channel die compression of a polycrystalline pure Al. The simulations predict the overall texture in the crystal as well as to track the evolution of the orientation of individual grains. Results are obtained as functions of relevant loading and material parameters, as well as temperature, and the predicted texture and orientation evolution are compared with published experimental data. The simulations predict overall texture evolution that is in agreement with experiment, but the rotation of individual grains in the specimen does not match experimental observations. To explore this discrepancy, we study the influence of material parameters, the initial orientation of the grain, and the orientation of surrounding grains on the evolution of orientation within a particular grain in the specimen.

8:50 AM
Numerical Simulations of Void Growth by Coupled Grain Boundary Sliding, Diffusion and Dislocation Creep During Elevated Temperature Deformation of Al AA5083: *Ningning Du*[1]; Allan Bower[1]; Paul Krajewski[2]; Eric Taleff[3]; [1]Brown University; [2]General Motor Research and Development Center; [3]University of Texas at Austin

Finite element simulations are used to calculate void growth rates in Al alloy AA5083 during uniaxial tensile straining at elevated temperature. The finite element model incorporates grain interior dislocation creep, grain boundary diffusion and grain boundary sliding mechanisms. The simulations are used to predict the influence of applied strain rate, grain size, void volume fraction and stress triaxiality on the void growth rate. In particular, we contrast the cavitation behavior in the grain boundary sliding and dislocation creep regimes. The results are found to be in good agreement with experimental observations of cavitation in AA5083.

9:05 AM
The Use of Experimentally-Based Material Models in Simulating High-Temperature Forming of Aluminum and Magnesium Sheet: *Eric Taleff*[1]; Louis Hector[2]; Ravi Verma[2]; John Bradley[2]; Paul Krajewski[2]; [1]University of Texas; [2]General Motors R&D Center

High-temperature deformation of aluminum and magnesium sheet materials was investigated through experiments and simulations. Sheet materials were subjected to tensile tests and gas-pressure bulge tests at temperatures in the range of 450 to 500°C. Tensile test data over a range of strain rates were used to construct constitutive models of material behavior. Bulge tests were then simulated using a finite element methodology and these constitutive models. The results of the simulations were compared with bulge experiments. An experimentally-derived, two-term constitutive model based on creep phenomenology and experimental data was found to provide good agreement between simulations and experiments.

9:25 AM
Effect of Fe on Texture Evolution and Microstructures of the Cold Rolling AA5052 Alloy Sheets from Continuous Cast Processing after Anneal at Different Temperatures: *Xiyu Wen*[1]; Yansheng Liu[1]; Lirong Tong[2]; Tongguang Zhai[2]; Zhong Li[3]; Subodh Das[1]; [1]Secat Inc.; [2]University of Kentucky; [3]Aleris International Inc.

Texture characters of the cold rolling AA5052 alloy with the two Fe levels were determined and studied by use of the orientation distribution function method. The microstructures were observed by use of optical and scanning electronic microscopes. The effect of Fe on texture and microstructures of the alloy from continuous cast processing after anneal at temperature from 450F to 900F was analyzed and discussed. It was found that Fe influence recrystallization texture components and microstructures of the aluminum alloy. When Fe is low level, the Cube component is more.

9:45 AM
Aluminum Plate Hot Rolling Process Modeling: *Zhengdong Long*[1]; Yansheng Liu[2]; Randall Bowers[2]; Weimin Yin[2]; Shridas Ningileri[2]; [1]University of Kentucky; [2]Secat, Inc.

The hot rolling process plays a vital role in the microstructure, texture and tensile properties determination of the aluminum plate and sheet products. A multi-passes hot rolling process was simulated using the ABAQUS/Explicit algorithm. The heat transfer from the ingot to its environment and to the rollers as well as friction between the ingot and the rollers were defined. Industrial rolling parameters of ingot temperature, work roll rotation velocity, reduction rate, and initial ingot velocity were set as boundary conditions. A thermally coupled plain strain mesh type was used for the model. The flow stress as a function of plastic strain, strain rate and temperature was used in the material model. Temperature, plastic strain and stress evolution during the rolling process and its distribution along the through thickness direction were evaluated. The results indicate that the explicit dynamics approach produces a reliable solution for hot rolling simulations.

10:05 AM
Influence of Microstructure and Material Variables on Hemmability of Aluminum Alloys: *Rajesh Raghavan*[1]; Pinaki Biswas[1]; Raja Mishra[1]; [1]General Motors Research and Development

The influence of material and microstructure on hemmability of aluminum sheets are investigated using finite element analysis. First, the three separate steps of flanging, prehemming and hemming along with springback analysis after each of the steps are performed using a homogeneous material model. Macroscopic stress-strain data as the only input failed to discriminate materials for their hemmability. Next, a substructure modeling is performed wherein second phase intermetalics are added as a microstructural input. It is shown that even single subsurface elastic particle alters local stress distribution dramatically. Large particles located close to the surface or a string of particles have the largest effect. Different materials with similar particles exhibit different local stress enhancements. Since particles can crack when the local stress exceeds a threshold value leading to micro-cracking, this points to the need for simultaneous optimization of material properties and microstructure to improve hemmability.

10:25 AM Break

10:35 AM
Calculating Behavior of 5xxx in Marine Service: *Catherine Wong*[1]; [1]Naval Surface Warfare Center

5xxx aluminum alloys have long been used in Navy decks and bulkheads. Although the material is restricted to routine service below 65°C, deck temperatures on sunny days in southern latitudes can exceed this temperature. Thermocoupled 5xxx test specimens were situated on the flight deck and interior of the ONR research ship Seafighter in order to record the thermal profile seen by the material. 5xxx samples were thermally treated in furnaces with varying time and temperature followed by corrosion testing using ASTM G67 in order to develop the sensitization rates. The thermal profile and sensitization rates were combined to estimate the amount of sensitization to be expected on the Seafighter exposed specimens.

10:55 AM
Effect of Asymmetric Rolling on Texture and Anisotropy of AA6016 Alloy for Automotive Applications: *Linzhong Zhuang*[1]; Menno van der Winden[1]; L. Kestens[2]; J. Sidor[3]; [1]Corus Research, Development and Technology; [2]Delft University of Technology (Netherlands); [3]Netherlands Institute for Metals Research

Asymmetric rolling (ASR) was applied to aluminium alloy AA6016 with various circumferential velocity ratios between the top and bottom rolls. This gives rise to intense plastic shear strains, and in turn the shear deformation textures through the sheet thickness. The developed texture in asymmetrically rolled materials is weaker in comparison to that developed during conventional rolling. The recrystallization texture in asymmetrically rolled materials is also considerably weaker than that in conventionally rolled materials. With proper design of the ASR parameters, the shear deformation textures are still stable in recrystallized materials after SSHT. The presence of shear texture orientations significantly improves both the minimum r value and delta r value. Furthermore, with 25% ASR reduction and annealing, an average grain size is about 3 times smaller in comparison to that observed in conventionally rolled material.

11:15 AM
The Residual Stress Investigation in Al Using β-Particles Penetration Method: *Behrooz Salehpour*[1]; H. Pirhoseinloo[1]; M. Taheri Hashjin[1]; [1]University of Tabriz

Investigation of residual stress, RS, in a work-piece of industrial equipments is one of the very important test to finding out the lifetime and reliability of that device. In this work we tried to trace-out RS in the industrial Aluminium (99.5%) by means of measuring the penetration range, R_p and absorption coefficient (μ) of energetic β particles in the samples. Where plates of Al with different thickness were exposed to thermo-mechanical induced stresses and amount of R_p and μ of β particles were determined using a conventional nuclear instrumentation way. Hence the values of μ and R_p for the stressed samples were compared with that of the stress free (annealed) samples. Experimental results show increasing of the penetration range, and/or lowering the absorption coefficient μ of β particles for the cases of stressed samples.

11:35 AM
Numerical Simulations of a New Local Hot Forming Process for Making Imprints in Aluminium Extrusions: *Hallvard Fjær*[1]; Børge Bjørneklett[2]; [1]Institute for Energy Technology; [2]Hydro Aluminium

A local hot forming method to fabricate imprints in closed aluminium extrusions has been developed. This forming method involves an interplay between an induction coil heating a defined region for a few seconds, and a ceramic tool pressed subsequently into the profile. The process combines high formability with low forming loads and it allows forming of profiles without internal tools. The shape of the work piece is determined both by the movement of the tools and by local variation in flow stress resulting from large thermal gradients. Simulating this process requires a thermomechanical model incorporating the effects of temperature, microstructure and strain rate on the flow stress. Numerical simulations can be applied to assess the feasibility of a local hot forming process and to identify improved design of tools and forming procedures. Results from numerical simulation of forming of triggers in crash boxes of aluminium are compared with measurements.

11:55 AM
Orientation Gradient Evolution in Precipitation Hardening Aluminum Alloys: *Reza Shahbazian Yassar*[1]; Mark Horstemeyer[2]; David Field[3]; [1]Michigan Technological University; [2]Mississippi State University; [3]Washington State University

Development of sound physics-based plasticity models in precipitation hardening materials requires better understanding of the interaction between the nano-scale precipitates and orientation evolution of the individual grains. This investigation is concerned with characterization of in-grain orientation gradient as a function of precipitate characteristics. Several polycrystals of 6xxx series aluminum alloys containing hardening precipitates with different morphologies and characters were subjected to tensile deformation and dislocation structure evolution was investigated using high resolution electron backscatter diffraction and transmission electron microscopy. Local misorientation and geometrically necessary dislocation content were studied for various grains as a function of precipitate characteristics. It is found that precipitate characters affect the density and distribution of geometrically necessary dislocations as well as the misorientation boundaries within the grains.

12:15 PM
An Internal Variable Approach on Load Relaxation and Creep Transition Behavior of an Extruded Al-6061 Alloy: *Min Soo Kim*[1]; Young Won Chang[1]; [1]Pohang University of Science and Technology

The high temperature deformation behavior of an extruded Al-6061 alloy has been studied by using the load relaxation test and constant load creep test at temperature ranging from 300°C to 500°C. A series of load relaxation test has been carried out to obtain the flow curves. The change of strain rate sensitivity and creep transition of dispersion strengthened Al alloy were analyzed by using the internal variable theory proposed by Chang et al. The framework of the theory is built on the basis of well known dislocation dynamics to provide the concept of an internal strain tensor as the most fundamental deformation state variable. In addition, the constant load creep test was performed and the creep resistance of an extruded Al-6061 alloy was observed to enhance at the range of low strain rate and stress.

12:30 PM
Simulation of PLC-Effect in Al6061/Al_2O_3-Alloy: *Galina Lasko*[1]; Ye. Ye. Deryugin[2]; S. Schmauder[3]; [1]Institut für Materialprüfung, Werkstoffkunde und Festigkeitslehre, Universität Stuttgart; and Institute of Strength Physics and Material Science, SB RAS; [2]Institute of Strength Physics and Material Science, SB RAS; [3]Institut für Materialprüfung, Werkstoffkunde und festigkeitslehre, Universität Stuttgart

The instability of plastic flow in aluminium alloys in the form of PLC-Bands has attracted attention of scientific and metallurgical society. This is very extensively investigated and still less understood phenomenon. In the present contribution the attempt was made to investigate the mechanism of jerky flow in aluminium alloys by simulation of the evolution of the localized plastic deformation on mesoscopic scale level. The approach is based on the recently introduced and little known Relaxation element method. The unambiguous connection between the stress drop in the local volume of solid and plastic deformation in it is on the basis of this method. Based on this method the model of evolution of plastic deformation operates on principle of cellular automata, where each grain is considered as a proper hexagon, which can be in two states-plastically and elastically deformed. The influence of the following parameter on the patterns of the plastic strain localization has been investigated: externally applied strain rate, rigidity of the machine, the value of Young's modulus of the material. The results of simulations are accompanied by experimental findings. The ways of further improvement of the model are discussed.

Aluminum Reduction Technology: Cell Development Part I and Operations

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Aluminium Tech

Tuesday AM — Room: 298
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Paul Hemburrow, NZAS - New Zealand Aluminium Smelters Limited

8:30 AM
FECRI Approach and the Latest Development of AP3X Technology: *Oliver Martin*[1]; Serge Despinasse[1]; Claude Ritter[1]; Renaud Santerre[1]; Thierry Tomasino[1]; [1]Alcan

More than 3800 AP30 cells are presently operating in the world, 1500 additional AP30 cells will start in the coming years. In order to continuously improve the AP30 technology, Alcan has launched an intensive technology development program. The aim of the program is to push the amperage in the range 350-400 kA and to reduce specific energy consumption below 13000 kWh/t. In addition Alcan has started in January 2005 an internal project targeting a significant reduction of the full economic cost for a new smelter: the Full Economic Cost Reduction Initiative (FECRI). This article describes the operational tests allowing bringing the AP3X above 350 kA and the last improvements made in the modelling of the AP30 cell, especially the coupling between thermic and MHD. It shows great economical benefits resulting from FECRI for the future smelters.

8:50 AM
Expansion of the Potline in Slovalco: *Marvin Bugge*[1]; Milos Koniar[2]; Kamil Skladan[2]; Milan Stas[2]; [1]Hydro Aluminium; [2]Slovalco

The reduction technology in Slovalco, HAL230, was developed in the 80's. The potline was started at 230 kA in 1995. The amperage in the potline was gradually increased to 250 kA before the start up of the expansion in July-August 2003. The 54 expansion pots were started in 5 weeks at 250 kA. In the expansion several technical changes were introduced. The busbar system was improved, a new potshell was designed and new pot control, HAL3000, was applied. New types of cathode blocks were selected in cooperation with the cathode block suppliers. After 4 years of operation the expansion pots show good results with high current efficiency, low energy consumption and low anode effect frequency. The amperage in the potline has been increased to 258 kA. No expansion pot has been relined so far.

9:10 AM
The Alcan's P155 Smelters Now Operating at 195 kA: A Successful Assets Optimization Strategy: *Claude Richard*[1]; Patrice Desrosiers[1]; Louis Lefrançois[1]; Bruno Gaudreault[1]; [1]Alcan

Alcan started up its first Alcoa P155 technology potlines in 1980. During the 80's and 90's, Alcan improved both operational practices and computer control logic at its three P155 smelters resulting in a current efficiency increase of 3 % as well as a significant decrease of the anode effect frequency, therefore the PFC emissions. With a much more robust cell operation and benchmark performances, Alcan undertook, in 2001, an intensive technology development program aiming at increasing the amperage as well as reducing the specific energy consumption while maintaining excellent current efficiency and low emission performances. This article describes the industrial trials allowing the amperage increase up to 195 kA as well as the main key results at this amperage level. In addition to providing excellent technical performances, the P155 HALE program (High Amperage and Low Energy) was a successful assets optimization strategy resulting in a low investment and operating cost project for two Alcan plants in Canada, Grande-Baie and Laterrière smelters.

9:30 AM
Design Enhancements to GP320 Technology at Balco: *J. Ramaswamy*[1]; C. Sukumaran[1]; Gude Narendra Kumar[1]; [1]Bharat Aluminum Company

Bharat Aluminium Company Ltd (BALCO) is part of the Vedanta Resources Plc. BALCO, located in Korba district of Chhattisgarh State, India has enhanced its production capacity from 135000 tpa to 385000 tpa by constructing a state of art 320 kA prebaked pot line with 288 cells. In GAMI technology, if the Anode Beam-to-Beam aluminium welding plates for distribution of current or compensating bus bar gets damaged, the pot is cut off from the pot line circuit. To avoid this authors have designed a temporary bus bar connections, which can be fitted in the pots without cutting out the pot, till the rectification is done. In GAMI technology, to cut out a pot, complete power outage is required. The authors have developed a technical procedure, by which the pots are being cut out at full line load, without any reduction in the line current.

9:50 AM
The Continuous Development of Sami's SY300 Technology: Zhu Jia Ming[1]; Yang Xiao Dong[1]; Liu Ya Feng[1]; *Baoguo Chen*[1]; [1]Shenyang Aluminum and Magnesium Engineering and Research Institute(SAMI)

The first SY300 potline began operation in June 2002 in HeNan province. Today, there is a total of thirteen SY300 potlines in operation in China. In the past five years, SAMI has made vast improvement and continuously optimizing its SY300 technology. This paper describes features of the SY300 technology with focus on cell design, pot data and process control. Major developments made in the technology and the performance of a SY300 potline operating at 335kA will also be discussed.

10:10 AM Break

10:30 AM
Increase of Amperage at Sayanogorsk Aluminum Smelter: Victor Mann[1]; *Victor Buzunov*[1]; Oleg Burkatsky[1]; Alexander Krasovitsky[1]; Iliya Puzanov[1]; [1]United Company «Russian Aluminum»

In order to increase metal production in an operating area starting in 2001 until 2006 specialists of RUSAL Company's Sayanogorsk Aluminum Smelter and The Engineering and Technology Center have successfully realized a package of measures which facilitated an increase in the smelter's average amperage from 208.3 to 248.5 kA. Among these measures are: 1. mathematical modeling of the feasibility of an amperage increase for operating cell designs and finding ways to stabilize heat balance and MHD stability, 2. improved practice of forming and maintaining the anode carbon cover, 3. altered anode setting patterns, 4. evaluation of process capability of the potrooms to increase amperage, 5. upgrading of busbar of potline 4, and several other measures. The Amperage increase resulted in an increased output of the Sayanogorsk Smelter from 405,800 tons to 521,600 tons per year.

10:50 AM
Anode Signal Analysis: The Next Generation in Reduction Cell Control: *Jeffrey Keniry*[1]; Eugene Shaidulin[2]; [1]Alumination Consulting Ltd; [2]Russian Engineering Company, Engineering and Technology Centre

Reduction cells have achieved excellent control capability over the past 20 years by improved acquisition and treatment of the cell resistance signal, which provides the basis for regulation of alumina feeding, thermal balance and cell stability. As cells become larger in size however, there is an increasing need for control sensors that can recognize and react to spatial variation in the cell both in terms of anode performance and alumina feed control. Monitoring of individual anode current signals can provide the 'next generation' control capability that will be necessary to ensure that very large cells can deliver the same process efficiencies as their smaller predecessors. Coincidently, anode signals can now be acquired, stored and processed with far greater convenience and lower cost than was possible in the past, making an enhanced control capability within practical reach.

11:10 AM
Image Analysis for Estimation of Anode Cover Material Composition: *Jayson Tessier*[1]; Carl Duchesne[1]; Claude Gauthier[2]; Gilles Dufour[2]; [1]Laval University; [2]Alcoa, Inc

Anode cover material, a mixture of secondary alumina and electrolytic bath, is used to cover newly set anodes and to fill crust holes following crust meltdown. However, maintaining a constant composition over time is difficult since bath mass balance, from a potroom point of view, must be respected. Alumina and bath, particulate products with different properties, are mixed together and delivered to pot-tending-machines or hoppers. Hence, uneven mixing and segregation are frequently encountered, resulting in important deviations from target composition. To follow these variations, a few grabbed samples are analyzed on a daily basis which is not enough to capture the composition variations. Nevertheless, this composition plays an important role in keeping a good crust integrity ensuring good productivity and low gas emissions. Image analysis is presented in this paper, to extract features from anode cover material images that are related to alumina composition. Methodology and monitoring results are presented.

11:30 AM
An Automated Metal Purity Optimization System for Enhanced Revenue Capture: *Jason Lodzik*[1]; Michael Van Hauwermeiren[2]; Doc Murdoch[3]; [1]Century Aluminum; [2]Vose Consulting; [3]Crystal Ball (Oracle)

A project recently undertaken at the Century Aluminum reduction facility in Hawesville will increase metal sales revenue with a more efficient capture and sale of premium grade metal. Crystal Ball was utilized because of its optimization and adaptability capabilities. The project required a user-friendly automated application that could satisfy several constraints while easily altering goals based on changing market or plant conditions. Tests have proven a more consistent and optimal use of available purity metal has increased the premium metal stream from the potroom. Opportunities gained by an automated optimization tool come from eliminating human error, inaccurate inputs, and paper work. The optimization strategy must operate with a large number of physical constraints which creates a considerable level of discontinuity when compared to basic linear problems. The automated blending system has been developed and implemented in four purity lines. Key issues and findings from this project are described in detail.

11:50 AM
Impact of Alumina Fines Content on Potline Performance Ouro Preto Smelter: *Thiago Simões*[1]; João Martins[1]; Márcio Guimarães[1]; João Costa[1]; [1]Novelis

On the present paper we discuss the influence of alumina fines on pot performance and stability on HS Soderberg pots. The environment aspects are also taken into account; we discuss the challenge of compliance with the local environment agency requirements. Alumina size is an essential characteristic to be analyzed in the raw material of the primary aluminum production process. The presence of considerable quantity of fine particles – 325# screen undersize – generates a series of problem in potlines. Alumina fines play an important role in order to achieve process performance; operational results and environment can be seriously charged by changes in alumina fines. Supported by statisticals tools an evaluation is done to better understand the influence of alumina fines in the process.

Biological Materials Science: Bioinspired Design and Processing

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Ryan Roeder, University of Notre Dame; Robert Ritchie, University of California; Mehmet Sarikaya, University of Washington; Lim Chwee Teck, National University of Singapore; Eduard Arzt, Max Planck Institute; Marc Meyers, University of California, San Diego

Tuesday AM — Room: 390
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Marc Meyers, University of California, San Diego

8:30 AM Keynote
Designing Hard Tissue by Genetic Knock-In Approaches: *Malcolm Snead*[1]; Y. P. Lei[1]; W. Luo[1]; P. Bringas, Jr.[1]; S. N. White[2]; M. L. Paine[1]; [1]University of Southern California, Center for Craniofacial Molecular Biology; [2]University of California, Los Angeles

Enamel covers the teeth. Enamel is the hardest tissue in the vertebrate body, containing the longest biologically produced HAP crystallites. Enamel is the only ectoderm-derived tissue to biomineralize and unlike bone, contains no collagen and does not remodel. Enamel must be formed properly for it to survive a lifetime of mastication, in a wet, bacterial-laden environment. Ameloblasts are the cells that fabricate the enamel tissue, producing a tissue with unique materials properties under conditions amenable to life. Amelogenin is the dominant protein of forming enamel. Enamel tissue starts life during late gestation as a 100% protein matrix that undergoes self-assembly in the extracellular space to form a device capable of directing its own replacement by the mineral phase. Amelogenin contains highly conserved domains, suggesting their role during enamel biomineralization. Using protein engineering in vitro, we show the importance of these domains to self-assembly. We use homologous recombination technique to eliminate the native amelogenin gene, replacing it with engineered protein confirming the importance of these conserved domains to enamel biomineralization in vivo. We reduce the complexity of amelogenin protein isoforms used to biosynthesize a suitable enamel extracellular matrix, suggesting strategies for producing an enamel biomimetic. Supported by NIH, National Institute for Dental and Craniofacial Research.

9:10 AM Invited
Enzymatic Control of Biomineralizing Interfaces: Structuring of Mollusc Shells via Myosin-Dependent Chitin Synthesis: *Ingrid Weiss*[1]; [1]University of Regensburg

The multifunctional transmembrane structure of the mollusc myosin chitin synthase opens new horizons to explain various mollusc shell microtextures. Chitin self-assembly provides a dynamic extracellular biomineralization interface. Phase transitions that the chitin undergoes during enzymatic polymerization are likely to be monitored by the shell forming epithelial cells of the mollusc mantle tissue via their cytoskeleton. The mollusc myosin chitin synthase is thus identified as an outstanding key player in the cross-talk between mechano-chemical cell signaling, extracellular polymer self-assembly, mineralization, and the control of biomineralization related gene expression. Our current research indicates that supramolecular myosin chitin synthase arrays provide an effective and highly sensitive trans-cellular control mechanism. It can be speculated that the mollusc chitin synthase also bears a tremendous potential to contribute in both ways, enzymatically and via signaling pathways to the implementation of hierarchical patterns into chitin mineral-composites such as prismatic, nacre, and crossed-lamellar shell types.

9:40 AM
Mimicking Bone Formation via a Polymer-Induced Liquid-Precursor (PILP) Process: *SangSoo Jee*[1]; Laurie Gower[1]; [1]University of Florida

The intrafibrillar mineralization that is achieved during bone formation has eluded scientists who seek to duplicate this nanostructured architecture using conventional synthetic techniques. We have been able to achieve intrafibrillar mineralization using a polymer-induced liquid-precursor (PILP) process, in which acidic polypeptides transform the solution crystallization process into a precursor process by sequestering ions and generating liquid-liquid phase separation within the crystallizing medium. This fluidic mineral precursor can be drawn into the gaps of the collagen fibrils by capillary forces. Once the collagen is infiltrated with mineral precursor, the amorphous phase crystallizes, leaving the collagen fibrils embedded with HA nanocrystals. SAED and DF-TEM and selective etching studies reveal the interpenetrating structure of the mineral-collagen composite, which has a strikingly similar appearance to natural bone. We are currently examining biomaterials applications for the fabrication of synthetic bone graft substitutes which can replace the need for autograft and cadaver bone substitutes.

10:00 AM
Peptide Hydrogels for Cell Delivery: Lisa Haines-Butterick[1]; Daphne Salick[1]; Darrin Pochan[2]; *Joel Schneider*[1]; [1]University of Delaware, Department of Chemistry and Biochemistry; [2]University of Delaware, Department of Material Science and Engineering

Hydrogels are hydrated materials finding use in tissue regeneration efforts. The design of "smart" peptides that undergo sol-gel phase transitions in response to biological media enable minimally invasive delivery of extracellular matrix substitutes in-vivo. Towards that goal, we have designed a family of peptides that undergo triggered self-assembly to form a rigid hydrogel. When dissolved in aqueous solutions, these peptides exist in an ensemble of random coil conformations rendering them fully soluble. The addition of an exogenous stimulus results in peptide folding into β-hairpin conformation. This folded structure undergoes rapid assembly into a highly crosslinked hydrogel network whose nanostructure is defined and controllable. Peptides can be designed to fold and assemble in response to changes in pH or ionic strength, the addition of heat or even light. In addition to these stimuli, cell culture media is able to initiate gelation. When hydrogelation is triggered in the presence of cells, gels become impregnated with cells. A unique characteristic of these gels is that when a shear stress is applied, the gel will shear-thin. However, after the application of shear has stopped, the low viscosity gel quickly self-heals producing a gel with a mechanical rigidity nearly identical to the original hydrogel. This attribute allows gel/cell constructs to be delivered to target tissues via syringe where they quickly recover complementing the shape of the tissue defect. Gels are cytocompatible towards NIH 3T3 murine fibroblasts, mesenchymal stem cells, chondrocytes, osteoblasts and hepatocytes. As an added bonus, MAX1 hydrogels possess broad spectrum antibacterial activity suggesting that adventitious bacterial infections that may occur during surgical manipulations can be greatly reduced.

10:20 AM Break

10:30 AM Invited
Molecular Design of Peptides for Materials Assembly Using Genetic Engineering: *Candan Tamerler*[1]; Mehmet Sarikaya[2]; [1]Istanbul Technical University; [2]University of Washington

High selectivity and precise organization in biological systems lead to control of functions, in particular mechanics. Biological molecular building blocks, in particular proteins, in addition to lipids and polysaccharides, play a major role in biological design for controlled assembly of materials from the nanometer to the macroscale. In a cross-disciplinary approach at the confluence of materials and molecular biology, we utilize peptides as molecular building blocks in synthesizing, assembling, and fabricating materials systems for applications in engineering and medicine. Here, we first genetically select and then tailor inorganic binding peptides towards specific functions, as molecular linkers, couplers and growth modifiers. Using computational and experimental tools we study peptide molecular recognition of solids, kinetics of their binding and assembly. Here we describe molecular mechanical stability of engineered polypeptides in nano/nanobiotechnology with examples that include: i. Inorganic biofabrication through synthesis; ii. Molecular thin films iii. Molecular couplers. Supported by NSF/MRSEC-BioMat, TR-SPO programs.

11:00 AM Invited
Biomimetic Materials Processing: *Osamu Takai*[1]; [1]Nagoya University

Living organisms produce a wide variety of materials at room temperature and atmospheric pressure. Moreover, each material produced plays a key role in each function in biological systems. Biomimetic materials processing (BMMP) is defined as the design and synthesis of new functional materials by refining knowledge and understanding of related biological products, structures, functions, and processes. Hence BMMP is not a simple imitation of biological materials processes, but is advanced materials processing for bionics, electronics, photonics, mechanics, magnetics, medicine, and so on. BMMP, therefore, is closely related to nanotechnology and by using BMMP we can make a new nanotechnology named "biomimetic nanotechnology" which is based on lessons from nature. We are developing transparent ultra water-repellent surfaces imitating lotus leaves, three-dimensional cell cultures using ultra water-repellent surfaces, site-selective cell cultures using hydrophobic-hydrophilic patterning, and SAM (self-assembled monolayer) processing for nano/micro fabrications.

11:30 AM
A Paradigm for the Integration of Biology into Materials Science and Engineering: *Ryan Roeder*[1]; [1]University of Notre Dame

The integration of biology and disciplines such as materials science can be complicated by the lack of a common framework and language for synthesizing the diversity of thought and expertise of those trained in otherwise disparate disciplines. History may offer a valuable lesson as modern materials science and engineering itself resulted from the integration of traditionally disparate disciplines that were delineated by classes of materials – metals, ceramics and polymers. The integration of metallurgists, ceramists and polymer scientists into materials science was facilitated in large part by a unifying paradigm based upon processing-structure-property relationships that is now well accepted. Therefore, a common paradigm might also help unify the vast array of perspectives and challenges present in the field of biomaterials. The traditional materials engineering paradigm was modified to account for the adaptive and hierarchical nature of biological materials. Various examples of application to research and education will be considered.

11:50 AM Invited
Biomaterials Funding at NSF (DMR Biomaterials Program): *David Brant*[1]; [1]National Science Foundation

The NSF Division of Materials Research (DMR) initiated a new Biomaterials program (BMAT) in 2006 to support fundamental research on bioderived, bioinspired, biomimetic, and biocompatible materials. This talk will describe the kinds of proposals BMAT received and funded during the intitial (2007) funding cycle. Any trends or changes in the BMAT program evident at the time of the Symposium will be reported.

12:20 PM Question and Answer Period

Bulk Metallic Glasses V: Structures and Modeling I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter K. Liaw, University of Tennessee; Wenhui Jiang, University of Tennessee; Guojiang Fan, University of Tennessee; Hahn Choo, University of Tennessee; Yanfei Gao, University of Tennessee

Tuesday AM — Room: 393
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Daniel Miracle, US Air Force; Brett Conner, US Air Force, Office of Scientific Research

8:30 AM Keynote
Effect of Liquid Fragility on Glass Forming Ability of Metallic Alloys: *Oleg Senkov*[1]; [1]UES Inc

Analysis of the fragile behavior of glass-forming liquid in the temperature range between the liquidus T_l and glass transition T_g temperatures allowed us to identify unique correlation between the critical cooling rate for glass formation R_c, liquid fragility index m and reduced glass transition temperature $T_{rg}=T_g/T_l$. Correspondingly, a new GFA parameter F_1, which is proportional to $-\log(R_c)$ and is a function of T_{rg} and m was proposed. F_1 increases with an increase in T_{rg} and a decrease in m and varies from ~ 0 in the case of the extremely fragile liquid (e.g. pure metals) to ~ 0.8 in the case of the extremely strong liquid (e.g.

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SiO_2). Validity and universality of this GFA parameter F_1 was experimentally verified for a number of bulk metallic and non-metallic glasses. On the other hand, when used separately, T_{rg} and m were shown to be able to predict GFA only in special cases.

9:00 AM Invited
Anomalies in the Thermophysical Properties of Undercooled Glass-Forming Alloys: *Robert Hyers*[1]; Jan Rogers[2]; Kenneth Kelton[3]; Anup Gangopadhyay[3]; [1]University of Massachusetts; [2]NASA Marshall Space Flight Center; [3]Washington University

The surface tension, viscosity, and density of several bulk metallic glass-forming alloys have been measured using non-contact techniques in the electrostatic levitation facility (ESL) at NASA Marshall Space Flight Center. All three properties show unexpected behavior in the undercooled regime. Similar deviations were previously observed in titanium-based quasicrystal-forming alloys, but the deviations in the properties of the glass-forming alloys are much more pronounced. New results for anomalous thermophysical properties in undercooled glass-forming alloys will be presented and discussed.

9:20 AM Invited
Shear Banding in Metallic Glasses as Investigated through Atomic-Scale Simulation: *Michael Falk*[1]; [1]University of Michigan

Recent simulations of shear banding in metallic glasses reveal the structural changes that accompany plastic deformation and localization. We have simulated 2D and 3D systems in nanoindentation,[1,2] uniaxial tension[3] and compression[4] in plane strain. The degree of localization depends on the quench rate, with localization only arising in more gradually quenched samples. A systematic analysis of simulated systems in simple shear geometries[5] reveals a Boltzmann-like relationship between strain rate and structure over eight orders of magnitude in strain rate. The consequences of this scaling for constitutive models of glass plasticity will be discussed. [1]Y. Shi and MLF, App. Phys. Lett., 86, 011914 (2005). [2]Y. Shi and MLF, Acta Mat., 55, 4317 (2007). [3]Y. Shi and MLF, PRL, 95, 095502 (2005). [4]Y. Shi and MLF, PRB, 73, 214201 (2006). [5] Y. Shi, M.B. Katz, H. Li and MLF, PRL, 98, 185505 (2007).

9:40 AM Invited
Shear Band Thermal Profiles in Metallic Glasses: *Daniel Miracle*[1]; Yi Zhang[2]; Lindsay Greer[2]; Reza Yavari[3]; [1]US Air Force; [2]University of Cambridge; [3]Institut National Polytechnique de Grenoble

The characteristic temperatures, times and spatial dimensions of a moving shear band are expected to exert a profound influence on the mechanical behaviour of metallic glasses, and are a topic of active debate. Only limited experimental measurements are available, which typically suffer from spatial and temporal resolution that masks important features. The results of a thermal diffusion analysis for the calculated thermal profile behind a moving shear band will be presented and discussed, along with the importance of the boundary conditions used for the calculations. These profiles will be compared with recent experimental measurements. Relevant microstructural and sample length scales arise from coupling of characteristic dimensions of the thermal profiles. These length scales will be introduced and discussed.

10:00 AM
Correlations between Nearest Neighbor Bond Energy and Glass Forming Ability in Metallic Glass Forming Systems: *James Dahlman*[1]; Daniel Miracle[1]; [1]Air Force Research Laboratory

Experimental evidence has demonstrated the tendency for metallic glass systems to form structures with a high degree of short range order. Ordering amongst nearest neighbors indicates the presence of an underlying thermodynamic criteria, as interatomic bond strength largely governs nearest neighbor interactions. In particular, earlier empirical guidelines suggest that the heat of mixing exerts a strong influence on glass stability. However, previous work has failed to show a correlation between the heat of formation and glass forming ability. This work seeks to establish a connection between nearest neighbor bond energy and glass-forming ability through evaluation of bond energies and the heat of formation for a range of metallic glass forming systems.

10:15 AM Invited
Alloy Design for Zr TM Al (TM: Co, Ni, Cu) Bulk Glassy Alloys: *Y. Yokoyama*[1]; T. Yamasaki[2]; Peter K. Liaw[3]; A. Inoue[1]; [1]Tohoku University, Institute for Materials Research; [2]University of Hyogo, School of Engineering; [3]University of Tennessee

With the aim to locate the key for designing useful bulk glassy alloys, the mechanical and thermal properties were examined in different component and composition of ternary Zr-TM-Al (TM: Cu, Ni or Co) alloys. The intrinsic mechanical-properties change with the component and composition, which result in the glass structural or atomic-bonding change, shows high correlation coefficients with some thermal-properties change. As a result, the glass-transition temperature has large positive correlation coefficients with Young's modulus and Vickers hardness. The liquidus surface temperature has large negative correlation coefficients with the Charpy impact value, fracture strain, and volume-change ratio due to the structural relaxation. Based on the systematic analyses between the thermal and mechanical properties, we found that the relationship between the Young's modulus and volume-change ratio reveals the suitable guide for the design of glassy alloys. By using this guide, we suggested that hypoeutectic bulk-glassy alloys exhibit significant resistance against the structural-relaxation embrittlement, and noble-metal-added Zr-Cu-Al bulk-glassy alloys exhibit extremely high fatigue limits.

10:35 AM Break

10:40 AM Invited
Nucleation Issues in Metallic Glasses: *James Morris*[1]; [1]Oak Ridge National Laboratory

A key issue in glass formation is the competing process of crystal nucleation. Metallic glasses are ideal systems for examining this process, and for testing theory and simulation. These issues will be reviewed, including recent work on nucleation simulations and the comparison with classical nucleation rates. In metallic glasses, the role of chemical composition is particularly of interest. When the nucleating phase has a different composition than the amorphous matrix, classical nucleation theory is shown to be inapplicable, as diffusive dynamics must be considered. Nevertheless, calculations based on critical nuclei energetics may be useful. Even when the stable phase has the same composition as the amorphous matrix, metastable phases may form during devitrification; again, chemical flow plays an important role. 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 with DE-AC05-00OR-22725 with UT-Battelle.

11:00 AM Invited
Atomic Level Characterization of the Devitrification Path of a $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ Bulk Metallic Glass: *Michael Miller*[1]; Xun-li Wang[1]; David Larson[2]; Robert Ulfig[2]; [1]Oak Ridge National Laboratory; [2]Imago Scientific Instruments

Atomic level characterization methods are required to establish the earliest stages of devitrification of bulk metallic glasses. The application of laser-assisted field evaporation has enabled zirconium-based bulk metallic glasses to be reliably characterized in the three-dimensional atom probe. The recent introduction of energy compensation to wide field-of-view three-dimensional atom probes has significantly improved the mass resolving power and lowered the background noise. A new local electrode atom probe equipped with these improvements in conjunction with small angle X-ray scattering experiments and differential scanning calorimetry have been used to study the initial stages of the devitrification path of a $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ bulk metallic glass. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by Basic Energy Sciences, U.S. Department of Energy.

11:20 AM Invited
Molecular Dynamics Simulation of Mechanical Properties of Cu-Zr Amorphous Alloys: *Mikhail Mendelev*[1]; David Rehbein[1]; Ryan Ott[1]; Matthew Kramer[1]; Daniel Sordelet[1]; [1]Ames Laboratory

We will present a new many-body potential for simulating the structure and mechanical properties of Cu-Zr amorphous alloys. As a first test of the potential we determined the bulk and Young's moduli of the Cu-Zr alloys for comparison with values found from ultrasound measurements. While the simulation underestimates the acoustic velocities by ~10%, it does correctly predict that this quantity increases with increasing Cu concentration. Analysis of the structural changes reveals that Zr-Zr pair-correlations are affected by an applied stress more strongly than the Cu-Cu and Cu-Zr pair-correlations. Additionally, we simulated the elastic-plastic response for a bi-axially deformed Cu-Zr amorphous alloy. The obtained stress – strain relationship was found to be consistent with our experimental data. The structural changes accompanying this deformation will be discussed in the end of the talk. Work at Ames Laboratory was supported by Department of Energy, Office of Basic Energy Sciences, under Contract No. DE-AC02-07CH11358.

11:40 AM
Experimental and Computational Investigations of Structural Changes Associated with Deformation of Bulk Metallic Glasses: *Ashwini Bharathula*[1]; Weiqi Luo[1]; Katharine Flores[1]; Wolfgang Windl[1]; [1]Ohio State University

Bulk metallic glasses exhibit a number of unique capabilities, including excellent mechanical behavior combined with unusual processability in the supercooled liquid regime. In the present study, we examine the structural changes associated with the homogeneous flow of Zr58.5Cu15.6Ni12.8Al10.3Nb2.8 (nominal at%) bulk metallic glass over a range of strain rates at temperatures above and below the glass transition. Mechanical testing was performed on a high resolution electro-thermo-mechanical test frame, and structural changes were investigated via SEM, TEM, DSC, and positron annihilation spectroscopy. Experimental results for this complex alloy are discussed in light of a molecular dynamics study of deformation in a simpler binary glass subjected to simulated mechanical loading modes including tension, compression and shear. Based on the fluctuations of the electron density distribution in the structure, evolution of low atomic density regions with flow is analyzed. These results are compared and contrasted with a hard-sphere model.

11:55 AM
First-Principles Calculations of Shear Bands in Zr-Based Bulk-Metallic Glasses: *Ning Ma*[1]; Bruce Kang[1]; G. Wang[2]; Peter Liaw[2]; [1]West Virginia University; [2]University of Tennessee

Bulk-metallic-glass (BMG) materials, due to their great strength and corrosion resistance, have attracted much research interest. However, these materials are usually brittle because they lack the dislocation-flow systems and only respond to the external stress by forming and propagating shear bands. The understanding of the mechanisms and characteristics of shear banding is of crucial importance to plasticity-improvement efforts. Shear bands are areas of high local stresses where the chemical environment is vastly different from their bulk amorphous counterparts. Empirical potential methods are, therefore, inadequate to describe the complicated structures and the chemical complexity within the shear bands. Using a first-principles tight-binding package, we simulated shear-band formation in Zr-based BMGs. Structural, thermal, and mechanical properties are analyzed within and outside shear bands. In addition, we studied the electronic structures and properties of chemical bonds, which may provide a mechanistic understanding of shear-band formation and the associated mechanical properties.

12:10 PM Invited
Structure and Phase Transformation in Metallic Glasses: *Xun-li Wang*[1]; Alexandru Stoica[1]; Dong Ma[1]; Matthew Kramer[2]; James Richardson[3]; Chain Liu[1]; Michael Miller[1]; [1]Oak Ridge National Laboratory; [2]Iowa State University; [3]Argonne National Laboratory

Bulk metallic glasses were discovered more than 20 years ago. Despite extensive research efforts since then, mostly experimental, the structure of metallic glasses is still not well understood. In this talk, we describe some recent neutron and synchrotron diffraction experiments and the insights thus gained on the local and medium-range ordering in these glassy alloys. The structural evolutions at local and nano scales are studied by in-situ scattering experiments during devitrification. The experimental results are correlated with analysis by atomic probe tomography. This research was supported by Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC05-00OR22725 with UT-Battelle, LLC. The work at Ames Laboratory was supported by the U.S. Department of Energy through Iowa State University under contract No. W-7405-ENG-82. Work at Argonne National Laboratory was supported by U.S. Department of Energy under Contract No. W-31-109-Eng-38.

12:30 PM Invited
Entropic Contribution to Glass Stability Based on the Pair Distribution Function: *Donald Nicholson*[1]; [1]Oak Ridge National Laboratory

Increased entropy is frequently sited as one of the reasons that bulk metallic glasses are usually multicomponent. However, competing phases such as solid solutions would seem to have identical entropic contributions based on the regular solution model thus undermining the entropic argument for improved glass stability. The Wallace formula for the entropy in terms of the pair distribution function shows that there are terms beyond regular solution theory that can contribute to stability. Although experimental and calculated pair distributions decline in reliability as a function of distance the short range contributions to the entropy can indicate entropic contributions to stability. These contributions will be described for FeZrB, CuZrPd, and AgGa systems. Research sponsored by the 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.

Carbon Dioxide Reduction Metallurgy: Mechanisms

Sponsored by: National Materials Advisory Board, Metallurgical Society of the Canadian Institute of Mining Metallurgy and Petroleum, The Minerals, Metals and Materials Society, American Iron and Steel Institute, TMS Light Metals Division, TMS Extraction and Processing Division, TMS: Reactive Metals Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Neale Neelameggham, US Magnesium LLC; Masao Suzuki, Al Tech Associates; Ramana Reddy, University of Alabama

Tuesday AM Room: 294
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Ray Peterson, Aleris International Inc; Mahesh Jha, US Department of Energy

8:30 AM Introductory Comments by Neale Neelameggham, Organizer

8:35 AM Keynote
From the Manhattan Project to Global Warming: The Science and Technology of CO_2 Mitigation: *Meyer Steinberg*[1]; [1]Brookhaven National Laboratory (Retired)

Some early experiences of a Cooper Union Chemical Engineer on the Manhattan project and his latest studies on CO_2 Greenhouse Gas Mitigation Technologies will be presented. Mitigating the Global Greenhouse effect while maintaining a fossil fuel economy, requires improving the efficiency of conversion and utilization of fossil fuels, use of high hydrogen content fossil fuels, decarbonization of fossil fuels and sequestration of carbon and CO_2 applied to all energy consuming sectors of the economy including electric power generation, materials production, transportation and industrial domestic heating. Decarbonization means removal of carbon as C or CO_2 either before or after fossil fuel utilization and sequestration means long term disposal of C or CO_2. The review will cover the principles of removal and recovery from power plant stacks, the ceanographic and geological disposal of CO_2 and the conversion of CO_2 to gaseous and liquid transportation fuels. The global economic aspects of CO_2 mitigation technologies will be touched upon.

9:20 AM
Chemical Utilization of Sequestered Carbon Dioxide as a Booster of Hydrogen Economy: *Michael Moats*[1]; Jan Miller[1]; Wlodzimierz Zmierczak[1]; [1]University of Utah

For many, hydrogen in a pure hydrogen economy would be like natural gas in today's energy economy. Unfortunately, hydrogen's physical properties are unsuited to the energy market's requirements in terms of packaging, storage, transfer, and delivery. In this paper, a hybrid energy economy that packages hydrogen chemically on carbon atoms from various sources including recycled carbon dioxide is introduced and discussed. For this new hybrid energy economy to become a sustainable reality, the ability to recycle carbon dioxide and attach hydrogen to create a usable energy product, such dimethyl ether (DME), is needed. DME is recognized as a potential next generation, "beyond-petroleum",

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environmentally benign commodity for energy storage and distribution. To maximize the sustainability of the proposed hybrid energy economy, "green" hydrogen will be utilized. The "green" hydrogen would be created by the electrolysis of water powered by renewable energy sources, such as solar, wind or geothermal heat.

9:40 AM
Novel, Synergistic Oxygen Supply Technology for Metals Processing: Phillip Armstrong; *John Gordon*[1]; Charles Lewinsohn[1]; [1]Ceramatec Inc.

This paper describes the opportunity offered by employing systems based on mixed conductive ceramics, in particular, ceramics conductive to oxygen ions and electrons, in reducing CO_2 emissions from metallurgical production. Membranes based on these mixed conducting properties enable commercial viability of several attractive metallurgical processes. One of these processes utilises modular, ceramic-membrane structures for the generation of pure oxygen, in tonnage quantity. The membrane-based systems offer significant capital savings and operating efficiencies in various metallurgical processes.

10:00 AM Break

10:15 AM
CO_2 Reduction by Dry Methane Reforming over Hexaluminates: A Promising Technology for Decreasing Global Warming in a Cost Effective Manner: *Maria Salazar-Villalpando*[1]; Todd Gardner[1]; [1]National Energy Technology Laboratory

Efficient utilization of CO_2 can help to decrease global warming. Methane reforming using CO_2 has been of interest for many years because CO_2 provides a source of clean oxygen. Coal power or metallurgical plants have wasted heat streams that can be utilized in CO_2 reduction. The product of this reaction is syngas, which could generate electrical power in a SOFC or used in the production of synthetic fuels. Hexaaluminate catalysts prepared at NETL may represent a product that can be utilized for the conversion of CO_2 to syngas. A series of BaNiyAl12-yO 19-z catalysts were prepared by co-precipitation followed by calcination at 1400°C. Reactions were carried out to determine catalyst performance and catalyst characterization was conducted to determine surface area, pore size, catalyst phases and structures. Moreover, catalyst characterization analysis of three samples (y = 0.2, 0.6, and 1.0 in BaNiyAl12-yO19-z) was performed by EXAFS and temperature programmed reduction.

10:35 AM
A Proposal for Global Reduction Method of Global Atmospheric Co_2: Isolation of Surplus Co_2 from the Biosphere and Fix it to form an Insoluble Mineral onto a Sea Bottom: *Katsuyoshi Tatenuma*[1]; [1]Kaken Inc.

In order to mitigate the effect of global warming by preventing the accumulation of atmospheric CO_2, a permanent fixation method of surplus CO_2 as an insoluble mineral onto a sea bottom is proposed. As a new and practical reduction method of atmospheric CO_2, a concrete concept of this method is that an insoluble carbonate mineral ($CaCO_3$) formed by direct electrolysis of the seawater, Ca^{2+}+2HCO_3^-+OH-<>$CaCO_3$(insoluble)+CO_3^{2-}(soluble)+H^++H_2O (not coral reef reaction), is directly disposed by itself onto a sea bottom. By this treatment, a concentration of carbonate is reduced at the section of superficial seawater, and the absorption capacity of atmospheric CO_2 is therefore increased as a result of chemical equilibrium between the superficial ocean and atmosphere. The proposed method without any additives, generation of secondary wastes and CO_2 itself, and disturbance of the environmental balance as a green-orientated method has an ability to resolve fundamentally the problem of global warming.

10:55 AM
CO_2 Capture and Sequestration – Implications for the Metals Industry: *Mark Berkley*[1]; Jim Sarvinis[1]; Jason Berzansky[1]; David Clarry[1]; [1]Hatch

Technologies developed to sequester CO_2 or use CO_2 for enhanced fossil fuel recovery are currently in operation. Taxation regimes and CO_2 credit trading are becoming drivers for a number of projects. Some mining companies have also identified an opportunity to sequester CO_2 in their by-products; recent work by Alcoa is a prime example. In addition to process plant emissions, mining companies are becoming increasingly aware of their overall carbon footprint, including CO_2 generated during the production of power they use. New power plant designs, in various stages of development, include CO_2 separation and sequestration techniques. This paper summarizes the state-of-the-art for these technologies, including integrated gasification combined cycle (IGCC power plants. A concept to use the gasification products as reductant in metallurgical processes is also described. The relevance of technology from power plant designs with post-combustion capture to the processing of off-gas generated in metallurgical facilities is also reviewed.

11:15 AM Panel Discussion

Cast Shop Technology: Casthouse Operation

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Hussain AlAli, GM Casthouse and Engineering Services, Aluminium Bahrain Company (ALBA); David DeYoung, Alcoa Inc

Tuesday AM — Room: 295
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Ghyslain Dube, Centre de Recherche et de Développement Arvida; Barbara Rinderer, Rio Tinto Aluminium Technology

8:30 AM Keynote
ICON: Pre-Treatment of Pot-Room Metal: *Erik Aspen*[1]; Bjarne Heggset[1]; Jo Vaagland[1]; [1]Heggset Engineering

A new process for sodium removal has been developed. The technology is based on direct injection of AlF3 into the melt while tapping metal from potcells. The ICON technology makes sure AlF3 is injected directly into the melt and ensures excellent mixing of AlF3 with the melt causing good conditions for the chemical reaction. Preliminary results from testing indicate total sodium removal down to a level of 5-10 ppm(90%), before casting. The technology can be installed at several locations; integrated in the tappingvehicle and integrated in the cranetapping system. ICON is flexible, easy to install and can be fitted to any tapping system, no limitations on crucible size. Furthermore ICON can be characterized by low installation and operation cost, very low maintenance, no rotor system, no extra crucible handling systems, minimal metal temperature drop in the crucible, dust extraction to existing plant systems, fully automated and operator friendly.

9:00 AM
Gas Fluxing of Molten Aluminum: An Overview: Geoffrey Sigworth[1]; *Eddie Williams*[1]; Corleen Chesonis[1]; [1]Alcoa Inc

The aluminum industry is under continual pressure to improve metal quality, while at the same time reduce costs. Although a reasonably mature technology, there has been a continual evolution in degassing equipment over the years. A detailed review and theoretical analysis is given of the chemical and kinetic factors which control the metal quality after gas fluxing, and the evolution of degassing technology in Alcoa is summarized. Particular emphasis is placed on hydrogen removal, minimization of chlorine use, reduced operating costs and minimization of environmental emissions. Considerations related to inclusion removal are also discussed briefly.

9:20 AM
Monolithic Refractory Furnace Linings Designed for Rapid Commisioning: Andrew Brewster[1]; *Zena Carden*[1]; [1]Vesuvius

Monolithics are widely used for lining aluminium melting and holding furnaces and have many advantages in performance and security. One major disadvantage is the time taken to fully cure and dry out the structure. The use of high cost pre-cast and dried pieces can reduce this down-time but unless all the furnace is built in pre-cast pieces the need to cure and dry the in-situ installed monolithics prevails. New materials have been developed with a different bonding system such that the lining does not require any curing time and all the water can be removed quickly at low temperatures. Savings of several days can result. These new products are very versatile with the option to install the same material by casting, pumping or shotcreting. They have excellent aluminium penetration resistance and are well proven in service. The paper describes the product development principles and gives examples of application.

9:40 AM
Design Considerations for Holding and Casting Furnaces: *Jan Migchielsen*[1]; Tom Schmidt[1]; Hans-Walter Gräb[1]; [1]Thermcon Ovens BV

Holding and casting furnaces are common tools in aluminium cast houses. This paper addresses the aspects to be considered for selecting the most appropriate furnace design. The differences in design are discussed between holding/casting furnaces, melting furnaces and combined melting-holding furnaces. The paper addresses the main consideration in designing a holding furnace with respect to mechanical, thermal-process and control aspects. The last part of the paper considers the choice to be made for using a combined furnace versus a separate casting furnace.

10:00 AM
Producing Gloss Extruded Shape Products for Special Applications-Experience from Practice: *Jana Dressler*[1]; Dietmar Bramhoff[1]; Hubert Koch[1]; C. Deiters[1]; [1]Trimet Aluminium AG

The TRIMET/GERHARDI gloss alloy is a high puritiy aluminium extrusion alloy. It is characterized by an extremely homogenous micro structure without internal defects or inclusions. This material composition and the special processing know-how allow a considerably higher productivity. Due to optimal polishing and brightness properties as well as an excellent bending quality the gloss alloy is especially appropriate for the production of surface sensitive components like trim parts, roof racks, decorative fittings, door handles, cover plates for automotive application and power train or design elements for high end audio equipment or the lighting industry. This gloss alloy allows any surface treatment from anodizing to coating. The paper covers the production of this special extrusion alloys including quality requirements, qualifying of the alloying material, and process parameters, as well as latest results from laboratory examinations covering podfa analysis and inclusion identification. Finally examples of applications are shown and discussed.

10:20 AM Break

10:30 AM Keynote
Almex Minicast™ Casthouse Philosophy Helps Southern California Extrusion Company Meet Their Billet Demand Requirement and Optimize Turnaround Scrap Economics: *Shaun Hamer*[1]; Ravi Tilak[1]; [1]Almex USA Inc

Universal Molding Company Inc. started up a new casthouse in Southern California in the third quarter of 2007 designed and empowered by Almex's Minicast™ series of equipment and technology. Minicast™ has been developed by Almex specifically for extrusion companies to cast billet from their in-house scrap. Minicast™ is a single source supply of the entire casting line from melting to homogenization furnaces, configured specifically for this application. UMC, a well respected, privately owned extrusion company, in operation since 1944, recognized the benefits of the system and, on an available 1 acre (0.288 hectare) site, decided to install the Minicast™ system. This paper describes the installation of the 28,000 ton per year facility, and touches on some of the specific design, technology and environmental technologies in use.

11:00 AM
Nonmetallic Inclusions in the Secondary Aluminium Industry for the Production of Aerospace Alloys: *Bernd Prillhofer*[1]; Helmut Antrekowitsch[1]; Holm Böttcher[2]; Phil Enright[3]; [1]University of Leoben; [2]AMAG Rolling GmbH; [3]N-Tec Limited

Due to the enormous growth of the secondary aluminium industry and the higher content of impurities in the input materials, the development of methods for inclusion removal has become highly important. To produce high quality aerospace alloys with very low inclusion contents, casthouses must analyse and optimise their production processes from the beginning to the end. Nowadays there are several methods available for process evaluation to determine the inclusion level from one step to another. Furthermore the most important parameters for control of the final inclusion level have to be investigated. This paper characterises the change of the inclusion content for the alloy AA 7075 during standard casthouse processing. Additionally, possibilities for the improvement of the melt quality are discussed.

11:20 AM
Effect of Temperature and Charge Borings in the Fluidity of an Aluminum-Silicon Alloy: *Eulogio Velasco*[1]; Guillermo Ruiz[2]; [1]Nemak; [2]Facultad de Ingeniería Mecánica y Eléctrica-Universidad Autónoma de Nuevo León

In the processing of secondary alloys, mixed of different aluminum scrap grades are typically used in the melting furnace charges. Turnings and borings are materials with high availability to in the metal market with competitive price compare with others aluminum scrap grades, the drawbacks with turnings are related with melting practices and the cost of devices and equipment to obtain a high yields and avoid possible contamination by fine aluminum oxide particles. Molten Aluminum-Silicon alloy was prepared in a crucible gas furnace and several percentages of chips were added during melting, after each addition, fluidity test at different temperatures and PreFilÔ samples were taken in order to evaluate the effect of temperature and cleanliness of metal in the fluidity of the alloy.

11:40 AM
Production of Aluminium-Silicon-Magnesium Wrought Alloy Rod with Application in the Manufacture of Extra-High Conductivity AAAC for Overhead Electrical Transmission Lines: *Mohamed Rafea*[1]; [1]Midal Cables

Aluminium-Silicon-Magnesium (Al-Si-Mg)wrought alloy rods have been used extensively in the manufacture of wires stranded into conductors used in high voltage transmission lines. Midal Cables has modified the productions process of this type of rod for use in the manufacture extra-high conductivity AAAC for applications as overhead transmission line conductors. These have lowe DC Resistance thus in turn have higher current carrying capacity. With modifications in the chemical composition, casting and hot rolling parameters Midal has developed a rod which respond differently to heat treatment after wire drawing resultig to a product that has a higher electrical conductivity than conventionally manufactured Al-Si-Mg alloy wires for the same application. This paper discusses the various production parameters used in the manufacture of Al-Si-Mg alloy rods and establish the mechanisms that effect the extra-high conductivity property after wire drawing and heat ageing.

12:00 PM Panel Discussion

Characterization of Minerals, Metals, and Materials: Characterization of Microstructure and Properties of Materials I

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee
Program Organizers: Jian Li, Natural Resources Canada; Toru Okabe, University of Tokyo; Ann Hagni, Intellection Corporation

Tuesday AM Room: 284
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF; Jeongguk Kim, Korea Railroad Research Institute

8:30 AM
Characterization of Aging in U-Nb Martensites: *Robert Hackenberg*[1]; Amy Clarke[1]; Robert Field[1]; Heather Volz[1]; Don Brown[1]; Dave Teter[1]; Dan Thoma[1]; Michael Miller[2]; Kaye Russell[2]; [1]Los Alamos National Laboratory; [2]Oak Ridge National Laboratory

Artificial aging at temperatures <300C gives rise to strengthening accompanied by undesirable ductility loss in U-5.6wt.%Nb and U-7.7wt.%Nb. Two hypotheses have been suggested to account for this phenomenon: 1) diffusional decomposition of Nb-supersaturated thermoelastic martensite into Nb-rich and Nb-lean clusters or phases, and 2) Nb or impurity segregation to twin boundaries. X-ray and neutron diffraction and transmission electron microscopy (TEM) could not unambiguously discern microstructural changes to support either hypothesis. Thus, atom probe tomography was performed to show Nb and impurity atom distribution as a function of aging time and temperature at very fine length scales. Neither phase separation nor segregation was observed at T=200C, forcing a reevaluation of the accepted aging mechanisms in U-Nb alloys. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by Basic Energy Sciences, U.S. Department of Energy.

8:50 AM
Mechanical Characterization of Ramie Fiber Reinforced Polyester Composites: *Sergio Monteiro*[1]; Luiz Fernando Santos[1]; Frederico Margem[1]; [1]State University of the Northern Rio de Janeiro - UENF

Lignocellulosic natural fibers have been used as reinforcement for polymeric composites as low cost type of biodegradable and renewable materials. For this purpose, sisal, jute, hemp, coir and flax are successful examples of reinforcement fibers in composites, which are currently applied as components in building construction and automobiles. Among these fibers, ramie (Boehmeria nivea) has received less attention. The objective of this work was to evaluate the mechanical behavior of ramie reinforced polyester matrix composites. Rectangular specimens with up to 30wt.% aligned ramie fibers were embedded in polyester resin and the cured at room temperature. Bend tests were performed in an Instron machine. The results showed that a sensible improvement in strength occurs with the introduction of ramie fibers into the polyester matrix. SEM analysis revealed that the surface cellular structure of the ramie fiber contribute to a better adherence to the matrix an to a higher fiber/matrix interfacial resistance.

9:10 AM
Surface Characterization in Aluminium 6063 by Mecano-Chemical Treatment: *Isaías Hilerio*[1]; Miguel Barrón[1]; [1]Universidad Autonoma Metropolitana Azcapotzalco

The principal objective of finishing surfaces is protected and/or decorates them. This can be obtained by adding or removing material of the work surface. In this work it is important to develop the improvement of surfaces by removal of material. Between these are used Barreling or Polishing. The pieces are at first sandblasted and arranged in the barrel with a natural or synthetically abrasive and an additive (water or a tensoactif). When the abrasives passes against the surfaces of the pieces to treat, produces an action of rubbing and impact that clean the surface of the pieces. In this work are studied evolution surfaces in aluminum 6063 by this method. The process was carried out in 5 samples with the following dimensions: 35 x 100 x 8 mm. It has been done micro hardness study, MEB and Ray X diffraction. It has been founded higher hardness values in samples treated.

9:30 AM
On the Determination of Compositional Gradients at Interfaces in Alloys and Multilayered Materials: *Arda Genc*[1]; Soumya Nag[1]; Rajarshi Banerjee[2]; Peter Collins[1]; Hamish Fraser[1]; [1]Ohio State University; [2]University of North Texas

It is important to be able to determine quantitatively the compositional gradients about interfaces in various types of materials. Such measurements have been made in Ti alloys and Nb/Cu multilayered materials. Use has been made of both the tomographical atom probe (Imago's LEAP) and analytical electron microscopy (FEI Titan 80/300 S/TEM with monochromator (0.15eV) and spherical aberration corrector for the probe and both an electron energy loss spectrometer (0.15eV) and energy dispersive x-ray spectroscopy. Emphasis has been placed on optimizing and assessing the quality and reliability of data from both instruments, and comparing the results of measurements of the same gradients in both machines. The results from the study of the multilayers are very promising, where similar results from both techniques have been obtained. However, the Ti alloys studied have been quite complex, e.g., Ti-5-5-5-3, and work has been focused on assessing and optimizing the quality of the data obtained.

9:50 AM Break

10:10 AM
Influence of Weave Damage on the Strength of Composites Reinforced with Jute Fabric: Amanda Lima[1]; *Sergio Monteiro*[1]; Luis Augusto Terrones[1]; [1]State University of the Northern Rio de Janeiro - UENF

Jute fabric detached from sackcloth is being considered as reinforcement of polymeric matrix composites. Discarded sackcloth usually has weave damages, which affect the fabric mechanical resistance and could impair its reinforcement effect in composites. The objective of this work was to investigate the effect of pre-existing weave damage in the fabric, cut from jute sackcloth and used as reinforcement, on the strength of polyethylene composites. Fabrics were obtained from either new or already damaged sackcloth. Composites were fabricated by 160°C compression molding interlayer fabric pieces together with either new or recycled polyethylene pellets. The composite strength was evaluated by bend test and the fracture analyzed by SEM. The consideration of Godfrey and Rossetos model has shown that the discarded jute fabric has a significant lower tolerance to damage. This results in a comparative decrease in the strength of the composites reinforced with fabrics from discarded jute sackcloth and recycled polyethylene.

10:30 AM
The Effect of Hydrostatic-Testing on Pipeline SCC Propagation: *Jian Li*[1]; [1]Natural Resources Canada

Stress corrosion cracking (SCC) is an important failure mechanism for oil and gas pipelines. In the past, hydrostatic testing has been frequently used to assess and mitigate stress corrosion cracking. It is commonly agreed that an effective hydrostatic test not only eliminates critical crack-like flaws, but also blunts the sub-critical crack tip thereby suppressing further SCC propagation. However, little study has been done on the plastic deformation that results from the high stress intensity at the crack tip due to hydrostatic testing pressure and its possible role in subsequent SCC propagation. In this study, microstructural details were examined of an API 5L X52 SCC-containing pipe removed from field service. Plastic deformation generated by the hydrostatic testing pressure was revealed by using high-resolution focused ion beam (FIB) microscope. The existence of the microscopic plastic zones around some crack tips suggests that caution should be taken when setting up pipeline hydrostatic tests.

10:50 AM
Damage Evolution and Fracture Behavior of Ceramic Matrix Composites during Monotonic Loadings: *Jeongguk Kim*[1]; [1]Korea Railroad Research Institute

Ceramic matrix composites (CMCs) have been identified as potential candidates for high-temperature structural applications, such as aircraft structures, fusion reactors, and heat management systems due to their high-temperature strength, lightweight, and excellent corrosion and wear resistance. In this paper, damage evolution and failure mechanisms of CMCs during tensile loadings were investigated using nondestructive evaluation (NDE) techniques and microstructural characterization. The CMCs used for this investigation is continuous fiber reinforced ceramics matrix composites. NDE techniques were employed to characterize defect information before and during mechanical testing. Microstructural characterizations using scanning electron microscopy (SEM) were performed to investigate fracture modes and failure mechanisms of CMC samples. In this investigation, the NDE technique and SEM characterization were employed to facilitate a better understanding of damage evolution and progress of Nicalon/CAS composites during monotonic and cyclic loadings.

11:10 AM
Dynamic Thermo-Mechanical Characterization of Coir Fiber Reinforced Polymeric Composites: *Sergio Monteiro*[1]; Rubén Sanchez[1]; Helvio Santafé[1]; Lucas da Costa[1]; [1]State University of the Northern Rio de Janeiro - UENF

The fibers extracted from the coconut fruit, also known as coir fibers are being applied as reinforcement in polymeric composites. In the present work, a study on the temperature variation of the dynamic-mechanical parameter of polymeric matrix composites incorporated with continuous coir fibers was carried out. Thermoset polymers were mixed with different amounts of coir fibers and then press-molded cured for 24 hours at room temperature. DMA tests permitted to obtain the storage modulus and the tangent delta for each studied condition. The results revealed that the incorporation of coir fiber decreases the viscoelastic stiffness of the polymeric matrix. Moreover, modifications in the glass transition temperature and in the alpha relaxation peak could be associated with the incorporation of coir the fiber into the matrix. The interaction of the coir fiber with the polymeric matrix, which reduces the molecular mobility, is the proposed mechanism for these experimental results.

Complex Oxide Materials - Synthesis, Properties and Applications: Functionally Cross-Coupled Heterostructures

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division
Program Organizers: Ho Nyung Lee, Oak Ridge National Laboratory; Zhiming Wang, University of Arkansas

Tuesday AM — Room: 277
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Satoshi Okamoto, Oak Ridge National Laboratory; John Freeland, Argonne National Laboratory

8:30 AM Invited
Correlated Oxide Heterostructures: *Ramamoorthy Ramesh*[1]; [1]University of California
Complex perovskite oxides exhibit a rich spectrum of functional responses, including magnetism, ferroelectricity, highly correlated electron behavior, superconductivity, etc. The basic materials physics of such materials provide the ideal playground for interdisciplinary scientific exploration. At Berkeley, we are exploring the science of such materials (for example, colossal magnetoresistance, ferroelectricity, etc) in thin film form by creating epitaxial heterostructures and nanostructures. Among the large number of materials systems, there exists a small set of materials which exhibit multiple order parameters; these are known as multiferroics. Using our work in the field of ferroelectric and ferromagnetic oxides as the background, we are now exploring such materials, as epitaxial thin films as well as nanostructures. Specifically, we are studying the role of thin film growth, heteroepitaxy and processing on the basic properties as well as magnitude of the coupling between the order parameters. A new development has been the discovery of the formation of spontaneously assembled nanostructures consisting of a ferromagnetic phase embedded in a ferroelectric matrix that exhibit very strong coupling between the two order parameters. This involves 3-dimensional heteroepitaxy between the substrate, the matrix perovskite phase and spinel phase that is embedded as single crystalline pillars in this matrix. In this talk I will describe to you some aspects of such materials as well as the scientific and technological excitement in this field.

9:00 AM Invited
In Pursuit of Strongly Coupled Multiferroic Oxides: *Craig Fennie*[1]; [1]Argonne National Laboratory
To realize novel electrically controlled magnetic devices based on multiferroic oxides - materials that are simultaneously ferroelectric and magnetic - an external electric field is required to strongly couple to a switchable magnetization. Given the known challenges simply to synthesize and to measure the properties of new strongly coupled multiferroics, a theory-driven, first-principles search for new candidates will greatly advanced the discovery of such structurally and chemically complex materials. In this talk we discuss our recent identification of a new class of multiferroic oxides. Combining density-functional calculations with group-theoretic arguments we elucidate the interplay between the electrical polarization and the spins to show how the direction of the magnetization can be switched between symmetry equivalent states with an applied electric field.

9:30 AM
Non-Linear Modulation of Transport Properties in Magnetic-Ferroelectic Bilayers: *Zsolt Marton*[1]; Ho Nyung Lee[2]; Takeshi Egami[3]; [1]University of Pennsylvania, Department of Materials Science end Engineering; [2]Oak Ridge National Laboratory, Materials Science and Technology Division; [3]University of Tennessee, Department of Materials Science end Engineering/Physics and Astronomy
In doped manganites, strong correlation between structural, electronic and magnetic properties leads to fascinating phenomena such as metal-insulator transition and colossal magnetoresistance. We utilize the ferroelectric polarization to modulate the charge carrier density in high quality $La_{1-x}Sr_xMnO_3$ (LSMO) (x = 0, 1/8, 1/5, 1/3, and 1/2) epitaxial films by using highly-polar ferroelectric $PbZr_{0.2}Ti_{0.8}O_3$ (PZT) films with $P_r \approx 80\ \mu C/cm^2$. All epitaxial LSMO/PZT heterostructures on (001) $SrTiO_3$ with epitaxial $SrRuO_3$ electrodes were grown by pulsed laser deposition. We will present a systematic study on the screening and transport/magnetic properties of the doped manganites under the influence of ferroelectric polarization of PZT. The role of thickness, strain, and composition of the manganite film, besides the effect of external magnetic field, on the magnetoelectric properties will be also discussed. Research sponsored by the LDRD Program of ORNL, managed by UT-Battelle, LLC for the U.S. Department of Energy and by NSF DMR-0404781.

9:50 AM Invited
Exploiting Oxide Interfaces to Generate New Functionalities: *Nicola Spaldin*[1]; [1]University of California Santa Barbara, Materials Department
Recent developments in thin film growth techniques have enabled the production of oxide heterostructures with interfaces of unprecedented quality. A range of unusual behaviors have been reported at such interfaces, including electrical conductivity between oxides that are insulating in their bulk form, and ferromagnetism between non-magnetic bulk oxides. Here we describe first-principles computational explorations of functionalities that exploit the fact that interfaces break space-inversion symmetry. We demonstrate computationally a novel linear magnetoelectric effect at the interface between a non-magnetic, non-polar dielectric and a metal with spin-polarized carriers at the Fermi level. We show that an electric field induces a linear change in magnetization in the metal at the interface, with the magnetic response mediated by the accumulation of spin-polarized charge at the interface. As a result of the magnetoelectric response, magnetism and dielectric polarization coexist in the interfacial region, suggesting a route to a new type of interfacial multiferroic.

10:20 AM Break

10:50 AM Invited
Magnetism and Electronic Structure at the Interfaces of Complex Magnetic Oxides: *Jak Tchakhalian*[1]; [1]University of Arkansas
Atomically controlled interfaces between two materials can give rise to novel physical phenomena and functionalities not exhibited by either of the constituent materials alone. Modern synthesis methods have yielded high-quality oxide heterostructures with competing order parameters. Magnetic correlations at the interface are expected to be important in determining the macroscopic properties of such nanosystems, but quantitative determination of the interfacial magnetic structure in oxides has thus far been very limited. Here we examine superlattices composed of the half-metallic ferromagnet La2/3Ca1/3MnO3 and the high-temperature superconductor YBa2Cu3O7 by polarized soft x-rays and by diffuse neutron reflectometry. The resulting data yield microscopic insight into the interplay of spin and orbital degrees of freedom at the interface. The data also reveal an extensive rearrangement of the magnetic domain structure at the superconducting transition temperature. The combination of techniques establishes an incisive probe of the interplay between competing order parameters in complex oxide heterostructures.

11:20 AM Invited
Piezoelectric Strain Control of Thin Film Magnetism: *Kathrin Dörr*[1]; Diana Rata[1]; Andreas Herklotz[1]; Orkidia Bilani[1]; [1]IFW Dresden
Multiferroic (magnetic and ferroelectric) nanofabricated composites principally offer the “magnetoelectric” functionality of controlling magnetization by electric voltage. In recent years, intense research has been devoted towards the search for thin film (nano)structures being capable of efficient coupling of the components. In most composites, magnetoelectric coupling originates from joined elastic strain in the components. Here, the approach of epitaxial growth of strain-sensitive magnetic oxide films on a single-crystalline piezoelectric substrate (PMN-PT(001), with 28% PbTiO3 and 72% PbMg1/3Nb2/3O3) is discussed. Application of an electric voltage to the rhombohedral pseudocubic substrate allows one to reversibly cycle the biaxial in-plane strain of substrate and film. In this way, the impact of (uniform) biaxial strain on the magnetization of epitaxially grown films of ferromagnetic manganites (La,A)MnO3 and cobaltates (La,Sr)CoO3 has been quantitatively studied. The immense strain sensitivity of manganites is reflected in the observation of large magnetoelectric coupling at ambient temperature (PRB 75, 054408).

11:50 AM
Theoretical Modeling of Bifunctional Multilayer Systems: *Donald Ellis*[1]; Norman Tubman[1]; Daniel Wells[1]; [1]Northwestern University

Crystalline multilayer systems with structure ABABA... offer the possibility of combining functional properties of two distinctly different materials, and of exploiting the interfaces to couple functionality of one component to the other. The multilayer environment permits the amplification of interface properties as would be important for device applications. The roles of interfacial strain and orientation, chemical variability at the interface, and film thickness are explored, taking as a starting point the classic $BaTiO_3||Fe_3O_4$ ferroelectric||ferrimagnetic interactions. First principles band structure calculations are used to determine relaxed interface structures and residual stresses, as well as the underlying electronic distributions. Embedded cluster methods are then used to extract local chemical bonding characteristics and hyperfine properties, including contact spin densities, anisotropic spin distributions, and electric dipolar and quadrupolar fields. Mixed composition interfaces involving cation substitution; e.g., (Fe,Co) and (Ti,Pd), are examined as a means to enhance coupling between functional slabs.

Computational Thermodynamics and Kinetics: Phase Field Crystal

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, ASM Materials Science Critical Technology Sector, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Phase Transformations Committee
Program Organizers: Yunzhi Wang, Ohio State University; Long-Qing Chen, Pennsylvania State University; Jeffrey Hoyt, McMaster University; Yu Wang, Virginia Tech

Tuesday AM — Room: 288
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Jeff Hoyt, McMaster University; Ken Elder, Oakland University

8:30 AM Invited
Phase Field Modeling from Atomic Mesoscopic Scales: *Ken Elder*[1]; [1]Oakland University

Understanding the formation and characteristics of non-equilibrium structures has been greatly enhanced by the use of continuum or 'phase' field models. Such models typically describe phenomena on mesoscopic length and time scales. More recently an extension to this approach, known as phase field crystal modeling, has been introduced to describe processes on atomic length and diffusive time scales. This approach has the advantage of incorporating elastic and plastic deformations in a natural and simple fashion. In this talk I would like to discuss the connection between standard microscopic descriptions, phase field crystal and standard phase field models. Emphasis will be placed on the physical significance and microscopic origin of the parameters and fields that enter phase field models.

8:55 AM Invited
Phase-Field-Crystal Modeling of Interface Coalescence: Jesper Mellenthin[1]; Mathis Plapp[2]; *Alain Karma*[3]; [1]Ecole Polytechnique and Northeastern University; [2]Ecole Polytechnique; [3]Northeastern University

Crystal cohesion during the late stages of solidification, and its failure often described as "hot tearing", depends critically on the coalescence of crystal grains impinging upon each other. In metallic systems, crystal density waves decay slowly into the liquid over several atomic layers. Therefore crystal-melt interfaces interact when the envelopes of density waves from nearby grains start to overlap. This interaction is studied as a function of temperature and misorientation in a pure material using a phase-field crystal approach. This method provides a natural description of crystal density waves, is computationally efficient, and allows for a precise determination of the excess free energy due to the interaction of the interfaces. The nature of this interaction, whether attractive or repulsive, is found to depend both on misorientation and separation between interfaces. Results shed light on the physics of coalescence and are compared to the predictions of sharp-interface theories and multi-phase-field models.

9:20 AM Invited
Multi-Scale Modeling of Solidification: Connecting Density Functional Theory to Phase Field Crystals and Phase Field Models: *Nikolas Provatas*[1]; Sami Majaniemi[1]; [1]McMaster University

The connection of phase field crystal modeling in pure materials and alloys to density functional theory (DFT) is reviewed. We discuss new approaches for using higher order correlation functions, critical to modeling complex crystal structures in phase transformations. We then move up in length scale and discuss new course graining strategies for projecting out anisotropic phase field models, the form and coefficients of which are more fundamentally connected to first principles than their phenomenological counterparts used traditionally. Outstanding challenges for modeling interface kinetics in two-sided phase field crystal (or DFT) models –akin to those that plagued traditional phase field models— are discussed.

9:45 AM Invited
Incorporating Mechanics into Phase Field Models: *James Warren*[1]; Daniel Wheeler[1]; William Boettinger[1]; [1]National Institute of Standards and Technology

The addition of hydrodynamics to diffuse interface models of phase transformations has now been well studied in some relatively simple systems. However, properly coupling such flows to a description of an elastic solid remains an area where the models need substantial improvements. In recent years we have been interested in the problem of reactive wetting, where a droplet disolves the substrate upon which it spreads. Describing this system mathematically, for realistic materials (such as Sn-Bi) in a manner that admits numerical solution in reasonable time scales is the focus of this work. We will also discuss related approaches, including phase field crystal models, of the same phenomenon.

10:10 AM
Molecular Dynamics Validation of Phase Field Modeling: *James Belak*[1]; Patrice Turchi[1]; Milo Dorr[1]; Bryan Reed[1]; David Richards[1]; Jean-luc Fattebert[1]; Michael Wickett[1]; Fred Streitz[1]; [1]Lawrence Livermore National Laboratory

Recently, phase field models have been introduced to model the crystallography during polycrystal microstructure evolution.[1,2] Here, we assess these models with molecular dynamics and phase field simulations that overlap in time and space. Large parallel computers have enabled MD simulations of sufficient scale to observe the formation of realistic microstructure.[3] We compare the two methods by calculating the phase field order parameter (quaternion) from the atomic coordinates and drive the evolution with the MD. Results will be presented for the solidification of tantalum. [1]R. Kobayashi and J.A. Warren, Physica A, 356, 127-132 (2005). [2]T. Pusztai, G. Bortel and L. Granasy, Europhys. Lett, 71, 131-137 (2005). [3]F. H. Streitz, J. N. Glosli, and M. V. Patel, Phys. Rev. Lett. 96, 225701 (2006). This work was performed under the auspices of the U.S. Department of Energy by University of California, LLNL under Contract W-7405-Eng-48.

10:25 AM Break

10:45 AM Invited
The Phase Field Crystal Model: Atoms, Defects and Nonlinear Elasticity: Pak Yuen Chan[1]; *Nigel Goldenfeld*[1]; Zhi Huang[1]; Badrinarayan Athreya[1]; Jon Dantzig[1]; [1]University of Illinois at Urbana-Champaign

The phase field crystal (PFC) model describes the local atomic density in a material as the solution of a sixth order partial differential equation in space and time. The periodic solutions represent crystalline materials, but as with real materials, approach to the true equilibrium is kinetically-limited, resulting in the full plethora of microstructural phenomena found in real materials. Here we show how our multiscale representation of the PFC leads naturally to a way to represent the full nonlinear elasticity of materials. We use the PFC to simulate the dynamics of dislocations, observing a variety of avalanche, scaling and intermittent acoustic phenomena that strongly resemble observations of plastic flow in pure materials. Thus, the PFC model provides a unified description of materials from the atomic scale up to continuum mechanics.

11:10 AM Invited
Phase-Field Crystal Model: Deformation and Ferroelectric Phenomena at the Nanoscale: *Mikko Haataja*[1]; [1]Princeton University

Traditionally, the spatio-temporal evolution of morphologies during phase transformations has been modeled through numerically solving either a set of physically-based sharp-interface or diffuse-interface ("phase-field") models. While such approaches have proven to be very useful in elucidating the physics behind the evolving morphologies in, e.g., phase transformations and thin film growth, incorporating plastic effects and other atomistic features in these models becomes quite cumbersome. The so-called "phase-field crystal" method introduces a continuous atomic mass density field in which fast atomic vibrations have been integrated out. The free energy functional of the system supports spatially periodic states, and naturally incorporates elastic and plastic effects, grain boundaries, free surfaces, and arbitrary crystal orientations. Dissipative dynamics can be constructed to govern the temporal evolution of the density field at diffusive time scales, inaccessible by direct Molecular Dynamics simulations. In this talk I will discuss recent efforts in incorporating ferroelectric phenomena within the PFC formalism.

11:35 AM Invited
Diffusive Molecular Dynamics: *Ju Li*[1]; William Cox[1]; Thomas Lenosky[1]; Ning Ma[1]; Yunzhi Wang[1]; [1]Ohio State University

Starting from the variational Gaussian approach of LeSar et al. to approximate the vibrational entropy in solids, we develop a method called diffusive molecular dynamics (DMD) to overcome the timescale limitation of MD. Akin to the phase field crystal model, DMD has atomic spatial resolution but evolves at diffusion timescale. We assign fractional occupation to Gaussian basis to represent diffusion. The occupation on a Gaussian site evolves according to the chemical potential differences with its nearest neighbors. The DMD model can be shown to be equivalent to the regular solution model in treating configurational entropy. Gradient thermodynamics, long-rangeelastic stress and short-range interaction effects are all taken into account automatically. We have applied DMD to model dislocation climb and sintering.

12:00 PM
Phase-Field Crystal Model for Epitaxial Growth: *Kuo-An Wu*[1]; Dong Hee Yeon[2]; Katsuyo Thornton[2]; Ken Elder[3]; Peter Voorhees[1]; [1]Northwestern University; [2]University of Michigan; [3]Oakland University

The original phase-field crystal model by Elder has successfully modeled various non-equilibrium systems. We present here a modified phase-field crystal approach to simulate epitaxial growth in which the vapor phase is introduced by a new phase field. This model extends the original phase-field crystal model which describes liquid and solid phases to a model that can be used to model vapor, liquid and solid phases. This model is used to investigate step flow dynamics, island formation and growth, and the effects of stress generated by a lattice misfit between the film and substrate on the dynamics of film growth.

12:15 PM
Phase Field Method on a Discrete Lattice: *Tetsuo Mohri*[1]; Munekazu Ohno[1]; Ying Chen[2]; [1]Hokkaido University; [2]University of Tokyo

Phase Field Method (PFM) has been attracting broad attentions. PFM, however, is basically a phenomenological method in a sense that the length scale and time scale are not determined self-consistently. Cluster Variation Method (CVM) and Path Probability Method (PPM) were devised as the approximate schemes to calculate phase equilibria and time evolution kinetics on a discrete lattice. By combining with electronic structure calculations, one is able to derive various thermodynamic quantities including a phase diagram from the first principles. We combined PFM with CVM+PPM to fix the length and time scales, and first principles calculation of microstructural evolution is attempted.

Deformation Twinning: Formation Mechanisms and Effects on Material Plasticity: Experiments and Modeling: Twinning and Associated Defect Structures

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: George Gray, Los Alamos National Laboratory; Subhash Mahajan, Arizona State University; Ellen Cerreta, Los Alamos National Laboratory

Tuesday AM — Room: 383
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: David Bacon, University of Liverpool

8:30 AM Invited
Constitutive Modeling of the Slip-Twinning Transition and Its Applications to the High Strain Rate Regime: *Marc Meyers*[1]; Hussam Jarmakani[1]; Eduardo Bringa[1]; [1]University of California

Slip and twinning are presented as competing deformation mechanisms. This concept, originally proposed by G. Thomas, is applied to different situations and sucessfully rationalizes the differences in microstructure and behavior. 1. Effect of Grain Size, Temperature, Stacking-Fault Energy, and Strain Rate: the model is applied to FCC, BCC, and HCP metals and the effects are predicted and compared with observations. 2. Shock and Isentropic Compression: results from gas gun and laser experiments are analysed in conjunction with molecular dynamics simulations. The Zerilli-Armstong and Swegle-Grady relations are used to predict the critical twinning pressure as a function of grain size in shock and isentropic compression of Ni. The grain size is varied from the conventional to the nanocrystalline regime. 3. Martensitic Transformation: the criterion is applied to steel and sucessfully explains and predicts the transition from lath to twinned martensite at a critical carbon content.

9:00 AM
Dislocation Nucleation at the Vicinal Coherent Twin in FCC Cu and Al: *Mark Tschopp*[1]; Douglas Medlin[2]; David McDowell[3]; [1]Air Force Research Laboratories/University Transportation Center ; [2]Sandia National Laboratories; [3]Georgia Institute of Technology

We have used atomistic simulations to investigate dislocation nucleation from the Σ171 vicinal coherent twin boundary. Simulation results show that in copper, this symmetric tilt boundary structure is composed of alternating 1/3<111> disconnections that possess a dissociated structure. Deformation simulations of the bicrystal configuration under uniaxial tension and compression show very different nucleation mechanisms. For instance, our simulations of copper under compression predict that full crystal lattice dislocations nucleate from the 1/3<111> disconnections. The lattice dislocation emits from one 1/3<111> disconnection leaving a glissile 1/6<112> type defect on the boundary plane that glides to react with the adjacent 1/3<111> defect, forming a second lattice dislocation that also emits into the lattice, thereby restoring a perfect coherent twin boundary. Furthermore, the analysis of the dislocation reactions that occur during dislocation nucleation from the vicinal twin may provide insight into the behavior of twin boundaries during the plastic deformation of polycrystalline materials.

9:20 AM
Localized Twin Shear at Grain Boundaries Leading to Fracture Nucleation: *Thomas Bieler*[1]; Philip Eisenlohr[2]; Deepak Kumar[1]; Marty Crimp[1]; Franz Roters[2]; Dierk Raabe[2]; [1]Michigan State University; [2]Max-Planck-Institut Fur Eisenforschung

In gamma-TiAl, certain mechanical twins interact with a grain boundary to nucleate fracture, which can be accounted for with a fracture initiation parameter based upon the geometry of the Burgers vectors as they are related to slip transfer across the grain boundary and the global stress state. The fracture initiation parameter is able to separate cracked and intact boundary populations. Whether the intense shear will cause cracking depends on elastic anisotropy, primary twin (slip) systems vs. secondary slip systems. Cracked and intact boundaries were also characterized to assess tilt and twist character and whether

TUESDAY AM

they are low SIGMA (or coincident site lattice) boundaries, but these did not strongly influence crack nucleation at the grain boundary. Finite-element crystal plasticity simulations are used to assess heterogeneous deformation that accounts for observed microcrack nucleation in one of the characterized microstructures. This approach is also examined for its application in other metals.

9:40 AM
MD Simulation of Dislocation Slip and Twinning in Tensile Deformation of Single Crystal Magnesium: *Bin Li*[1]; En Ma[1]; Kaliat Ramesh[1]; [1]Johns Hopkins University

The mechanism for plastic deformation of HCP magnesium is rather complex. We present molecular dynamics (MD) simulations on the dislocation structure and twinning in single crystal magnesium, in which external strain was applied perpendicular and parallel to the c axis, respectively. Prismatic slip with type <a> screw dislocation, twinning and detwinning of (10-12) tension twins was obtained when the strain was perpendicular to the c axis [0002], whereas when parallel to the c axis, a new mechanism of pyramidal slip on the (10-12)[10-1-1] was obtained. This pyramidal slip was composed of alternate slips on the two intercepting slip systems 1/3(30-34)1/6[20-2-3] and 1/3(30-38)1/6[40-4-3] with a stable stacking fault. The simulations show that this new dislocation slip mechanism can nucleate the (10-12) tension twinning, hence properly associate the dislocation slip with the (10-12) twinning in HCP Mg.

10:00 AM
1/3<111> Dislocations at FCC Twin Boundaries: *Douglas Medlin*[1]; E. Marquis[1]; F. Léonard[1]; S. Foiles[1]; [1]Sandia National Laboratories

We discuss experimental observations and theoretical analysis of the structure and interactions of 1/3<111> dislocations at twin boundaries in face-centered-cubic metals. These defects can arise at twin boundaries in several ways: as dissociation products of crystal lattice dislocations; via the clustering of point defects; or to accommodate deviations from the exact twin misorientation. We have studied 1/3<111> dislocations at twins in both aluminum and gold by high-resolution transmission electron microscopy and atomistic modeling. Depending on the sign of the Burgers vector, the defect can dissociate by emitting a Shockley partial dislocation and stacking fault into the matrix and leaving a stair-rod dislocation at the interface. The availability of this dissociation path allows for some novel interactions of the defect. Specifically, we discuss how the interaction of the 1/3<111> dislocation with {111}/{112} twin junctions can stabilize an extended fault that bridges two closely spaced twin lamellae.

10:20 AM
Neighbor Effects on Deformation Twinning in Hexagonal Materials: *Erin Barker*[1]; Carlos Tome[1]; Ricardo Lebensohn[1]; [1]Los Alamos National Laboratory

Elasto-plastic and visco-plastic self-consistent polycrystal deformation models have been utilized to simulate the mechanical response of hexagonal materials. These models represent grains as ellipsoidal inclusions within a homogeneous equivalent medium. By using an average neighborhood, all grains within a sample with the same orientation respond the same. This results in over predicting quantities such as the twin volume fraction. The approach discussed herein is a local finite element simulation that explicitly represents the grain geometry. Each grain is assigned its own elastic, visco-plastic material properties and lattice orientation in order to investigate the influence of a grain's neighborhood on its response. Multiple analyzes are conducted on varying the grain orientations to observe the impact on the stress state of the overall sample and the central grain which is held constant and allowed to twin. Such investigation can produce statistical information about twinning and improve the fidelity of averaged polycrystal models.

10:40 AM Break

11:00 AM Invited
Twinning and Twin Interactions in Uranium and Uranium-Niobium Alloys: *Robert Field*[1]; Rodney McCabe[1]; Amy Clarke[1]; [1]Los Alamos National Laboratory

Alpha-U (orthorhombic, Cmcm) displays a plethora of twinning modes, including Type I, Type II, and Compound systems. Interactions between these different twins also are varied and unique. As a result, investigations of uranium and its alloys have often led to new understanding of deformation twinning. Early works by R. Cahn and A. Crocker not only provide insight into twinning mechanisms in this material, but also are considered standard references in twinning theory, including the crystallography of twin crossings and the role of shuffle processes in multiple lattice structures. Similar twinning mechanisms are observed in alloys, particularly U-14at.%Nb, which forms a monoclinically distorted, thermoelastic martensite and displays the shape-memory effect (SME). SME deformation modes display even more interesting and complex twin interactions than those observed in the pure metal. In this talk, twinning systems and interactions will be discussed along with results from recent investigations of both U and U-Nb alloys.

11:30 AM
EBSD Analysis of Deformation Twinning in Alpha-Uranium: *Rodney McCabe*[1]; David Alexander[1]; Robert Field[1]; Donald Brown[1]; George Gray[1]; Carl Cady[1]; [1]Los Alamos National Laboratory

The contribution of twinning to the deformation and fracture of alpha-uranium was examined using electron backscatter diffraction (EBSD). Twinning fractions were measured for quasi-static deformation of textured, fine grained samples for tension and compression in several orientations. Similarly, twinning fractions were measured as a function of depth in a shock loaded uranium plate with the same initial grain size and texture. In both cases, {130} twins are the dominant twinning mode with smaller numbers of '{172}' and {112} twins. Interestingly, while the {130} twins make a significant contribution to deformation, they have a minor effect on the overall texture. For large grained uranium, '{172}' twinning is the dominant twinning mode during tensile loading with smaller numbers of the other twinning modes.

11:50 AM
Study of Dislocations and Twins in Zr Compressed at Liquid Nitrogen Temperatures: *Dhriti Bhattacharyya*[1]; Ellen Cerreta[1]; Rodney McCabe[1]; Amit Misra[1]; Carlos Tome[1]; [1]Los Alamos National Laboratory

Zirconium undergoes extensive twinning during deformation at low temperatures. In this study the activity of dislocations within twins in Zr has been investigated. Clock-rolled and annealed samples of Zr were compressed at 76 K in the through-thickness direction. The deformation microstructure was examined using Electron Backscatter Diffraction and Transmission Electron Microscopy. The twins formed were mainly hcp compressive twins, with K1= {11-22} and η1= <11-2-3>. Both 'a' and 'c' type dislocations were found inside the twins. A fraction of the 'c' type dislocations were visible as elongated loops inside the twins, and others terminated at the twin boundary and are probably sessile products of twin formation. The 'a' and 'c' dislocations were found to be elongated parallel and perpendicular to the twin boundaries, respectively. Crystallographic analysis showed that the dislocations were formed within the twins, implying that the twins themselves were deforming through a dislocation mechanism involving the 'a' dislocations.

12:10 PM
Mechanical Twinning of TWIP-Steels from Scattering Data Using the Pair Distribution Function: *Jae Suk Joung*[1]; Yang Mo Koo[1]; Il-Kyoung Jeong[2]; [1]POSTECH; [2]Pusan University

The good mechanical properties of TWIP austenitic steels are mainly due to mechanical twinning (stacking faults). Therefore, it is important to find correlations between planar-defects and strain, and between planar-defects and constitutions quantitatively. In order to study the correlation, we increased mechanical twinning using tensile tests. In the tests, the strain rate and the temperarture(298K) was constant while the degree of strain varied in specimens with different constitutions. The formation of twins and dislocation cells during the plastic deformation was confirmed by microscopic methods. The mechanical twinning was quantitatively identified by a (X-ray, neutron) diffraction line profile analysis for the defects study. The local structure of TWIP-steels was determined by a direct interprettion of the peaks observed in the pair distribution function(PDF). The function was Fourier-tranformed the scattering data from neutron scattering experiment performed up to high momentum transfer.

Electrode Technology Symposium (formerly Carbon Technology): Anode Raw Materials and Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Carlos Zangiacomi, Phelps Dodge International Corporation; John Johnson, RUSAL Engineering and Technological Center LLC

Tuesday AM — Room: 299
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Abdulmunim Binbrek, DUBAL; Stephen Whelan, Alcoa Inc; Charles Mark Read, Bechtel Corporation

8:30 AM
Coke Blending and Fines Circuit Targeting at the Alcoa Deschambault Smelter: *Michel Gendron*[1]; [1]Alcoa Deschambault Smelter

The continued increase of the demand for Aluminium metal combined with the fluctuations in the quality of aluminium grade coke makes it more challenging for the anode manufacturing plants to deliver steady quality anodes. The low sulphur coke material is becoming less available on the market and the price is steadily increasing. Environment regulations are aiming at reducing sulphur emissions, while the coke suppliers are offering higher sulphur material. The use of low sulphur material as feed stock increases anode CO_2 reactivity and therefore making the product less attractive for the downstream process. An attempt was made to use only the high sulphur material in the fines fraction, in order to optimise CO_2 reactivity while respecting sulphur emissions limit and coke supplier agreements. This paper presents the detailed results of this test, which later became a process flowsheet modification.

8:55 AM
Design Criteria for Petcoke Calciners: *Ravindra Narvekar*[1]; Jose Botelho[2]; Arun Kumar Mathur[1]; [1]Goa Petcoke Consultancy Services; [2]Baltin Metal Ltd.

Goa Petcoke Consultancy Services provides comprehensive consultancy services and solutions in the field of petroleum coke calcination. During the course of working on various assignments, we probed into fundamental principles underlying design of rotary kiln systems for petcoke calcination, resulting into evolution of this paper. The important process criteria in calciner design are retention time, heating up rate and operating drafts. These factors depend on kiln dimensions, slope, heat transfer rate and air injections. To understand this well, we have worked out mass and heat balance across kiln, cooler and incinerator. The mechanical design should consider load factors, thermal expansions and strength of materials at elevated temperatures to ensure mechanical soundness and protection against flexing and distortion of kiln shell. The refractory design should take into account differential thermal expansion coefficients, ability to withstand high temperatures, resistance to spalling, chemical attack, abrasion and good coatability.

9:20 AM
Understanding the Calcined Coke VBD- Porosity Paradox: *Bernard Vitchus*[1]; Frank Cannova[1]; Randall Bowers[2]; Shridas Ningileri[2]; [1]BP Coke; [2]Secat, Inc.

Carbon plants use either calcined coke bulk density or porosity analysis to predict pitch content and anode density. However, there is a calcined coke VBD-porosity paradox. That is, high bulk density calcined coke often has higher porosity while low porosity coke tends to make more dense anodes. Understanding why this paradox exists should help lead to consistent high density anodes. Mercury porosimetry and novel techniques were used to characterize calcined coke's shape, internal porosity and packing characteristics. Calcined cokes sieved to a mesh size of -30+50usm from different coke sources were analyzed in the natural, jaw crushed, and roll crushed states. For particle shape analysis, calcined coke particles were suspended in a moving fluid and analyzed with a high speed camera to determine aspect ratios. Internal porosities were investigated by cross-sectioning particles, using optical microscopy techniques, and image analysis to determine area fraction per particle and pore morphology.

9:45 AM Break

9:55 AM
New Analytical Methods to Determine Calcined Coke Porosity, Shape, and Size: *Randall Bowers*[1]; Shridas Ningileri[1]; David Palmlund[2]; Bernard Vitchus[3]; Frank Cannova[3]; [1]Secat, Inc.; [2]Fluid Imaging Technologies, Inc.; [3]BP Coke

Carbon Anode properties especially density are dependent on the structure and porosity of calcined coke used to fabricate the anode. Optimum anode density is produced from blending together natural, rolled, and crushed calcined coke with their inherent structure and porosity characteristics. Two tests were developed to characterize the particle shape including internal and between particle porosity. The first method is a novel technique developed by Fluid Imaging of suspending particles in a moving fluid during which a high speed camera is used to obtain a silhouetted outline of the particle shape and size. Secondly, using traditional optical microscopy techniques and image analysis, internal porosity of the calcined coke was determined. The results of the two methods are presented and compared with evaluation techniques found in literature. These techniques were used to obtain a better understanding of the Calcined Coke VBD - Porosity Paradox presented in a subsequent paper.

10:20 AM
Coke and Anode Desulfurization Studies: Les Edwards[1]; *Lorentz Lossius*[2]; [1]CII Carbon LLC; [2]Hydro Aluminium

The sulfur level of high sulfur cokes used by the calcining industry for blending is increasing. During calcination, petroleum coke partially desulfurizes and the rate of sulfur loss is dependent on both the sulfur level and final temperature. Desulfurization negatively affects coke properties such as real density and porosity and additional desulfurization during anode baking can negatively affect anode properties. This paper is a follow-up to a 2007 TMS paper on desulfurization and presents the results of additional studies on anode and coke desulfurization versus equivalent baking level and discusses the effect of the level of calcination of high sulfur cokes on anode reactivity. The results indicate that the coke calcination levels must be set with coke sulfur levels in mind. It also shows that blending with high sulfur cokes need not be detrimental to most anode properties.

10:45 AM
Structural Evaluation of Petroleum Coke and Coal Tar Pitch for the Elaboration of Anodes in the Industry of the Aluminum: *Rafael Tosta*[1]; Evelyn Inzunza[1]; [1]CVG Alcasa

The present work shows a microstructure's evaluation and changes during the thermal treatment of two types of cokes and three types of Coal Tar Pitch. The objective was to establish the applicability of the optic and electronic microscopy technique, as well as XRD to determine the grade of calcinations of the coke and pitch through the Lc, as a source of additional information and quality control to select cokes and pitches for desirable anode properties. The investigation was carried out at laboratory scale; for the analysis both coke and pitch were calcined and then evaluated at different temperatures and times. The results showed that with the application of technical microscopic and XRD, it was possible to selected better raw materials quality and made the necessary process adjustments to tackle with different raw materials quality.

Emerging Interconnect and Packaging Technologies: Advanced Interconnects

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Carol Handwerker, Purdue University; Srinivas Chada, Medtronic; Fay Hua, Intel Corporation; Kejun Zeng, Texas Instruments, Inc.

Tuesday AM Room: 275
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Kejun Zeng, Texas Instruments Inc; Srinivas Chada, Medtronic

8:30 AM Introductory Comments

8:35 AM Invited
Advances in Electronic Packaging Technologies: *Darrel Frear*[1]; [1]Freescale Semiconductor
The trend for advanced packages for microelectronics is towards smaller feature size, faster speeds, more complexity, higher power and lower cost. With the tremendous growth of wireless telecommunication, RF, Analog and Sensor applications are beginning to drive many areas of microelectronics packaging traditionally led by the development of the microprocessor. An increasingly dominant factor in RF, Analog and Sensor packaging are the materials in the package because they have an increasing dominant affect performance. Trends in Advanced Packaging technology will be highlighted including the revolutionary Redistributed Chip Package (RCP) that offers a one third reduction in size and cost over the best current packages solutions. This presentation will also discuss key materials processing and reliability issues that must be overcome to enable all advanced packages. This includes interconnect (including solder), substrate, and encapsulation materials (metals, polymers and ceramics) with a focus on interfacial behavior in the package.

9:05 AM Invited
Interconnection Alternatives - Is Nanotechnology the Solution?: *Alan Rae*[1]; [1]Nanodynamics Inc
In the electronics industry we are between a rock and a hard place. As more complex devices exhibit a decreased tolerance to process temperatures we have increased the soldering temperature due to the adoption of SAC (tin-silver-copper). Not only that, but in an attempt to reduce the cost of the alloy we reduce the silver content at the same time as we are soldering more complex boards, both of which increase the reflow temperature further! iNEMI has initiated a "warm assembly" program aimed at reducing the process temperature back to tin-lead temperatures or even lower by using nano-sized solder and carbon nanotube arrays. Others are using novel build-up techniques, microsprings and other concepts to enable "room-temperature assembly. This paper outlines some of the promising technology solutions with examples.

9:35 AM Invited
An Overview on Pb-Free, Flip-Chip Wafer Bumping Technologies: *Sung Kang*[1]; Peter Gruber[1]; Da-Yuan Shih[1]; [1]IBM Corporation
To meet the RoHS guidelines and the demand for lower costs, finer pitch and higher quality, considerable work is going on in electronic industry to develop Pb-free solutions for flip chip technology. In this paper, various solder bumping technologies developed for flip-chip applications are reviewed with an emphasis on a new wafer bumping technology called C4NP (controlled collapse chip connect new process). Several inherent advantages of C4NP technology are discussed over other technologies. This paper will also discuss the recent development and implementation of Pb-free C4 interconnections for 300 mm wafers demonstrated at IBM. In addition, some metallurgical issues associated with C4NP technology are discussed.

10:05 AM Break

10:20 AM
Thermal Degradation of Al/Ni(V)/Cu-UBM in Flip Chip Solder Joints: *John Osenbach*[1]; [1]LSI/Package Development
The resistance-time behavior of flip chip solder joints made with Al/Ni(V)/Cu-UBM , eutectic Pb-Sn solder bumps on substrates with either Electroless Ni(P)-Immersion Gold (ENIG) or Cu surface finish was determined at temperatures between 150C and 175C. Degradation of the ENIG based devices was well characterized by a single Arrhenius expression with an effective activation energy, Ea, of approximately 1.57eV and a pre-exponential constant, At, of approximately $1x10^{-16}$/hour. In contrast the resistance remained essentially constant over the entire aging time interval for the device made on Cu independent of aging temperature. The stability at 175C of solder joints made on Cu was found to be more than 50 time greater than equivalent joints made on ENIG . The detailed degradation mechanism based on detailed microstructural studies will be reported in a companion paper "Microstructural Changes in Al/Ni(V)/Cu-UBM Resultant From High Temperature Induced Degradation of Flip Chip Solder Joints."

10:35 AM
Microstructural Changes in Al/Ni(V)/Cu-UBM Resultant from High Temperature Induced Degradation of Flip Chip Solder Joints: *John Osenbach*[1]; [1]LSI/Package Development
The time dependent microstructural and chemical characterization of flip chip solder joints made with Al/Ni(V)/Cu-UBM and eutectic Pb-Sn solder bumps on either Electroless Ni(P)-Immersion Gold (ENIG) or Cu aged at 150C to 175C are reported. The microstructure and chemical composition of these joints were determined with a combination of techniques including Scanning Electron Microspcopy (SEM), Energy Dispersive Spectroscopy (EDS), Focused Ion Beam Microscopy (FIB), and Transmission Electron Microscpopy (TEM). The microstructural characterization clearly showed that electrical degradation of the ENIG devices was a direct result of the conversion and interfacial oxidation of the as deposited Ni(V)-barrier UBM layer into a porous layer that contained a web like fine grain V3Sn IMC. This conversion was driven by the Au from the ENIG surface finish. No such conversion was observed for the devices assembled on Cu-SOP surface finish substrates.

10:50 AM
Lead Free Solder Using TRIP Effect: *Kyu-Oh Lee*[1]; John Morris[1]; Fay Hua[2]; [1]University of California, Berkeley; [2]Intel Corporation
The research reported here explored the use of transformation induced plasticity effect to enhance stress relaxation in lead free solder. The materials tested were Sn-In solders with 9-15 wt. per cent indium, with either Cu:Cu or Ni:Ni metallization. Samples were tested in simple shear at various temperatures to measure the deformation-induced martensite transformation temperature and study creep and stress relaxation behavior. Deformation-induced martensitic transformations were observed in this set of alloys. The transformation is characterized by a significant increase in elongation to failure, and by the appearance of lens-shaped martensite plates in the microstructure. The potential uses of TRIP behavior in solder joints is discussed on the basis of these results.

11:05 AM
Direct Observation of Hot Spots in Flip-Chip Solder Joints under Current Stressing Using Infrared Microscopy: *Hsiang-Yao Hsiao*[1]; Chih Chen[1]; [1]National Chiao Tung University
The temperature and hot spot in the SnAg3.5 solder joints were directly obtained using thermal infrared microscope in this study. Previous simulation results indicate that there exists a hot spot. However, there are no experimental results to verify it. In this study, a hot spot is observed when the applied current is higher than 0.4A. The horizontal thermal gradient near the chip side of the bump with a 5-µmCu UBM can be as high as 571°C/cm when 0.4A was applied at 100°C. As the applied current increased,the difference between the hot spot and the average temperature continue to increase. It may be as large as 29°C when the solder joint was powered by 0.6A. In addition, the hot spot and average temperature decrease with a thicker UBM consisting 5-µmCu/3-µmNi UBM. This hot spot may play an important role in predicting the MTTF of solder joints and the failure mode of electromigration failure.

11:20 AM
Interfacial Reactions of Die Attached AlN-DBC Module Using Zn-Sn High Temperature Solders: *Seongjun Kim*[1]; Keun-Soo Kim[2]; Do-seop Kim[2]; Katsuaki Suganuma[2]; [1]Osaka University; [2]Osaka University, Institute of Scientific and Industrial Research

For the higher thermal dissipation and operating temperature, power modules in a hybrid car employ the AlN-DBC substrate, which has an excellent thermal conductivity. And the high temperature solder for this application must be used beyond 150ºC. In spite of the RoHS regulation, however, very few attempts have been made for developing an alternative high temperature solder which can replace the high Pb solders. In the previous study, we suggested Zn-xSn(x=20, 30, 40wt%) solders as one of the best candidates. To evaluate the possibility of this alloy further, we have investigated the interfacial reactions of the die attached AlN-DBC module by using Zn-xSn solders. Through the microstructural analysis, the effect of soldering condition and the alloying contents were evaluated. Thermal conductivity of solder alloys were examined using a Xe-flash measurement. And the reliability of joint was evaluated by using the reflow test and thermal cycling test.

11:35 AM
The Microstructure Changes, Diffusion Mechanism and Kinetics of the Intermetallics Growth in Ag/Ag Interconnections: *Pawel Zieba*[1]; Joanna Wojewoda[1]; Anna Wierzbicka[1]; [1]Polish Academy of Sciences

Application of the diffusion soldering technology with transient presence of liquid indium or tin solder allowed to obtain stable Ag/Ag interconnections. Three different techniques were verified where the solder was: sputter deposited on the substrate surface, electrochemically deposited or clamp as the thin foil between silver pads. Comprehensive description of the joint microstructure and interfaces was performed using optical, scanning and transmission electron microscopy. Moreover, the chemical composition and the sequence of appearance of the intermetallic phases was determined. On the other hand, the intermetallic growth kinetics study resulted in evaluation of the diffusion mechanism and diffusivity values.

Emerging Methods to Understand Mechanical Behavior: Indentation and Time-Resolved Methods

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Brad Boyce, Sandia National Laboratories; Mark Bourke, Los Alamos National Laboratory; Xiaodong Li, University of South Carolina; Erica Lilleodden, Forschungszentrum

Tuesday AM	Room: 285
March 11, 2008	Location: Ernest Morial Convention Center

Session Chairs: Xiaodong Li, University of South Carolina; Amit Misra, Los Alamos National Laboratory

8:30 AM
Quasi-Static and Dynamic Testing of Wear Tested Single Crystal Nickel: *Neville Moody*[1]; Megan Cordill[2]; Somuri Prasad[1]; William Gerberich[2]; [1]Sandia National Laboratories; [2]University of Minnesota

Nanoindentation is often the test chosen for measuring mechanical properties of small volumes. It is the test we use to measure properties of materials for MEMS applications. However, properties can differ significantly depending on the use of quasi-static or dynamic test methods. As a consequence, we used both test methods to measure mechanical properties in wear tested single crystal nickel. The results show that hardness values from quasi-static tests exhibit the same effect of load and number of cycles used to generate wear patterns as those from dynamic tests but are consistently higher. More importantly, when bulk baseline hardness values are subtracted from the measured values the values superimpose. In this presentation, we will show that both techniques accurately describe the effects of wear on properties and discuss the factors that lead to differences in measured values. This work was supported by Sandia National Laboratories under USDOE contract DE-AC04-94AL85000.

8:50 AM
Study on the Indentation Size Effect in CaF_2: Dislocation Structure and Hardness: *Karsten Durst*[1]; Peiman Sadrabadi[1]; Mathias Göken[1]; [1]University Erlangen-Nürnberg

Understanding size effects in indentation testing requires the analysis of the underlying dislocation structure. In this study nanoindentations have been performed on a cleaved CaF_2 crystal. The deformation during indentation is first purely elastic until dislocations are created, observable in a pop-in the load displacement data. After pop-in a relatively high hardness is observed, which gradually decreases, until a nearly constant hardness is found. This behavior is modeled from 50 nm to 3500 nm indentation depth using a modified Nix/Gao model, where geometrically necessary dislocations and statistically stored dislocations are considered. The dislocation structure is quantified using a "nano" dislocation etch pit technique in conjunction with AFM. By using polishing and sequential etching and imaging, the dislocation structure is studied in three dimensions around and underneath the indented material. The observed 3D dislocation structure will be discussed within the Taylor dislocation hardening model.

9:10 AM
Mechanical and Electrical Properties of Sputtered Single Crystal Cu Films with Nanoscale Growth Twins: *Osman Anderoglu*[1]; Amit Misra[2]; Vibhor Jain[1]; Haiyan Wang[1]; Filip Ronning[2]; Michael Hundley[2]; Richard Hoagland[2]; Xinghang Zhang[1]; [1]Texas A&M University; [2]Los Alamos National Laboratory

We have synthesized single crystal Cu thin films via magnetron sputtering technique onto Si substrates with different orientations. Single crystal Cu films grown on Si (110) substrate possess high density {111} type growth twins. Single crystal Cu films with nanotwins have a hardness of 2.5 GPa with electrical resistivity comparable to that of bulk Cu. Single crystal Cu of {100} type grown on Si (100) substrate has a hardness of 1.2 GPa with bulk Cu resistivity. The influence of deposition parameters on the electrical and mechanical properties of polycrystalline Cu films will also be discussed.

9:30 AM
Improvement of Extracting Material Properties by Sharp Nanoindentation with Valid Reduced Modulus for Elasto-Plastic Materials: *Insuk Choi*[1]; Yanping Cao[2]; Ruth Schwaiger[1]; Oliver Kraft[1]; [1]Forschungszentrum Karlsruhe; [2]Max-Planck-Institut für Eisenforschung GmbH

Recent computational parametric studies have developed reverse algorithms to extract material properties of elasto-plastic materials using experimental sharp nanoindentaion. These methods used reduced modulus in their parameters to include the effect of indenter compliance. To investigate the validity of using reduced modulus, we performed a finite element study for sharp indentation of a wide range of sample materials with rigid, diamond, and sapphire indenters. Then, we conducted experimental indentation of several representative cases. Both computational and experimental results indicate that the usage of reduced modulus is invalid for a certain material regime. For example, the indentation loading responses of Ni are identical for all three tips, whereas the previous predictions using reduced moduli generated deviation of 8%. This difference can cause a substantial error in extracting material properties by reverse algorithms. We will provide comprehensive and systematic study of using reduced modulus so as to improve reverse algorithms.

9:50 AM
Nanoindentation Analysis as a Two-Dimensional Tool for Mapping the Mechanical Properties of Complex Surfaces: *Nicholas Randall*[1]; Jill Powell[1]; [1]CSM Instruments

Nanoindentation has now become established for the single point characterization of hardness and elastic modulus of both bulk and coated materials. This makes it a very good technique for measuring mechanical properties of homogeneous materials. However, many composite materials comprise material phases that cannot be examined in bulk form ex-situ (e.g., carbides in a ferrous matrix, calcium silicate hydrates in cements, etc.). The requirement for in-situ analysis and characterization of chemically complex phases obviates conventional mechanical testing of large specimens representative of these material components. This paper will focus on new developments in the way that nanoindentation can be used as a two-dimensional mapping tool for examining the properties of constituent phases independently of each other. This approach relies on on large arrays of nanoindentations and statistical analysis of the resulting data. Examples will be presented ranging from soft polymer blends to metal-matrix composites.

10:10 AM
Temperature Dependent Viscoelastic Properties of Polymers Investigated by Nanoscale Dynamic Mechanical Analysis: *Yuebin Lu*[1]; Douglas Shinozaki[2]; [1]University of Kentucky; [2]University of Western Ontario

The viscoelastic properties of polymers are investigated by nanoscale dynamic mechanical analysis (DMA) in the temperature range of -100 – +200°C. The polymers tested range from glassy polymer (atactic polystyrene), semicrystalline polymer (high density polyethylene), to rubbery polymer (polyisobutylene). The nanoscale DMA is performed with a flat-tipped microindenter by imposing a sinusoidal displacement of amplitude of less than 40 nm into the specimen and then measuring the responses of load and phase angle. The storage modulus, loss modulus and loss tangent are calculated and found to be independent upon the sample size (thickness). The results from nanoscale DMA are consistent with those from conventional dynamic mechanical analysis by testing the bulk materials in bending mode.

10:30 AM Break

10:45 AM
Nano-Mechanical Analysis of Mechanical Characteristics of a Ultra High Strength Pipeline Steel: *Byoung-Wook Choi*[1]; Jung-Eun Jung[1]; Byung-Gil Yoo[1]; Jae-il Jang[1]; Dong Seo[2]; [1]Hanyang University; [2]Pohang Iron and Steel Company

The mechanical characteristics in micro-phases of two different-typed API X-100 pipeline steels (ultra high strength steels recently developed for the transmission of crucial oil and natural gas) were evaluated by using nanoindentation technique. The steels' microstructures mainly consist of equi-axed/acicular ferrite and bainite. Nanoindentation experiments were made on each phase, and the hardness impressions were analyzed by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The results are discussed in terms of the microstructural effects on the deformation and hardening mechanisms in the steels. This work was supported by Pohang Iron and Steel Company.

11:05 AM Invited
Dynamic Testing: In Situ Diagnostics and Post Test Recovery: *Carl Trujillo*[1]; G.T. Gray III[1]; Shuh-Rong Chen[1]; Philip Rae[1]; [1]Los Alamos National Laboratory

Developing innovative dynamic experimental techniques has enhanced our understanding of mechanical behavior of materials. Various techniques such as velocity interferometer system for any reflector (VISAR) and high speed imaging provide in situ observation of numerous high rate phenomena such as failure, incipient damage, and pressure induced phase transformations. In addition, multiple experimental techniques have been designed to capture and soft recover these specimens for post test analysis. Recovery methods using momentum trapping allow a dynamic test to be arrested for either post test characterization and/or further mechanical testing. This allows for in situ diagnostics to be coupled with post test characterization of specimens via electron backscattered diffraction (EBSD), neutron diffraction, or transmission electron microscopy. The aim of this talk will be to give an overview of the capabilities and recent innovations in dynamic test diagnostics and recovery techniques within the dynamic test facilities in MST-8 at Los Alamos National Laboratories.

11:35 AM
Investigation of Inter and Intramolecular Deformation within Energetic Molecular Single Crystals: *Kyle Ramos*[1]; Von Whitley[1]; Daniel Hooks[1]; Marc Cawkwell[1]; Thomas Sewell[1]; David Bahr[2]; [1]Los Alamos National Laboratory; [2]Washington State University

Multiple materials testing and characterization techniques have been employed in tandem with molecular dynamics simulations in order to investigate inter and intramolecular deformation occurring within brittle molecular single crystals, both for energetic (RDX) and inert systems (sucrose). Nanoindentation coupled with scanning probe microscopy was used to measure hardnesses and reduced moduli while recording deformation features within pile-up zones around indentation impressions to determine deformation mechanisms within these brittle crystals. The onset of plastic deformation has been identified using this technique. Laser-driven shock experiments were used to recover samples for post shock microstructural characterization and were compared to deformation from quasistatic tests, while intramolecular deformation was mapped with confocal Raman microscopy. Plate impact experiments were conducted using a linear resolved optical recording velocity interferometry system (Line ORVIS) to record the spatial homogeneity of orientation dependent impact responses. Molecular dynamics simulations suggest microstructural responses associated with the nanoindentation and plate impact experiments.

11:55 AM
Electromagnetic Ring Expansion for High Strain Rate Tensile Testing: Glenn Daehn[1]; *Jason Johnson*[2]; Gregg Fenton[3]; [1]Ohio State University; [2]Michigan State University; [3]Applied Research Associates, Inc.

The electromagnetically driven expanding ring has been proposed as a test for high strain rate tensile and was highly developed by Gourdin in 1988. This is a very elegant test in that it provides true uniaxial tension and can drive to strain rates over 104 s-1. It is also a very useful test in studying high strain rate ductility and fragmentation behavior. This test never became widely used because the instrumentation was rather expensive and cumbersome and to boundary conditions very simple, rings of very slender cross-sections were used, limiting the strain rates that could be achieved. Here we will detail new attempts to build upon this framework using modern developments in instrumentation and numerical analysis. This efficient test can provide constitutive behavior, as well as ductility and fragmentation data in one straightforward test. Here construction, use and information available from this test will be discussed.

12:15 PM
Growth of Voids during Dynamics Fracture: *James Belak*[1]; Mukul Kumar[1]; John Kinney[1]; Lyle Levine[2]; Jan Ilavsky[3]; [1]Lawrence Livermore National Laboratory; [2]National Institute of Standards and Technology; [3]Argonne National Laboratory

Samples of high purity single and polycrystal Al and Cu were brought to incipient spallation fracture under dynamic loading conditions. X-ray, electron and neutron scattering were used to quantify the nucleation and growth of voids inside at the spall plane. The samples were first analyzed using x-ray tomography to reveal the 3D spatial distribution of porosity. Both small angle x-ray and neutron scattering experiments were performed on the same samples resulting in a dataset spanning four decades in lengthscale. The scattering as calculated from the tomography is compared to the neutron scattering at overlapping length scales. Finally, the samples were sectioned and EBSD was used to characterize the plastic zone surrounding the voids and the microstructural processes of nucleation and growth. This work was performed under the auspices of the U.S. Department of Energy by University of California, LLNL under Contract W-7405-Eng-48.

Enhancing Materials Durability via Surface Engineering: Superalloy Surface Durability

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: High Temperature Alloys Committee

Program Organizers: David Mourer, GE Aircraft Engines; Andrew Rosenberger, US Air Force; Michael Shepard, Air Force Research Laboratory/MLLMN; Bruce Pint, Oak Ridge National Laboratory; Brian Gleeson, University of Pittsburgh

Tuesday AM — Room: 388
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Andrew Rosenberger, US Air Force; David Mourer, GE Aircraft Engines

8:30 AM Introductory Comments

8:35 AM Invited
Directed Vapor Deposition of Advanced Coatings for Aerospace Applications: *Derek Hass*[1]; [1]Directed Vapor Technologies International

Alternative coatings and coating approaches are required to improve the performance of components used in aerospace applications including those in gas turbine engines and aircraft landing gear. Such coatings include thermal barrier coatings used to increase the durability and thermal resistance of hot-section engine components, environmental coatings for oxidation and hot corrosion protection and wear and corrosion protection coatings for landing gear components. Directed Vapor Technologies International (DVTI), is currently investigating the use of an advanced electron beam vapor deposition approach, Directed Vapor Deposition (DVD), as a method for applying high quality coatings at high rates onto aerospace components. The DVD process operates in a novel processing environment that employs a supersonic gas jet to "direct" vapor atoms onto components resulting in highly efficient deposition of complex coating structures and compositions. Here, the use of this approach to deposit advanced coatings for aerospace applications will be discussed.

9:05 AM
Mechanisms of Lifetime Improvement in Thermal Barrier Coatings with Hf and/or Y Modified CMSX-4 Superalloy Substrates: *Jing Liu*[1]; Kenneth Murphy[2]; Yong-ho Sohn[1]; [1]University of Central Florida; [2]Howmet Corporation

Superior thermal cyclic lifetime was observed for thermal barrier coatings (TBCs) with (Ni,Pt)Al bondcoat and Hf- and/or Y-modified CMSX-4 superalloy, where each thermal cycle consisted of 10-minute heat-up, 50-minute dwell at 1135°C, and 10-minute forced-air-quench. Microstructural degradation and failure of TBCs were examined by scanning and transmission electron microscopy. Distribution of Hf in the thermally gown oxide (TGO) scale was examined by secondary ion mass spectroscopy. While rumpling and racheting of the TGO-bondcoat interface were observed to be the main damage characteristic for TBCs on CMSX-4, the same interface remained flat for durable TBCs with modified CMSX-4. For TBCs with superior lifetime, monoclinic HfO2 was observed as find precipitates along the grain boundaries of TGO scale, the parabolic growth constant of the TGO scale was slightly lower, the columnar diameter of the TGO scale was smaller, and Hf and/or Y were observed to segregate to the TGO-bondcoat interface.

9:30 AM
Microstructural Investigation of Multiphase NiAl-Hf Bond Coats on Nickel-Base Superalloys: *Michael Bestor*[1]; Arthur Brown[1]; Richard Martens[1]; Mark Weaver[1]; [1]University of Alabama

It has been reported that NiAl overlay bond coatings containing Hf or Zr concentrations well above their solubility limits can exhibit oxidation resistance that is comparable to state-of-the-art platinum aluminide coatings. In this study, a microstructural investigation has been conducted at UA via tomographic atom probe and transmission electron microscopy of precipitation/dispersion-strengthened NiAl-Hf coatings deposited via DC magnetron sputtering onto single crystal superalloy substrates. Post-deposition annealing of NiAl-1.0 at.% Hf at 1000°C for 1 to 4 hours resulted in the formation of nanometer-sized Hf-rich precipitates. Longer pre-oxidation annealing times resulted in smaller mass gains during isothermal oxidation with values approaching those of model NiAl-0.05 at.% Hf coating alloys. Results suggest that suitable oxidation resistance can be obtained in "over-doped" alloys by optimizing the coating chemistry, grain structure and precipitate distribution.

9:55 AM
Opportunities in $YbO_{1.5}$-TiO_2-ZrO_2 for Application in Thermal Barrier Systems: *Rafael Leckie*[1]; Tobias Schaedler[1]; Anthony Evans[1]; Carlos Levi[1]; [1]University of California at Santa Barbara

Co-doping of yttria stabilized zirconia (YSZ) with rare earth (RE) oxides has provided a promising variety of next generation thermal barrier oxides with low thermal conductivity. However, the advantages offered by these novel compositions typically carry penalties regarding durability which have been broadly associated with a loss of toughness concomitant to the decline in tetragonality of the structure with increasing dopant addition. Ti additions have shown to be beneficial in enhancing toughness in t' zirconias by increasing the tetragonality of the structure. This paper discusses opportunities in the $YbO_{1.5}$-TiO_2- ZrO_2 system regarding the effects of chemical composition on toughness, phase stability, and thermal conductivity and compares it with previous experiences in the Y and Gd systems. The connection between chemical composition, phase equilibria, microstructure and potential toughening mechanisms will be discussed.

10:20 AM Break

10:30 AM
The Effects of Hot Corrosion Attack on the Fatigue Resistance of a Disk Superalloy: *Timothy Gabb*[1]; Jack Telesman[1]; Brian Hazel[2]; David Mourer[2]; [1]NASA Glenn Research Center; [2]General Electric Aircraft Engines

Recently developed powder metallurgy nickel-base superalloys can be employed in gas turbine engines at temperatures approaching 700°C. Hot corrosion attack could occur at such temperatures, if corrosive species are present. The effects of high temperature salt exposures were investigated for the disk superalloy ME3/Rene 104. Fatigue specimens were machined from a full scale turbine disk. The gage sections were coated with a salt-containing mixture and exposed at 704°C in air. Fatigue tests were subsequently performed at 427 and 704°C on exposed specimens. The salt mixture produced localized pitting damage, with sulfides forming below outer oxide layers there. Fatigue life was reduced up to 90% by this hot corrosion damage.

10:55 AM
Evaluating Stability and Erosion Wear Behavior of HVOF Sprayed Cr3C2-20%(Ni20Cr) Coatings at Elevated Temperatures: *Prajina Bhattacharya*[1]; K. Anand[1]; T. Shalini[1]; T. Kluge[1]; [1]General Electric

HVOF processed Cr3C2-20%(Ni20Cr) coating is deposited with GTW of 6 and 10 inches. XRD and SEM characterization reveals Cr3C2 presence with Ni-Cr and Ni-Cr-C solid-solutions. XRD peak sharpness, carbide size and volume fraction vary slightly in the two samples. The coating thermal behavior is studied with DTA coupled with TGA. Coating stability is assessed under exposure to extended aging at 700°C and 800°C. Cr7C3 is prominent after aging. At 700°C, fine carbide distribution increases and at 800°C, pores are evident. Jet erosion of the samples with silica show that the erosion rate decreases after 700°C aging and increases back to the as-deposited value after 800°C aging. Ploughing and compaction are identified as erosion mechanisms. The ploughing effect is more pronounced with elevated temperature aging. The erosion mechanism is investigated with single particle erosion. Samples are subjected to hard quartz and soft magnetite to understand the effect of erodent hardness on the erosion behavior.

11:20 AM
Grain Boundary Engineering of Superalloys for Aerospace Engine Applications: *Peter Lin*[1]; Virgil Provenzano[1]; Robert Heard[1]; Herbet Miller[1]; Gina Palumbo[1]; Timothy Gabb[2]; Kenneth Vecchio[3]; Fengchun Jiang[3]; Jack Telesman[2]; [1]Integran Technologies USA Inc.; [2]NASA Glenn Research Center; [3]University of California, San Diego

Grain Boundary Engineering (GBE) involves microstructural optimization via the strategic application of thermo-mechanical metallurgical processing and fabrication steps that increase the fraction of special, low-energy, and degradation-resistant grain boundaries (i.e., structurally ordered low Σ grain boundaries) in the microstructure. By elevating the fraction of special grain boundaries in a metal or alloy either in the bulk or in the near-surface region, a commensurate improvement in the material properties is achieved owing to the intrinsic degradation-resistance (corrosion, sliding, cracking) of the "special" grain boundaries. Alloys that can benefit from this treatment include nickel-based superalloys that are used in the hot sections of gas turbine engines. Under increasingly demanding operating conditions, these materials can be vulnerable to grain boundary-related elevated-temperature degradation processes including creep, fatigue, solute segregation, precipitation embrittlement, and intergranular environmental attack. In this study, the benefits of GBE-processing on the reliability and durability of Inconel 718 superalloy are presented and discussed with emphasis on the improvement of its resistance to creep and crack growth.

11:45 AM
Effects of Surface Orientation on High-Temperature Oxidation Behavior: *Louis Bonfrisco*[1]; Megan Frary[1]; [1]Boise State University

Surface orientation plays an important role in oxidation behavior; studies of single crystals suggest that {111} surfaces are most resistant to oxidation. However, most materials are polycrystalline and contain numerous orientations which contribute to the oxidation process. Here we determine the effect of orientation on oxidation behavior of metals over all surface orientations. The microstructure is characterized with electron backscatter diffraction (EBSD). After high temperature oxidation, the oxide topography is characterized using optical profilometry (OP). By correlating results from EBSD and OP, the oxide height can be determined for each orientation. The data suggests that as the surface normal deviates from the <111> direction, the oxide thickness increases. The oxidation rate may depend on not only the surface orientation, but the character of the grain boundaries. This technique allows a thorough understanding of the role of surface orientation on oxidation and may provide insight to the production of oxidation-resistant surfaces.

Frontiers in Process Modeling: Metallurgical Reactors

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Materials Processing and Manufacturing Division, TMS: Process Technology and Modeling Committee
Program Organizers: Andrew Campbell, WorleyParsons; Adam Powell, Opennovation

Tuesday AM — Room: 287
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Andrew Campbell, WorleyParsons

8:30 AM Introductory Comments

8:40 AM
Modelling Metal Powder Production by the Gas Atomisation Process: Claudio Lena[1]; Georgi Djambazov[1]; *Koulis Pericleous*[1]; [1]University of Greenwich

Metal powder in the range of 10-100 microns is widely employed in the production of Raney nickel type catalysts for hydrogenation reactions and hydrogen fuel cell manufacture. In this presentation we examine the modelling of powder production in a gas atomisation vessel using CFD techniques. In a fully coupled Lagrangian-Eulerian two phase scheme, liquid meal particles are tracked through the vessel following atomisation of a liquid nickel-aluminium stream. There is full momentum, heat and turbulence transport between particles and surrounding argon gas and the model predicts the position of solidification depending on particle size and undercooled condition. Maps of collision probability of particles at different stages of solidification are computed, to predict the creation of satellite defects, or to initiate solidification of undercooled droplets. The model is used to support experimental work conducted under the ESA/EU project IMPRESS.

9:00 AM
Simulating Morphology of Electrochemical Deposits in Shear Flow: *Adam Powell*[1]; [1]Veryst Engineering LLC

Electrodeposition often takes place in shear flow, either to promote mixing in the aqueous or ionic electrolyte, or sometimes to sweep away deposited powder. Modeling such a system requires solving coupled equations for mass transfer of ions, electric field, moving metal-electrolyte interface, and fluid-structure interactions across that interface. Phase field is a robust thermodynamics-based methodology for mass transfer and phase change, particularly when the interface topology changes. For this reason, several researchers have published phase field models of electrodeposition including dendritic growth. However, the method's diffuse interface complicates fluid-structure interaction modeling. The model presented here uses the mixed stress method to model fluid dynamics in the liquid electrolyte coupled with elastic behavior of solids, including solid particles which break off and float away. While presenting results for electromigration-controlled deposition of an isotropic material in 2-D, this presentation will also discuss extension to 3-D, anisotropy, and charge transfer resistance.

9:20 AM
Simulation of the Dual Cooling Rates of Oil-Quenched Alloy Droplets Produced by the Uniform-Droplet Spray Process: *Suresh Kumar Pillai*[1]; Teiichi Ando[1]; Tatsuya Shoji[2]; [1]Northeastern University; [2]Hitachi Metals Ltd.

A model was developed to simulate the thermal history of mono-size alloy droplets produced by the uniform-droplet spray (UDS) and quenched in oil. The model adopts a metallographic approach to account for the dual-phase heat transfer that the droplets experience in the oil. Model predictions are analyzed with the help of experimental data obtained with a glass-forming Fe45Ni22.5Co7.5Mo3B14Si8 alloy. The ability to predict the droplet cooling in liquid medium allows for the use of smaller spray chambers and inexpensive chamber gases.

9:40 AM
Boundary Element Modeling of Solid Oxide Membrane Process: *Rachel De Lucas*[1]; Adam Powell[2]; Uday Pal[1]; [1]Boston University; [2]Veryst Engineering LLC

Mathematical modeling is a useful tool for design of electrochemical systems such as the magnesium solid-oxide membrane process (Mg-SOM). In this study, the Boundary Element Method (BEM) was particularly suited to the 3-dimensional modeling of the Mg-SOM process. BEM modeling allows one to change the geometry without remeshing, a useful feature for parametric optimization. After benchmarks demonstrated the accuracy of the program, the resulting model calculated electric current density and heat generation rates for both single YSZ membrane and three YSZ membrane geometries of this process. The model then provided a platform for optimization of electrode placement, first to minimize total resistance of the system, and second to maximize current density uniformity at the anode.

10:00 AM
CFD Simulation of Mixing in Stirred Reactor: *Xiaochang Cao*[1]; Ting' an Zhang[1]; Qiuyue Zhao[1]; [1]Northeastern University

Stirred reactors are widely used in mineral and metallurgy industries. Computational Fluid Dynamics (CFD) provides a tool for determining detailed information on fluid flow inside stirred reactors. A new-style stirred reactor was designed for digestion process of minerals. This paper is a preliminary study to demonstrate the CFD simulation of fluid flow in an impeller stirred reactor using Multiple Reference Frames (MRF) impeller rotation model and the standard k–e turbulence model. The computational model is solved using commercial CFD code FLUENT 6.3(FLUENT Inc.). Simulations are achieved at various values of initial rotating speed and flux, and the influences of swirl motion has also been investigated. T he residence time distributions(RTD) curves predicted by CFD model show good agreement with experimental data. The information would be useful for optimizing stirrer's design and enlarging multiphase flow in stirred reactor.

10:20 AM Break

Frontiers in Process Modeling: Casting and General Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Materials Processing and Manufacturing Division, TMS: Process Technology and Modeling Committee
Program Organizers: Andrew Campbell, WorleyParsons; Adam Powell, Opennovation

Tuesday AM — Room: 287
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Adam Powell, Opennovation

10:30 AM
A Closed Form Simulation of Coarsening Analog System: *Vaughan Voller*[1]; [1]University of Minnesota

If a froth of soap bubbles is held between two closely spaced parallel plates it will coarsen-over time, the number of bubbles will decrease and the average area of bubbles will increase. This system has often been used as an analog system for grain growth in metals. In this paper a simple simulation for froth bubble dynamics is developed. This simulation is based on a random walk process for removing bubbles coupled to a Voronoi diagram visualization to track geometric changes in the froth. This simple approach can be reduced to a closed form expression for the bubble dynamics that produces the correct long time scaling and follows the form of the classic Mullins-Neaumann growth law. Predictions from the model are validated against available experimental data. The possible application of the simulation to grain growth is discussed.

10:50 AM
A Generic Enthalpy Based Approach to Incorporate the Kinetics of an Equiaxed Eutectic Microstructure Fromation in Castings – A Concept: *Suresh Sundarraj*[1]; [1]General Motors Corporation

This work summarizes the development of a new Generic Enthalpy Model (GEM) to describe the macroscopic heat transfer during solidification in binary equiaxed eutectic alloy system. For the first time, this model simultaneously accounts for the kinetics and thermodynamics of the solidification process in a seamless manner. The GEM approach accurately predicts the macroscopic cooling curve in a casting region. It has the potential to provide a framework for readily adapting to multi-component alloy systems.

11:10 AM
A Nouvelle Thermo Physical Solution to Casting Deformation Problem in DC Casting of Light Metals: *Dimitry Sediako*[1]; Olga Sediako[2]; [1]National Research Council of Canada; [2]Renfrew County District School Board, Ministry of Education, Ontario

3D model development has become a common place in process computational analysis and technology optimization in DC casting of light metal alloys. The computational systems are usually based on traditional formulation of 3D equation of thermal conductivity coupled with a set of boundary and initial conditions. A problem exists however in calculation of shrinkage/contraction of metal during casting and accommodating this deformation in the dynamically changing boundary conditions. A nouvelle solution has been developed to calculate and account for various types of deformation that are taking place during bloom/billet formation in the DC casting process: shrinkage and contraction, as well as swell and bow type deformation of the butt of the DC castings. The solution offers a new approach to designing the 3D system architecture and extends practical application of the models in the casting process analysis and optimization.

11:30 AM
The Development of Flexible Hot Rolling Technology Based on Through-Process Modeling: *Priya Manohar*[1]; [1]Robert Morris University

A new paradigm is proposed in this paper that underpins the development of flexible rolling technology in industrial processing of C – Mn and Low C - microalloyed steels. Scientific knowledge of industrially-significant processes for these materials is presently fragmented and scattered in published literature. This results in impediments to process optimization and new technology development. In the current work, it is demonstrated that new process sequences could be developed by breaking down existing process routes in to key elements and then by recombining them to generate novel alternative and more efficient hot processing sequences. The proposed methodology establishes a platform for a more realistic assessment of existing process routes and the development of new hybrid process routes that combine ideas from alternative processes. This enables the identification of an optimal process sequence for specified steel compositions that also satisfies simultaneous design criteria such as process feasibility and property maximization.

11:50 AM
Using Computational Techniques to Determine Aluminum Cast House Equipment's Environmental Carbon Contribution at the Design Stage: *Pritpal Sandhu*[1]; [1]Technology Innovation Centre

This paper looks at using computational design to determine the carbon content of cast house equipment. Reviewing current methods used in industry in evaluating carbon footprints and applying these before the production stage. By providing mathematical modeling techniques, which can be applied through software to determine the equipments carbon contribution as part of a Knowledge Based Engineering program. Using computer design simulations to evaluate the changes made towards energy requirements, for purpose of carbon labeling compliance. By automating this information the capturing of carbon footprints and embodied energy information at the design stage, engineers can determine its environmental effects and included them in the products specification. Allowing the end users of the equipment to make informed choices over what cast house products they plan to use.

12:10 PM
Overview: Modeling Material Properties Critical for Process Simulation: Zhanli Guo[1]; Nigel Saunders[1]; Peter Miodownik[1]; *Jean-Philippe Schille*[1]; [1]Sente Software, Ltd.

Process simulation requires reliable data for a wide variety of material properties, ranging from thermal conductivity to flow stress curves. Traditionally such data are gathered from experimentation, which can be time-consuming and costly. This presentation describes the development of computer models that can provide in situ calculations of many material properties, such as solidification properties and high temperature stress-strain curves. The solidification properties are affected by changes in composition within the specification range of an alloy; such changes in properties then also affect casting simulation itself. The mechanical properties are calculated by considering two competing deformation mechanisms (dislocation glide or dislocation climb), with automatic selection of the dominant mechanism. These models have been integrated into the computer software package JMatPro, which can then be used to export data files directly to FE/FD based packages used for casting, forging and deformation simulation.

General Abstracts: Light Metals Division: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Reactive Metals Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Neale Neelameggham, US Magnesium LLC; Anne Kvithyld, SINTEF

Tuesday AM — Room: 297
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Thorvald Abel Engh, Norwegian University of Science and Technology; Srinath Viswanathan, University of Alabama

8:30 AM
Microstructure and Properties of Gas-Atomised and Ingot-Route Burti: *Hui Jang*[1]; Michael Loretto[1]; Dawei Hu[1]; Xinhua Wu[1]; Francesco Paster[2]; Michael Preuss[2]; Phillip Withers[2]; [1]University of Birmingham; [2]University of Manchester

The response to imposed tensile deformation of hot isostatically pressed (HIPped) powder BurTi, the burn-resistant Ti alloy, Ti25V15Cr2Al0.2C (wt%), has been compared with that of extruded ingot-route material. Both gas-atomised powder and PREP powder have been used. Some powder HIPped samples were heat treated at a temperature above the HIPping temperature before testing. Acoustic emission, synchrotron radiation and transmission electron microscopy have been used to assess the response to strain up to failure. The importance of any porosity in the powders has been assessed and detailed observations will be reported on the influence of process-route on behaviour.

8:50 AM
Prefabricated Materials Preforms in Al and Ti Using Friction Welding: *Philip Threadgill*[1]; Bradley Wynne[2]; [1]TWI; [2]Sheffield University

Many complex aerospace components are machined from expensive large blocks of materials which often have long lead times. Many of these materials, especially titanium alloys, are difficult to machine, and since the fly/buy rations can be below 10%, this is a financially and environmentally unattractive approach. Many machining preforms can be easily manufactured from smaller pieces of materials for relatively low cost. Several approaches are available, but this presentation will consider the benefits of friction welding and friction stir welding. Examples will be given of recent work which highlights the possibilities for aluminium and titanium alloys, using various friction based approaches. The presentation will also include detailed microstructure analysis of welded samples via optical and scanning electron microscopy as well as orientation image maps obtained by electron backscattered diffraction, which demonstrate the excellent microstructures which can be obtained.

9:10 AM
High Temperature Oxidation Studies of Ti_3Al-4.0at%Mo in Pure Oxygen: *Christopher Williams*[1]; [1]University of Alabama

Ti_3Al-4.0at%Mo samples were oxidized under pure oxygen atmosphere at temperatures ranging from 1023-1223 K in a Thermogravimetric Analysis (TGA) apparatus. Effective activation energy of 294 kJ/mol was determined from the weight gain data. This composition oxidizes more rapidly than binary Ti_3Al alloy of comparable composition as well as alloys containing smaller concentrations of Mo found in the literature. Reaction products consisted of rutile and alumina particles.

9:30 AM
Fabrication of Carbon Fibers Reinforced Aluminum Matrix Composites: *Chitoshi Masuda*[1]; Yu-suke Nishimiya[1]; [1]Waseda University

Carbon fiber reinforced aluminum matrix composites were expected for the application of automobile engine parts such as radiator, piston head, and engine wall due to the high strength and high heat exchange rate. Composites were fabricated by powder metallurgy method. The aluminum powder were blended in to the carbon fibers and mixing with the ball mill in alcohol solutions. After milling the solutions were heated at low temperature for 24 hours. The mixed powders were consolidated by Spark Plasma Sintering machine at maximum temperature of 585°C for 1 hour. The microstructures were examined and thermal conductivity was measured using laser flash method and DSC method. The chopped carbon fibers were homogenously distributed up to about the volume fraction of 50wt%. On the other hand, the CNT was not so homogenous distribution up to about the volume fraction of 30wt%. Carbon nano-fibers(VGCF) were almost homogenously distributed after ball milling.

9:50 AM
Bonding of Steel Inserts during Aluminum Casting: Q. Han[1]; *Srinath Viswanathan*[2]; K. More[3]; M. Myers[4]; M. Warwick[4]; Y. Chen[4]; [1]Purdue University, Department of Mechanical Engineering Technology; [2]University of Alabama, Department of Metallurgical and Materials Engineering; [3]Oak Ridge National Laboratory, Materials Science and Technology Division; [4]Cummins, Inc., Columbus Technical Center

Technologies have been developed to reinforce aluminum castings with steel inserts. Defect-free bonds have been consistently obtained between the steel insert and the aluminum casting. A push-out experiment indicated that the bond strength was higher than previous approaches. This paper presents the results of the technology development and the characterization of the bonds.

10:10 AM Break

10:20 AM
Low Temperature Electrochemical Synthesis of Ti-Al Alloys Using Ionic Luquid Electrolytes: *Debabrata Pradhan*[1]; Ramana Reddy[1]; [1]University of Alabama

The synthesis of Ti-Al alloys were carried out using $AlCl_3$-1-butyl-3-methyl imidazolium chloride (BmimCl) electrolyte at different temperature between 70°C and 125±3°C. The alloy was deposited on titanium cathode at various voltages between 1.5V and 3.0V. The molar ratio of $AlCl_3$ and BmimCl is 2:1 for all experiments. Titanium sheet (>99.99 wt%) was used as anode. Morphology and composition of electrodeposited Ti-Al alloy were characterized using SEM/EDS and ICP-OES respectively. Also, the effect of applied voltage and temperature on cathode current density, current efficiency, composition and morphology of electrodeposit were investigated. The Ti-Al alloys containing 26-37 wt% Ti were produced with a current efficiency of about 80-85%.

10:40 AM
Residual Life Extension of Aged Transformers in Aluminium Smelters: *Santhosh Kumar Rajamoorthy*[1]; Muralidharan Kesavan[1]; Saravanan Krishnamoorthy[1]; [1]Madras Aluminum Company Ltd.

The modern world is increasingly dependent on electricity for economic prosperity. Without doubt the single most important piece of equipment in electrical networks in Aluminium Smelters is the Power transformer. However the average age of transformers around the world is increasing rapidly with transformer failures rising exponentially, and to replace all of the aged power transformers at risk would be prohibitively expensive. This paper focuses on the extension of residual life of these aged transformers through online reclamation of oil a process through which the quality of oil is renewed better than the new oil meeting the standards of a new oil thereby the life.

11:00 AM
Revamping of Rodding Shop Worldwide: *Jean-Jacques Grunspan*[1]; [1]BROCHOT SA

The decision to revamp a rodding shop after 20 or 30 years use is a strategic decision for an aluminium smelter in view of its potential threat to the continuity of supply of rodded anodes to the potrooms. BROCHOT has undertaken the revamping of 3 rodding shops in 3 different smelters (PNL Netherlands, Lynemouth UK and DUBAL in UAE). In this article, it shares its experience by highlighting those factors which must necessarily be considered before undertaking any such revamping project so as to prevent major problems.

11:20 AM
Expert System on Properties of Molten Electrolytes: *Alexander Redkin*[1]; Anatoly Shurygin[1]; Olga Tkacheva[1]; [1]Institute of High Temperature Electrochemistry

Development of new electrochemical technologies requires new electrolytes. Every change in the electrolyte composition leads to property change. Experimental investigation of molten electrolytes properties, such as density, electrical and thermal conductivity etc. takes much time. The physical model connecting above mentioned parameters is needed in order to evaluate the properties. We propose empirical model that allows describing the most of electrolyte properties as a function of molar volume and temperature. This model is used in our expert system "Molten Salt Properties" which makes possible to describe existing experimental data or evaluate some unmeasured properties for new electrolytes. The work is based on experience, knowledge and experimental results that have been collecting at the Institute of High Temperature Electrochemistry during 50 years of molten salts investigations.

General Abstracts: Materials Processing and Manufacturing Division: Composition Structure Property Relationships II

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: Global Innovations Committee, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Phase Transformations Committee, TMS: Powder Materials Committee, TMS: Process Technology and Modeling Committee, TMS: Shaping and Forming Committee, TMS: Solidification Committee, TMS: Surface Engineering Committee

Program Organizers: Ralph Napolitano, Iowa State University; Neville Moody, Sandia National Laboratories

Tuesday AM Room: 282
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Bryony James, University of Auckland; Christopher Roberts, Carnegie Mellon University

8:30 AM
Towards Single Crystals Using Strain Induced Grain Growth Part I: Experimental Results with Pure Iron: *Benjamin Fell*[1]; Tien Tran[1]; Corentin Guebels[1]; Jean-Pierre Delplanque[1]; Joanna Groza[1]; [1]University of California at Davis

Experimental results from creep tests on pure iron are presented in the context of static (SRX) and dynamic recrystallization (DRX). The aim of the experiments are to, first, provide necessary recrystallization and grain growth data across various strain rates and maximum strain values to calibrate DRX simulation models and, second, produce abnormal grain growth (AGG) through strain assisted recrystallization. Producing single crystals with solid state techniques has been shown to be advantageous for allotropic materials such as molybdenum and uranium. The results show that grain growth is a function of strain rate and that abnormal grain growth initiates in the neck region across all strain rates. The experimental program investigates strain rates in the creep regime (10-4 to 10-5 /s) at temperatures ranging from 800 to 900ºC (~0.6Tm) to determine the sensitivity of these parameters in initiating AGG.

8:50 AM
Towards Single Crystals Using Strain Induced Grain Growth Part II: Monte Carlo Simulations and Physical Time Predictions: *Corentin Guebels*[1]; Benjamin Fell[1]; Tien Tran[1]; Joanna Groza[1]; Jean-Pierre Delplanque[1]; [1]University of California, Davis

The microstructural evolution and kinetics of thermal and strain-induced grain growth were analyzed using a two dimensional Monte Carlo Potts (MCP) model. MCP simulations have captured various grain growth behaviors; however, the stochastic nature of the MCP time-step limits its verification with physical data. An energy-controlled relationship between MCP and physical time-steps was established using thermal grain growth simulations and published experimental data. The case of static strain-induced growth is considered next. The modeling strategies for the strain-induced energy, recovery and nucleation and their implementations are discussed in connection with experimental evidence.

9:10 AM
Elevated Temperature Uniaxial Compression Testing of Sintered ZrN and (Zr, Ti)N Pellets as Surrogate for PuN and (Pu,Zr)N Fuel Pellets: *Kirk Wheeler*[1]; Pedro Peralta[1]; Kenneth McClellan[2]; [1]Arizona State University; [2]Los Alamos National Laboratories

In this work, ZrN and (Zr,Ti)N were studied as possible surrogates for PuN and (Pu, Zr)N under the Global Nuclear Energy Partnership (GNEP) program. The mechanical properties of sintered ZrN and (Zr, Ti)N pellets were examined to investigate the effects that sintering temperatures and material segregation have on structural integrity at various working temperatures. Uniaxial compression testing was performed on ZrN and (Zr,Ti)N pellets sintered at various temperatures. Testing was performed in an ultra-high purity Argon atmosphere at various temperatures (25°C, 800°C, 1200°C). Post-Mortem fractography was performed using optical and electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS). The failure modes of the pellets and their mechanical properties are evaluated in terms of the initial microstructure. Applicability of the results to the understanding of the elevated strength properties of multi-element transmutation fuel pellets is discussed. Work supported under DOE/NE Agreement # DE-FC07-05ID14654.

9:30 AM
Mesh-Insensitive Analysis of Dynamic Shear Banding in Metal Alloys: X. Poulain[1]; *Amine Benzerga*[1]; [1]Texas A&M University

The cost performance of various manufacturing processes requires accurate prediction of shear band formation and propagation. Conventional finite-element simulations of shear banding and subsequent ductile failure in metallic alloys suffer from pathological mesh sensitivity due to the softening character of constitutive equations. Incorporation of material rate-sensitivity reduces this pathological dependence and can eliminate it. However, there are circumstances where significant mesh size effects remain, which complicates the interpretation of shear band induced fracture. Here, we develop a computational framework that aims at regularizing the boundary-value problem solutions by the introduction of a material length scale. To this end, a new ductile damage model is used with strain gradients over distances comparable with void spacing included along with work-conjugate higher-order stresses. After presenting the numerical methods used for implementation in finite element code, fully transient analysis of shear banding and shear-induced failure will be presented.

9:50 AM
Variations in Plasticity and Fracture with Respect to Crystallographic Orientation in Individual Grains of Sintered ZrN Pellets: *Kirk Wheeler*[1]; Pedro Peralta[1]; Kenneth McClellan[2]; [1]Arizona State University; [2]Los Alamos National Laboratories

ZrN was studied as a possible surrogate material for PuN under the Global Nuclear Energy Partnership (GNEP) program. In particular, this work addresses variability on mechanical properties of ZrN sintered at 1600°C on a sub-micron scale as a result of grain crystallographic orientations. Nano-indentation using Berkovich and Cube Corner indenters are used to determine plasticity variations and to induce cracking in different grains of a dense ZrN sample. Seven different crystallographic orientations were selected via Electron Backscattering Diffraction (EBSD) and isolated with fiducial markings using a Focused Ion Beam (FIB) and nano-indents are examined using Atomic Force Microscopy (AFM). Post indentation characterization and indent cross sectioning are performed using a FIB to correlate surface topography, cracking behavior and indentation shape to local crystallography. The implications of the results for multiscale simulations of structural reliability in ceramic nuclear fuels are discussed. Work supported under DOE/NE Agreement # DE-FC07-05ID14654.

10:10 AM Break

10:20 AM
Zener Pinning of a Nickel-Base Alloy: A Computational and Experimental Investigation: *Christopher Roberts*[1]; S. Lee Semiatin[2]; Anthony Rollett[1]; [1]Carnegie Mellon University; [2]Air Force Research Laboratory

The role of low volume fraction (0.2%) second phase particles in grain boundary pinning was examined. The wrought material, Waspaloy, was annealed isothermally at 1100C for various times to permit grain growth and pinning. A transition from normal grain growth to abnormal grain growth occurred at late times (>1h.) and was evident in the pinned microstructure which contained a bimodal grain size distribution. The fraction of particles located on the grain boundaries was found to be higher than the random case while a nonrandom correlation with grain boundary type was measured in the initial state; however, this correlation diminished after a one hour soak. Potts-based Monte Carlo grain growth simulations were conducted to evaluate the significance of the particle placement on the limiting grain size attained and subsequently compared to the original Zener prediction.

10:40 AM
A New Yield Criterion for Porous HCP Metals: *Oana Cazacu*[1]; J.S. Stewart[2]; [1]University of Florida/REEF; [2]Air Force Research Laboratory and University Florida/REEF

Hexagonal closed packed (hcp) polycrystals (e.g. Zr, Sn, Ti and their alloys) display plastic anisotropy and strong asymmetry between tension and compression, which is the result of the operation and interaction between crystallographic slip and deformation twinning. Despite recent progress, modeling damage of such materials remains a challenge. In this paper, we address modeling the effects of the tension-compression asymmetry of the matrix on void growth. A new plastic potential for porous randomly oriented hcp polycrystals is developed. It is shown that this criterion reduces to Gurson (1977) criterion if the matrix doesn't display strength differential effects (i.e. is described by the Von Mises yield criterion). The accuracy of the analytical criterion is assessed through comparison with results derived from numerical minimization procedure.

11:00 AM
Mechanical and Wear Properties of Copper-Lead Alloy Prepared by PM Processing: *Priyanka Dash*[1]; Anish Upadhyaya[1]; [1]Indian Institute of Technology - Kanpur

Cu-Pb alloys have no solubility in each other when they are in the solid state. However powder metallurgy processing techniques produces a more stable and homogeneous microstructure. In this work, water atomized Cu and premixed Cu-12Pb alloy powder were compacted and sintered at 500ºC, 700ºC and 900ºC and further characterized to evaluate their mechanical and tribological properties. The effect of sintering temperature on pore morphology and dimensional shrinkage were also investigated. It was observed that shrinkage in the direction of sintering was generally higher than that of the radial direction. There was a significant reduction in wear as Pb was added to Cu. Wear Test conducted at room temperature reveal that with increasing load the mechanism of wear also changes from oxidative to abrasive. At higher loads the wear is due to the rupture of the lead film resulting from plastic deformation, whereas at lower loads this film served as a lubricating layer to reduce the wear rate.

11:20 AM
The Effects of High-Intensity Ultrasonic Treatment on the Phase Morphology of Mg-9.0wt.%Al Binary Alloy: *Zhiqiang Zhang*[1]; [1]Key Laboratory of Electromagnetic Processing of Materials, Ministry of Education

Phase morphology is one of the most important microstructural characteristics determining the mechanical properties and therefore the application and service performance of metal components. The effects of ultrasonic treatment on the ß-phase(Mg17Al12) morphology of Mg-9.0wt.%Al alloy are investigated in this paper. The results show that the ß-phases in Mg-9.0wt.%Al alloy are significantly refined and discontinuously distributed throughout the sample when it was treated by high-intensity ultrasonic field during the solidification. After applying the ultrasonic field, spherical ß-phases were observed in the sample.

11:40 AM
Effect of Electric Current Pulse on Solidification Structure of Bearing Steel with Low Voltage and High Discharging Frequency: Jie Li[1]; Yulai Gao[1]; Jianhong Ma[1]; *Qijie Zhai*[1]; [1]Shanghai University

An experiment with respect to the influence of electric current pulse (ECP) on solidification structure of bearing steel with low voltage and high discharging frequency during solidification has been carried out. The results show that with the constant voltage and the pulse current peak, the primary dendritic arm shortened and the rate of equiaxed crystal increased with increasing the discharging frequency, but the secondary dendritic arm gets coarse. It is suggested that the transverse temperature gradient of ingot is reduced attributed to the the broadened mushy zone by the agitation of ECP, and meanwhile the distribution of solutes gets more homogeneous in virtue of interaction on electric and magnetic field, which also result in refinement of structure. In addition, the refined dendritic grains and the increased rate of equiaxed grain will obviously improve the central carbon segregation.

General Abstracts: Structural Materials Division: Structure/Property Relations

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Alloy Phases Committee, TMS: Biomaterials Committee, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Composite Materials Committee, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee, TMS: Product Metallurgy and Applications Committee, TMS: Refractory Metals Committee, TMS: Superconducting and Magnetic Materials Committee, TMS: Titanium Committee
Program Organizer: Ellen Cerreta, Los Alamos National Laboratory

Tuesday AM Room: 387
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Lisa Dougherty, Los Alamos National Laboratory; Subhadarshi Nayak, Intel Corporation

8:30 AM
Research Concerning the Structural Characterization of Hybrid Composite Materials: P. Moldovan[1]; *Gabriela Popescu*[1]; C. Popescu[1]; Brandusa Ghiban[1]; Oana Bajenaru[1]; [1]Polytechnic University of Bucharest

This paper's aim is to present the processing and characterization of some hybrid composite materials. The hybrids composite were realized using aluminum alloys as matrixes and silicon carbide and aluminum oxide particles as reinforcements. The ceramic particles were used in composite materials in different rates. Were analyzed the processes that take place at the interface between matrix and reinforcements, as well as the wetability of ceramic particles by the melt. Structural analyses of hybrid composites were realized using electron microscopy. The matrix nature and compounds have been investigated using an electron-probe microanalyzer.

8:45 AM
Quantitative Evaluation of Creep Curve of Alloy-Type and Metal-Type Creep in Magnesium-Aluminum Binary Alloys at around 0.6Tm: *Hiroyuki Sato*[1]; [1]Hirosaki University

Evaluation of creep curve is an important subject for life prediction of materials used in severe environment. Minimum creep rate is one of the major parameter used for characterization of deformation behavior of materials, but the values are evaluated from a part of creep curve. In this report, author propose new parameter that reflects shape of creep curve, analyze its stress dependence and compare with creep characteristics based on the minimum creep rates. Magnesium-aluminum alloys are one of the alloys whose creep characteristics are investigated and divided into classes with its stress dependence of minimum creep rates. The magnitude of strain-rate change before and after minimum strain rates are quantitatively evaluated and opposite stress dependence of the parameter in Alloy-type regime and Metal-type regime has been found. It is expected to improve precision of predicted lifetime of materials used in severe environment.

9:00 AM
Microstructures, Thermodynamic Modeling and Mechanical Properties in the Ti-Si-Sn Ternary System: *Jean-Claude Tedenac*[1]; Xaoyan Ma[1]; Nicolas Pradeilles[1]; Marina Bulanova[1]; Suzana Fries; [1]Institut C. Gerhardt-Universite de Montpellier 2

The determination of phase equilibria and microstructures of the alloys in the Ti-Si-Sn system were studied in as-cast and long-term annealed conditions by means of imaging with back scattered electrons (BSE) in a scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD). The isothermal sections at Ti-rich part in 25°C, 700°C and 1000°C have been experimentally established. Based on the present experimental results and the reliable literature data, a consistent set of thermodynamic parameters was obtained by means of CALPHAD method. The ternary interactions for Ti, Ti_3Sn, Ti_2Sn, Ti_5Sn_3 and Ti_5Si_3 phases were introduced in order to model the large solubility of Si in Ti-Sn alloys and Sn in Ti-Si alloys.

The isothermal sections and the liquidus surface calculated from the present thermodynamic description are in agreement with experiments. some resukts on mechanical properties have been obtained for titanium high content alloys.

9:15 AM
Structure-Property Correlations in Low Modulus Ti-Nb-Zr-Ta Alloys - the Role of Mechanical and Thermal History on Observed Macroscopic Properties: Srini Rajagopalan[1]; Gopal Viswanathan[1]; Rajarshi Banerjee[2]; *Soumya Nag*[1]; Hamish Fraser[1]; [1]Ohio State University; [2]University of North Texas

Recently developed Ti-Nb-Zr-Ta alloy systems exhibit several interesting mechanical properties, including low elastic moduli and high tensile strengths, of particular interest in biomedical and other structural applications. One particular aspect observed in these alloy systems is their ability to sustain significant amounts of deformation while experiencing a decrease in modulus. This paper explores the possible microstructural factors governing such observed mechanical behavior in two specific Ti-Nb-Zr-Ta based alloys. The role of prior mechanical history, such as cold-rolling, is discussed, in light of the apparently anomalous deformation behavior in certain compositions. The effect of thermal history, such as solutionizing heat treatments, on alloy microstructure, and correspondingly, deformation mechanisms, is also studied. Finally, an attempt is made to correlate these microstructural observations to observed macroscopic mechanical properties.

9:30 AM
Properties of Titanium Oxide Films Deposited by the Sol-Gel Technique: Pablo Favilla[1]; Miguel Alterach[2]; Mario Rosenberger[2]; Alicia Ares[2]; *Carlos Schvezov*[2]; [1]CEDIT/University de Misiones; [2]CONICET/University De Misiones

In the development of new material for use in mechanical heart valves there are conditions that must be satisfied. In particular for the present report the focus is in good surface quality, mechanical and corrosion resistance and biocompatibility for the intended period of use. The material object of this investigation is titanium oxide deposited on a substrate of Ti-6Al-4V. The oxide films were deposited by the sol-gel technique by dip-coating, withdrawing at a constant velocity, drying and thermal treatment. The films were characterized by optical and scanning microscopy observations and test of adherence. The adherence of the films were determined as a function of the number of layers deposited and also in relation with the surface morphology. The results show that the mono-layer films have much better adherence than the multilayer films and also, that the multilayer films presented small cracks during the different processing steps.

9:45 AM
Microstructural and Mechanical Properties of Alumina Coatings Produced by Microarc Oxidation Technique on 7039 Aluminum Alloy Substrate: Aytekin Polat[1]; Juhi Baxi[2]; Yusuf Kar[2]; Metin Usta[1]; Murat Makaraci[3]; Hong Liang[2]; *Ahmet Ucisik*[4]; [1]Gebze Institute of Technology; [2]Texas A&M University; [3]Kocaeli University; [4]Bogazici University

Aluminum alloy (7039) was coated using a microarc oxidation (MAO) process in an alkali solution. To very the coating's microstructure and thickness, various current densities and duration were used. The microstructure was studied using a scanning electron microscope and X-Ray Diffraction. Surface roughness was measured using a surface profilometer. Microhardness measurements on the coated layer, and tribological investigation in biofluids were also performed. The results showed that the coating consists of α-Al_2O_3, γ-Al_2O_3, and δ- Al_2O_3, and mullite phase. It was found that the high hardness and thickness are associated with the high concentration of α-Al_2O_3 resulted from the increased current density. MAO coatings typically exhibit micro hardness gradient across the coating thickness. Results also showed that the ticker the coating, the higher the surface roughness. Tribological experiments indicated that this coating is table and relatively wear resistant in synthetic biofluids.

10:00 AM Break

10:30 AM
Effect of Lattice Defects Induced by Hydrogen and Strain on Environmental Degradation: *Kenichi Takai*[1]; [1]Sophia University

Hydrogen absorbed during corrosion causes environmental degradation for mechanical properties. The principal factor of the degradation has been investigated using Inconel 625 for fcc metal and pure iron for bcc metal. Hydrogen content of Inconel 625 and pure iron applying constant strain with hydrogen is larger than that applying constant strain without hydrogen. This result suggests that hydrogen and strain induced formation of lattice defects in metals. When Inconel 625 and pure iron were applied constant strain with hydrogen and released hydrogen at 303 K and 473 K, the ductility does not recover at 303 K regardless of no hydrogen, in contrast, recovers at 473 K. This result suggests that the principal factor of hydrogen degradation is not hydrogen, but vacancy and vacancy cluster induced by hydrogen and strain.

10:45 AM
Micro-Structural Characterization and Fatigue Life Variability of a Ni-Base Single Crystal PWA1484: *Kezhong Li*[1]; Willam Porter[1]; Ryan Morrissey[1]; [1]University of Dayton Research Institute

Research has shown that fatigue life variability is closely associated with material response in the presence of small (<100 micron) fatigue cracks. In this project, the effect of elemental segregation on fatigue variability in the cast, single crystal superalloy PWA1484 is of interest. To that end, quantitative Electron Probe Micro Analysis (EPMA) and x-ray mapping were employed to determine the extent of elemental segregation in the dendrite core and interdendritic region in the hot isostatically pressed castings. The location of fatigue initiation sites and adjacent material relative to the EPMA maps was investigated to understand the effect of elemental segregation on variability in fatigue behavior.

11:00 AM
Microstructural Stability in Grain Boundary Engineered Materials: *Scott Schlegel*[1]; Megan Frary[1]; [1]Boise State University

Grain boundary engineering (GBE) is a thermomechanical process in which sequential straining and annealing cycles are used to increase the fraction of special, low-energy grain boundaries (with $\Sigma \leq 29$ according to the coincidence site lattice model). In the present work, two cubic face-centered materials, pure copper and Inconel 617, were processed by GBE. In addition, a conventionally-processed sample was processed with a single strain step, equal to the total strain in the GBE sample. The thermomecanically-processed samples were subjected to elevated temperatures for varying times. The GBE samples exhibited a resistance to change in the fraction of special boundaries and grain size (as determined by Orientation Imaging Microscopy (OIM)), while the conventionally-processed samples experienced abnormal grain growth. Monte Carlo grain growth simulations on the OIM-determined microstructures confirm the increased microstructural stability of the GBE samples. Therefore, GBE processing can produce more stable and predictable microstructures than can conventional processing.

11:15 AM
Microstructure and Strengthening Processes of Alloys for Next Generation Nuclear Plants: *Liu Liu*[1]; Deepak Kumar[1]; Raghav R. Adharapurapu[1]; Chris Torbet[1]; Gary Was[1]; Tresa Pollock[1]; J. Wayne Jones[1]; [1]University of Michigan, Material Science and Engineering

Structural materials for the next generation nuclear power plant will require microstructure stability, creep resistance and corrosion/oxidation resistance in impure helium environments at temperatures as high as 1000°C. Here, a preliminary alloy development study is described, based on modifications to Alloy 617. Compositional modifications were made that provide potential for enhanced solid solution strengthening and lower interdiffusion rates for improved creep resistance. Elevated temperature deformation behavior and environment attack in various impure helium environments is described. Creep behavior, including damage accumulation processes in candidate alloys in impure helium environments that promote carburization/decarburization or oxidation is also described. Microstructure stability under long time and its influence on long time creep resistance are examined.

11:30 AM
Brittle Crack Susceptibility of Low Temperature Separator Vessel Material: *Ahmad Nawaz*[1]; [1]GIK Institute

Low temperature separator vessel made from HSLA steel grade A516-70, is used in gas sector following the JT valve or the turbo-expander. Being at very low temperature (-7°F to -13°F), it is susceptible to brittle fracture due to ductile to brittle transition. The influence of temperature on the failure behavior of this material has been studied. Moreover, since the low temperature separator is manufactured from welded sheets, therefore, affect of temperature on the failure behavior on the post weld heat treated (PWHT) sample have also been carried out. In particular, SEM examination revealed the type of fracture that occurred in the parent metal as well as PWHT sample. A significant increase in the impact toughness of the PWHT samples as compared to the parent metal was observed which was explained by optical micrographs as well as SEM examination.

Hael Mughrabi Honorary Symposium: Plasticity, Failure and Fatigue in Structural Materials - from Macro to Nano: Dislocations: Work Hardening, Patterning, Size Effects II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: K. Jimmy Hsia, University of Illinois; Mathias Göken, Universitaet Erlangen-Nuernberg; Tresa Pollock, University of Michigan - Ann Arbor; Pedro Dolabella Portella, Federal Institute for Materials Research and Testing; Neville Moody, Sandia National Laboratories

Tuesday AM — Room: 386
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Michael Kassner, University of Southern California; Ladislas Kubin, CNRS-ONERA

8:30 AM Keynote
A New Model for Dynamic Recovery in Stage III Deformation of FCC Metals: *Ali Argon*[1]; [1]Massachusetts Institute of Technology

In the Seeger model of 1956, dynamic recovery in Stage-III deformation of FCC metals is controlled by massive cross slip of screw dislocations, circumventing linear LC dislocation barriers and resulting in decreasing strain hardening rates. There is little evidence for this mechanism which is also quite inconsistent with the cellular dislocation microstructure of Stage- III strain hardening. In a proposed new mechanism dynamic recovery results from the systematic removal of LC locks in cell walls under the repeated impingement of dislocation fluxes that, trigger associated collapse events in the pinned, redundant cell wall dislocation microstructure. This, permits the flow-noise-induced cell refinement process of Haehner and Zeiser, to develop unhindered, forming dislocation cells with fractal characteristics. The model correctly predicts the onset of dynamic recovery, its temperature dependence, as well as the temperature and stress dependence of the decreasing strain hardening rate of Stage-III, commonly referred to as the Voce Law.

9:00 AM Invited
Plastic Flow in Confined Volumes: *Nasr Ghoniem*[1]; [1]University of California, Los Angeles

Characteristics of plastic deformation of metals are strongly dependent on the underlying microstructure. Moreover, when dislocations are confined to move in small volumes, the yield strength and hardening rate in monotonic deformation and the fatigue strength under cyclic loading can be significantly different from corresponding conditions where dislocations are not confined. In this talk, we discuss results of dislocation dynamics simulations of plasticity in small and confined volumes. This includes plastic deformation in nano-scale anisotropic multi-layered structures, where confinement is achieved through elastic modulus mismatch, plastic flow in micron-size single crystals, where confinement is attained through free surfaces, and the confined motion of dislocations in Persistent Slip Band (PSB) channels, where confinement is determined by dislocation interaction with dipolar arrays under cyclic loading conditions.

9:20 AM Invited
Plastic Shear Stress-Strain Behaviour of Axially Oriented Microstructures: *Javier Gil-Sevillano*[1]; Isabel Gutiérrez[1]; [1]CEIT and TECNUN, University of Navarra

The torsional stress-strain response of several disparate axially oriented two-phase materials displays peculiar plastic transient behaviours. The response can be rationalised on account of the superposition of effects of both the instantaneous structural orientation of the material during the test and the relative strength of the two phases and their interface. Understanding this torsional behaviour provides hints for explaining observations of the behaviour of layered materials in plane strain modes easily accommodated by a composition of simple shears (shear kinking).

9:40 AM Invited
Size-Affected Exhaustion Hardening and the Correlation Length for Glide Resistance in Nickel Microcrystals: *Dennis Dimiduk*[1]; Michael Uchic[1]; Michael Mills[2]; Satish Rao[3]; David Norfleet[2]; Triplicane Parthasarathy[3]; [1]US Air Force; [2]Ohio State University; [3]UES Inc

Modeling the strength of small material volume elements that deform plastically by dislocation glide implicitly brings the question: what is the correlation length for the glide resistance of the dislocation ensemble (i.e. the characteristic dislocation length for which the intrinsically-stochastic mobile dislocation segment lengths act locally independently to defeat the strongest obstacles to glide)? A related question is what minimum number of locally-independent dislocation features must be sampled over the deforming volume to represent continuum behavior for the dislocation ensemble? There are historical deficiencies in addressing these questions which, in part, lead to the current active interest in strain-gradient plasticity mechanics, the statistical mechanics of dislocation ensembles, intermittency of flow and the myriad "size-effects" on plastic flow. Our studies of the effect of sample size on flow stress of single crystals show a systematic change in the transition from elastic to plastic flow with decreasing sample size. Analysis of those results suggest both a dislocation source-hardening effect and, a period of plastic straining that exhibits an unusually high strain-hardening rate called exhaustion hardening. This study reports results from both discrete dislocation simulations and TEM examinations of deformed microcrystals. In both studies efforts have been made to tie dislocation micromechanisms to the phenomenology found in small-strain flow of nickel microcrystals. The results are discussed in the context of a correlation length for glide resistance and its influence on size effects.

10:00 AM
Fracture of Edge Dislocation Cores during Fatigue Failures: *John Gilman*[1]; [1]University of California - Los Angeles

Since numerous materials obey Coffin's Law of fatigue failure not only during cyclic stressing, but also for the initial quarter cycle (tenson test), the intrinsic cause of failure seems to be only secondarily associated with repeated cycling. If this conjecture is true, something that is present during the initial plastic deformation must lead to the final fracture. The only culprits present are individual dislocations, dipoles, and higher multipoles; i.e., dislocations themselves. In fact, the cores of edge dislocations are sources of weakness. The author has shown this in: bubble rafts; prestrained LiF crystals; and bent zinc crystals. Others have demonstrated the weakening effect of plastic torsion on fracture. The local weakening effect is estimated to be about 30%. Griffith's fracture condition demands a high stress to convert these incipient cracks into running cracks, but as dislocations accumulate in persistent glide bands, the critical stress progressively decreases.

10:15 AM Break

10:25 AM Invited
Recent Experimental Developments in Examining Long Range Internal Stresses: *Michael Kassner*[1]; Lyle Levine[2]; Ben Larson[3]; Jon Tischler[4]; Peter Geantil[1]; [1]University of Southern California; [2]National Institute of Standards and Technology; [3]Oak Ridge National Laboratory; [4]Argonne National Laboratory

Hael Mughrabi was pioneering in advancing the concept of long-range Internal stresses (LRIS) in deformed microstructures. Evidence for such long-range internal stresses included the famous dislocation pinning by neutron irradiation experiments by Mughrabi, and x-ray line broadening by Mughrabi and colleagues. Experiments by others, including some relatively recent dipole-separation observations and convergent beam electron diffraction experiments in connection with LRIS, will be described as well. Most recently, the concept andthe interpretation of long-range internal stress was investigated using advanced x-ray microbeam diffraction on a synchrotron source; axial strains within individual cell interiors have been measured and measurements of strain in individual cell walls are in progress. These were accomplished using oriented monotonically deformed Cu single crystals. The results demonstrate that long-range internal stresses are consistent with the earlier findings of Professor Mughrabi. These recent synchrotron experiments are discussed in greater detail in another talk in the Symposium.

10:45 AM Invited
Slip Circle Constructions for Plasticity of Polycrystals: *Lawrence Brown*[1]; [1]University of Cambridge

Patterns of plastic flow around undeformable particles, and at hardness indenters, can be understood by rotational flow which transfers matter from where it is in excess to where it is in deficit. Such slip-circle constructions are a simple way of treating the operation of antisymmetrical combinations of slip systems. The constructions predict lattice rotations, geometrically necessary dislocation densities, and any size effects. Applied to flow in polycrystals, they predict a Hall-Petch law dependent upon the root-mean-square dispersion of Schmidt factors for the array of grains. They also predict residual stresses, and allow an estimate to be made of the maximum uniaxial flow stress sustainable by the polycrystal without loss of cohesion at the grain boundaries. In all cases, there should be a transition from laminar to rotational flow at small plastic strains and at small grain sizes, particularly in nanocrystalline materials.

11:05 AM Invited
Properties of Dislocations under Single Slip: *Patrick Veyssiere*[1]; [1]Laboratoire d'Etude des Microstructures, Centre National de la Recherche Scientifique-Onera

Dislocations of a given slip system engender obstacles to their own propagation forming entanglements. This property, essential in fatigue, has received renewed interest with the development of computer simulation experiments and with investigations of single crystal pillars but little is known on local mechanism. The talk concentrates on selected aspects related to self-organization. Namely: 1 – Some properties of dislocation contrast are re-visited. 2 – The model for the formation of loops and the further sweeping of these by mobile dislocations is confirmed experimentally by dislocation dynamics and MD simulations. 3 – Properties of dipoles are analyzed under isotropic and anisotropic elasticity in cubic systems. The passing stress under constrained deformation conditions, such as fatigue, is discussed.

11:25 AM
Local Shear Strain Amplitude and Half-Cycle Slip Activity of Persistent Slip Bands in Nickel Polycrystals: *Anja Weidner*[1]; Wolfgang Tirschler[1]; Christian Blochwitz[1]; Werner Skrotzki[1]; Jiri Man[2]; Tomas Kruml[2]; [1]Technical University of Dresden; [2]Academy of Science of Czech Republic

The local slip activity of persistent slip bands (PSBs) was studied after half-cycle deformation on a well-polished specimen surface at different stages of fatigue life using atomic force microscopy (AFM) and scanning electron microscopy (SEM). In-situ deformation experiments were carried out in order to investigate both the formation of slip steps during half-cycle loading as well as the development of extrusions with increasing number of cycles. It is shown that during half-cycle loading the slip steps do not develop proportionally to the applied plastic strain and not the whole accumulated PSB volume is active. Additionally, the half-cycle slip activity of PSBs depends on the stages of fatigue life. The local shear strain amplitudes of PSBs in polycrystals are comparable to those for single crystals and are nearly independent on the stage of fatigue life. PSBs in polycrystals have a development history which is characterized by active and inactive periods.

11:40 AM
Relationship between Dislocation Microstructure and Flow Stress during Cyclic Deformation in Al Single Crystals: *Toshiyuki Fujii*[1]; Shizuma Uju[1]; Hiroyuki Tanaka[1]; Chihiro Watanabe[2]; Susumu Onaka[1]; Masaharu Kato[1]; [1]Tokyo Institute of Technology; [2]Kanazawa University

Al single crystals with a multiple slip orientation and Al-0.7mass%Mg solid-solutioned alloy single crystals with a single slip orientation were cyclically deformed under constant plastic-strain amplitudes at room temperature. A sequential change, i.e. initial hardening, intermediate softening, and secondary hardening, was obtained in cyclic hardening curves for Al. The dislocation microstructure for Al was characterized by the labyrinth structure, which consists of two sets of mutually perpendicular {100} walls. On the other hand, cyclic hardening to saturation was obtained in the Al-Mg single crystals, and the cyclic stress-strain curve (CSSC) showed a plateau with the stress of 26MPa. At strain amplitudes within the plateau region of the CSSC, persistent slip bands with ladder structure were formed. The relationship between the macroscopic cyclic flow stress and the dislocation microstructure can be explained in both the materials by an analysis based upon Mughrabi's composite model.

11:55 AM
Modeling of Large Strain Deformation and Failure Modes in Crystalline Aggregates: Omid Rezvanian[1]; Jibin Shi[1]; *Mohammed Zikry*[1]; [1]North Carolina State University

The objective of this research is predict failure modes and scenarios at different physical scales that occur due to a myriad of microstructural factors, such as texture, grain size and shape, grain subdivision, heterogeneous microstructures, and grain boundary misorientations and distributions. The microstructurally-based formulation for inelastic deformation is based on coupling a multiple-slip crystal plasticity formulation to three distinct dislocation densities, which pertain to statistically stored dislocations (SSDs), geometrically necessary dislocations (GNDs), and grain boundary dislocations (GBDs). This dislocation density based multiple-slip crystal plasticity formulation is then coupled to specialized finite-element methods to predict how scale-dependent microstructural behavior and the evolving heterogeneous microstructure can contribute to failure initiation. Furthermore, a clear understanding of how GB strength changes due to microstructural evolution is obtained as a function of microstructural heterogeneities and dislocation-density transmission, blockage, and absorption.

12:10 PM
An Analysis of Geometrically Necessary Dislocation Evolution and Plasticity Size Effects in Micro-Crystals: *P. J. Guruprasad*[1]; A. Benzerga[1]; [1]Texas A&M University

Mechanism-based discrete dislocation dynamics is used to study the plastic behavior of micron scale single crystals under compression. Calculations were carried out for initially high (HSD) and low (LSD) dislocation source density. The results for both cases predict strengthening upon scale reduction. Significant work hardening was seen only in the HSD case. This was attributed to the emergence of a GND density locally, aided by lattice rotations associated with dislocation pattern formation. The net build-up of GND density leads to a strong Bauschinger effect in the smallest specimens. By way of contrast, strengthening in the LSD case was related to the dislocation source length distribution. Occasionally, a yield-strength asymmetry between tension and compression was observed and attributed to the initial dislocation structure. In addition, strain-rate sensitivity analyses indicate a significant size effect in the HSD case down to a strain rate of 10/s.

12:25 PM
Atomistic Simulations of Dislocation Nucleation in Copper Grain Boundaries under Uniaxial Tension and Compression: *Garritt Tucker*[1]; Mark Tschopp[2]; David McDowell[1]; [1]Georgia Institute of Technology; [2]Air Force Research Laboratories/Universal Technology Corporation

Atomistic simulations are used to investigate how grain boundary structure influences dislocation nucleation under uniaxial tension and compression for a specific class of symmetric tilt grain boundaries that contain the E structural unit. After obtaining the minimum energy grain boundary structure, molecular dynamics was employed based on an embedded-atom method potential for Cu at 10 K. Simulation results show that higher nucleation stresses are required in uniaxial compression than in tension. Additionally, analysis of the dislocation nucleation mechanisms show several differences between tension and compression. For instance, partial dislocations are nucleated in tension and full dislocations are nucleated in compression. The tension-compression asymmetry in mechanisms and responses can be partially explained by the resolved stress components on the slip plane on which the dislocation nucleates.

Hume-Rothery Symposium - Nanoscale Phases: Session III

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Sinn-wen Chen, National Tsing Hua University; David Cockayne, University of Oxford; Seiji Isoda, Kyoto University; Robert Nemanich, Arizona State University; K.-N. Tu, University of California, Los Angeles

Tuesday AM — Room: 276
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: David Cockayne, University of Oxford; Sinn-wen Chen, National Tsing Hua University

8:30 AM Invited
Structure and Composition Effect of (Co, Fe) Electrode on the Tunneling Magnetoresistance of AlO_x Based Magnetic Tunnel Junction: *Y. Chang*[1]; [1]University of Wisconsin

A direct experimental evidence of the crystal structure effect on the tunneling magnetoresistance (TMR) was observed. The TMR of AlO_x based magnetic tunnel junction (MTJ) with (Co, Fe) electrodes increased from 32.1% to 46.7% when the crystal structure of the $Co_{87}Fe_{13}$ bottom electrode is altered from fcc to bcc by using Cu and Cr buffer, respectively. This increase was attributed to the electronic structure change of the FM electrodes, as confirmed by *ab initio* calculation that spin polarization of bulk bcc is greater than bulk fcc. A compositional dependence of TMR was performed for AlO_x and (Co, Fe) based MTJs. The experimental results combined with *ab initio* calculations suggest that TMR is mainly determined by the s-like electron spin polarization values associated with different compositions and crystal structures. By further optimizing the crystal structure, interface roughness of the bottom electrode, more than 70% TMR was achieved at room temperature.

8:55 AM Invited
Alloying Events in Three-Layer Au/Pd Nanoparticles: *Miguel Yacaman*[1]; Domingo Ferrer[1]; Sergio Mejia[2]; Eduardo Perez-Tijerina[2]; Ubaldo Ortiz[2]; [1]University of Texas; [2]Universidad Autonoma de Nuevo Leon

High-angle annular dark field (HAADF) imaging in the electron microscope has been exploited to reveal the internal structure of bimetallic palladium-gold nanoparticles, prepared using a polyol-mediated wet-chemical method and protected with poly(Nvinyl2-pyrrolidone) (PVP). Our observations revealed a three-layer structure in the domains of the bimetallic nanoparticles, exhibiting core-shell morphology. New insights were gained on the composition of the domains that involve intimate alloy of the monometallic elements.

9:20 AM Invited
The Structure of Amorphous Nanovolumes: *David Cockayne*[1]; Konstantin Borisenko[1]; Guoqiang Li[1]; Yixin Chen[1]; [1]University of Oxford

The structure of amorphous materials is traditionally investigated using neutron or X-ray diffraction, including refining models against the diffraction data. However these techniques are not able to study nanovolumes of amorphous material, such as in thin films, rapid phase change "bits", intergranular films and insulating layers in devices. We have developed electron diffraction techniques which enable model structures to be refined against the experimental data for both elemental and alloy amorphous nanovolumes.

9:45 AM Invited
d-States in Complex Metallic Alloys: Jean-Marie Dubois[1]; *Esther Belin-Ferré*[2]; [1]Institut Jean Lamour; [2]Centre National de la Recherche Scientifique

Many complex alloys exist in metal systems like Al-Mg and Al-Mg-Zn or Al-Metal and Mg-Metal systems. Their electronic structure was investigated using X-ray emission spectroscopy technique, which provides separately the energy distribution of electronic states around each of the chemical species in the solid, averaging the data over their various sites. In this talk, we examine and discuss the Al 3s,d and Mg 3s,d electronic distributions of several complex alloys in comparison to the pure metals as well as to other simpler structures. In complex systems, the contribution of Al and (or) Mg d-like states is important at the Fermi level and in its close vicinity. By comparison to simple alloys containing a transition metal, it is suggested that Al or Mg d-like states play a significant role in the formation of a pseudo-gap at the Fermi level and therefore contribute to select and stabilize the complex structures.

10:10 AM Break

10:30 AM Invited
Shape Controlled Nanocrystals of Ceria and Platinum–Synthesis and Growth Mechanism: *Zhong Lin (Z.L.) Wang*[1]; [1]Georgia Tech

Two examples will be presented about shape control of nanocrystals. The first example is about the formation of spherical ceria nanocrystals. For chemical-mechanical planarization of advanced integrated circuits, the polyhedral shaped nanoparticles scratch the silicon wafers and increase defect concentrations. We present here an innovative approach for large-scale synthesis of single-crystal ceria nanospheres,[2] which can reduce the polishing defects by 80% and increase the silica removal rate by 50%. The second example is about shape controlled Pt nano nanocrystals.[3] Platinum NCs of very unusual tetrahexahedral (THH) shape were prepared at high yield by an electrochemical treatment of Pt nanospheres supported on glassy carbon by a square-wave potential, which has a much improved catalytic properties. [1]X.D. Feng, D.C. Sayle, Z.L. Wang et al., Science, 312, 1504 (2006). [2]Na Tian, Zhi-You Zhou, Shi-Gang Sun, Yong Ding, and Zhong Lin Wang, Science, 316, 732 (2007).

10:55 AM Invited
Nanocrystallization of Bulk Amorphous Alloys in a Magnetic Field: *James Li*[1]; [1]University of Rochester

For equilibrium in an externally applied magnetic field, the work done by the magnetic field should be considered. As a simple mechanical example, let a dead weight W be hung at the end of a vertical spring of length l (original length lo) with the other end fixed. The equilibrium is described by minimizing a free energy function (k/2)(l-lo)^2-W(l-lo) where k is the spring constant. The term Wl is similar to PV (pressure, volume) in the Gibbs free energy for equilibrium under constant pressure. So we need a term BH in the free energy expression for equilibrium under externally applied magnetic field H where B is the magnetic flux. This term has been neglected in the literature. By using this free energy expression, the enhancement or retardation of the nucleation or growth of crystals from amorphous solids in a magnetic field can be understood.

11:20 AM Invited
Properties of ZnO/SnO_2-Cosubstituted In_2O_3 Thin Films: *Robert Chang*[1]; M. Zhang[1]; D. Buchholz[1]; [1]Northwestern University

In indium tin oxide (ITO), indium is present as the In^{3+} ion, thus two atoms of indium can be replaced by a zinc atom, which forms a Zn_2+ ion and a tin atom, which forms a Sn^{4+} ion. In this manner up to 40% of the indium can be replaced by co-substitution of zinc and tin to form a new and less expensive TCO, zinc-indium-tin-oxide (ZITO). $Zn_{0.3}In_{1.4}Sn_{0.3}O_3$-δ (ZITO) thin films were grown by computer-controlled pulsed laser deposition (PLD) on (0001) Al_2O_3 substrate both at room temperature and at high temperatures (>700°C). The room temperature grown samples were found to be amorphous. Electrical measurements indicate that conductivities for room temperature and high temperatures were 1500 S/cm and 4000 S/cm respectively. Cross-section and plan-view transmission electron microscopy (TEM) revealed that the ZITO films grown at high temperatures were composed of twin-related domains with a lattice mismatch of approximately 2.8%.

IOMMMS Global Materials Forum 2008: Creating the Future MS&E Professional

Sponsored by: The Minerals, Metals and Materials Society, International Organization of Materials, Metals and Minerals Societies
Program Organizer: Robert Shull, National Institute of Standards and Technology

Tuesday AM — Room: 272
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Robert Shull, National Institute of Standards and Technology; Yoshimasa Kajiwara, Japan Institute of Metals

8:30 AM Introductory Comments by Dr. Robert Shull, TMS President and Dr. Yoshimasa Kajiwara, JIM, and Chair of IOMMMS

8:40 AM Keynote
International Perspectives on the Creation of Engineers for the 21st Century: *Norman Fortenberry*[1]; [1]National Academy of Engineering

Concerns about the quality and quantity of engineers to meet the needs of the 21st Century have emerged in multiple countries. These concerns are driven by increased competition in tightly coupled global economies as well as concerns about maintaining and enhancing domestic standards of living in increasingly technological societies. In more industrialized economies there are also concerns about being able to maintain the competitiveness of engineering as a career field when other avenues for personal and social progression are presenting themselves to tradition entrants into the field. This presentation summarizes the global context for engineering practice, reviews commonly cited global requirements for 21st Century engineers, and suggests nationally collaborative effort that might be pursued in order to enhance their attainment. The suggested collaborations are grounded in the formal educational preparation of engineers, but take advantage of emerging uses of information technologies as well as better utilization of existing collaborative mechanisms.

9:10 AM Invited
Requirement of Technical Professionals in the Indian Steel Industry over the Next Two Decades: *Amit Chatterjee*[1]; [1]Tata Iron and Steel Company Ltd

The Indian steel industry is going through a complete metamorphosis. Though only 40 million tonnes of capacity has been added in 60 years after Independence, it is foreseen that at least 150 Mt, if not 175 Mt additional capacity will be created in India before 2020. In fact, the projects that have already been announced envisage production of around 200 Mt by this time. Given this background, it is important that India prepares for meeting the requirements of the steel industry as far as Engineering Graduates and other professionals like Diploma holders and Trade Apprentices are concerned. The supply position is extremely bleak going by the present trend. Skilled and trained personnel are difficult to find. Diploma holders of the right quality are also not available. In the case of Engineering Graduates, even if the total number is satisfactory, they are not keen to join the steel industry and are attracted by lucrative options like the IT sector. Hence, innovations are required to attract professionals to the steel industry in future. Time is running out and an immediate action plan is called for. The paper will focus on these issues.

9:30 AM Invited
The Constitution and Practice for Training the Comprehensively Qualified Students in the Field of Materials Science and Engineering: *Xinxin Zhang*[1]; [1]University of Science and Technology

The discipline of materials science and engineering is developed on the basis of the mutual overlapping and integration from all those disciplines such as chemical metallurgy, physical metallurgy and mechanical metallurgy. The development started since the 60s of last century. The most obvious change is the research orientation transformation from the traditional focus on iron, steel, ceramics and plastics to the preparation and synthesis of advanced materials. Especially in the last 20 years, with the investigation and application of magnetic materials, high temperature superconductors, hydrogen storage materials, as well as nano materials and bulk glasses which are drawing more and more attention since last 10 years, many inherent concepts and ideas are broken through. The revolutionary progresses and the epoch-making achievements are appearing in the field of materials science and engineering. All these accomplishments are obtained and promoted by the scientists stepping into the materials field from physics and chemistry, and even working together with those in the fields of mining and metallurgy, chemical engineering and machinery fabrication etc. Under this new situation, we are trying to adapt teaching principles and improve courses offered. A new education system is established preliminarily for training such students with solid basis, self-study independency, practicing ability and innovative consciousness so that to meet the need of the development of society, economy, science and technology.

9:50 AM Invited
Materials Science and Engineering Education: Indian Perspectives: *Srinivasa Ranganathan*[1]; [1]Indian Institute of Science

The first university of the world was founded at Taxila closely followed by another in Nalanda in India. They had an international character drawing students from China and other nations. Due to historical forces there was a long decline over the past five hundred years in the educational sector, reversed only in the past century with the establishment of national universitiies in Bangalore and Varanasi. Metallurgical education began in 1923, materials science courses were started in 1964 and nanoscience courses were started in the early years of this century. A dochotomy has arisen as the demands of the Indian industry with explosive growth in steel and aluminium production are not responded to by the education sector. Globalization has had its own impact. Some prescriptions for addressing the gap in supply and demand of human resources will be advanced, as the Indian economy continues its onward march.

10:10 AM Break

10:25 AM Invited
Some Effective and Innovative Educational Efforts for Materials Engineers: *Chester Van Tyne*[1]; [1]Colorado School of Mines

The measure of an effective engineering education is the ability of graduates to perform well as engineers upon completion of their degree program. ABET is the accrediting agency in the US that evaluates engineering programs to determine if this goal is being achieved. ABET accrediting activities are often viewed with apprehension and skepticism by faculty and universities. One of the reasons that these reviews are perceived negatively is that the focus is often on the shortcomings that exist within the engineering program being accredited. The ABET review also provides an opportunity for the evaluator to comment on the program strengths. These strengths encompass a number of innovative and effective methods for educating future materials engineers. A summary of the strengths found in materials program over the last four years will be presented. Additionally, several unique educational opportunities that have been introduced by programs and universities will also be briefly described.

10:45 AM Invited
Education for Non-Ferrous Metallurgy in Japan—The Present Issues and the Future: *Takahiko Okura*[1]; Susumu Okabe[2]; Takashi Nakamura[3]; [1]Akita University; [2]The Mining and Materials Processing Institute of Japan; [3]Tohoku University

Japanese non-ferrous industry is producing environmental-soundly 9% of the world copper production, 6% of zinc, and 3% of lead, which account some 0.5% of GDP. The industry is receiving approximately 25 new graduates from universities per year these days, but it needs more in order to sustain its business in Japan as well as to develop it in the global market. However, the industry is faced with the crisis of less-educated graduates and its less supply. Since the aging of the society in this field is going on and the mind of professors and students tend to decline into so-called high technology, the lectures and researches in the field are becoming less and less in Japanese universities. It will be difficult to change these diverse challenges and to again increase the lectures and research themes as same as in the old days. In the presentation, such situations in Japan are reported in detail, and the recent countermeasure for the new century, developed and operated by universities, the industry, and the government collaboratively, will be discussed, including 3 subjects, (1) continuing education program (2) donated chair at a university, (3) Collaborative educations for Japanese and Asian engineers in non-ferrous metallurgy and recycling.

11:05 AM Invited
Current Topics on Continuous Education and Training of Professional Material Engineers in Japan: *Shu Yamaguchi*[1]; [1]University of Tokyo

Recent development of education and training of professional and experienced engineers at academic society, university, and other organizations in Japan will be reviewed and summarized in addition to the recent Governmental policy and support in cultivation of experienced engineers to support industry.

11:25 AM Invited
Online Materials Education Communities: Transforming More Than Just Content Delivery: *Adam Powell*[1]; [1]Veryst Engineering LLC

The World-Wide Web provides educators with much more than a means for delivering educational content. It has facilitated new types of visual and interactive content for improved educational effectiveness. It has changed the culture of distribution, from a profit motive to an ethic of sharing everything with everyone, from Wikipedia to MIT Open CourseWare. And it has facilitated collaboration in content development: on the web, everyone has thousands of proofreaders and a hundred potential collaborators. The resulting overwhelming cacophony of opportunities and resources requires new means of sorting and evaluating them for classroom use. Toward that end, Materials Technology@TMS provides a sorted collection of nearly 300 links to pre-screened resources, along with discussion forums where educators can share experiences on using resources and developing curricula. While centered around the MT@TMS Education Community, this talk hopes to provoke thoughts and discussion on the production and dissemination of educational content in general.

Magnesium Technology 2008: Thermodynamics and Phase Transformations

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Mihriban Pekguleryuz, McGill University; Neale Neelameggham, US Magnesium LLC; Randy Beals, Chrysler LLC; Eric Nyberg, Pacific Northwest National Laboratory

Tuesday AM — Room: 292
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Rainer Schmid-Fetzer, Technische Universität Clausthal; Luke Mackenzie, Novelis

8:30 AM
Elastic Constants of Magnesium Compounds from First-Principles Calculations: *Swetha Ganeshan*[1]; Hui Zhang[1]; Shunli Shang[1]; Yi Wang[1]; Zi-Kui Liu[1]; [1]Pennsylvania State University

The deformation behaviors of second phases are important in understanding the mechanical properties of Magnesium alloys. In the present work, the elastic constants of Mg based binary compounds have been predicted from first principles calculations using the stress strain method[1]. The Mg-X systems that have been investigated in the present work include Mg-As, -Ba, -Ca, -Cd, -Cu, -Dy, -Ga, -Ge, -La, -Lu, -Ni, -Pb, -Sb, -Si, -Sn and -Y. A comparative study between the results obtained in the current work and the available experimental data has been made. The accuracy in the calculated data sets off a landmark to use the same for any future work incorporating the design of Mg based alloys. [1]S. Shang, Y. Wang, and Z. K. Liu, "First-principles elastic constants of α- and θ-Al_2O_3", Appl. Phys. Lett., Vol. 90, 2007, 101909.

8:50 AM
The Role of Solutes for Grain Refinement by $(SiC)_P$: Experiment and Theoretical Calculation: *Robert Günther*[1]; Christian Hartig[1]; Okechukwu Anopuo[2]; Norbert Hort[2]; Rüdiger Bormann[1]; [1]Hamburg University of Technology; [2]GKSS Research Centre

Grain refiners for Al-containing Mg-alloys are still deficient and under investigation. To clarify the mechanisms of grain refinement by inoculation a simulation method for heterogeneous nucleation has been used, allowing the prediction of the grain size for a given particle size distribution, volumetric content of inoculants, cooling rate and alloy constitution. To proof the model, grain refinement efficiency of $(SiC)_P$ in Mg3wt%Al was examined experimentally. It was found that $(SiC)_P$ results in significant grain refinement. Experiments with $(SiC)_P$ in Al-free Mg-alloys have shown that additionally to the low lattice misfit of SiC, solutes play an important role for effective grain refinement. While the grain refinement via $(SiC)_P$ inoculation of pure Mg is negligible, a distinct effect has been achieved in Mg3wt%Zn. The model makes quantitatively correct predictions for the grain size and its variation with cooling rate. Relevant aspects for the consideration of particle-specific properties and solutes will be discussed.

9:10 AM
Thermodynamic Database of Mg-Al-Ca-Sr: A Resource for Alloy Development and Improvement: *Hongbo Cao*[1]; Jun Zhu[1]; Chuan Zhang[2]; Y. Chang[1]; [1]University of Wisconsin; [2]Universtiy of Wisconsin

We developed a thermodynamic database of Mg-Al-Ca-Sr, an important base system for a number of current magnesium alloys such as AXJ530. Our database focuses primarily on the Mg-rich phase equilibria. Using the Scheil model, the calculated solidification paths of several Mg-Al-Ca-Sr alloys are in accord with the reported data on the as-cast microstructure. Since the database was obtained from the constituent key ternaries modeled in terms of data and other ternaries obtained via extrapolation from the binaries, we believe that it is equally valid for other Mg-rich alloys containing Al, Ca and Sr. Additionally, we have calculated the equilibrium state of AXJ530 alloy at low temperature and found that the stable phases calculated agree with literature data. More importantly, the predicative nature of the calculation using either only thermodynamic database or coupled with kinetic model makes it possible to exploit new alloy compositions where experimental investigation has not been attempted.

9:30 AM
Thermodynamic Modeling of Porosity Formation during Non-Equilibrium Solidification in Magnesium Alloy Castings: *John Li*[1]; Jeff Wood[1]; John Auld[2]; Gerry Wang[3]; [1]University of Western Ontario; [2]Meridian Technologies Inc.; [3]Meridian Technologies Inc

Casting properties are mainly controlled by filling and solidification related defects. Due to the complex nature of flow and thermal process, the ability to accurately predict the solidification porosity is limited. Although commercial solvers are available to predict solidification hot spots, there is no established math model able to quantitatively predict the solidification porosity for magnesium alloy castings. This is because commercial solvers for porosity prediction are only based on the classic Niyama Criteria. In this research project, a new porosity model for magnesium alloy casting has been developed. The model uses combined pressure drops of inter-dendritic feeding, volumetric shrinkage, surface tension and gas evolution during the solidification stage. It also considers the non-equilibrium solidification process by using the Scheil Equation to predict the solute (Al) redistribution at the mushy zone. A series of step-shaped magnesium alloy plate castings have been produced and investigated to validate this new porosity model.

9:50 AM
Local-Electrode Atom-Probe Tomographic Investigation of Strengthening Precipitates in a Mg-7Zn-3Al Alloy at Peak: *Shengjun Zhang*[1]; Gregory Olson[1]; [1]Northwestern University

The precipitates in a Mg-7Zn-3Al (in wt. %) alloy aged to peak hardness at 150°C have been characterized by local-electrode atom-probe (LEAP) tomography. The chemical compositions of the matrix and the precipitates were measured and compared with the results calculated using the COST2 database by the Thermo-Calc software. Ternary nanoscale precipitates have been found consistent with the predicted ϕ phase demonstrating a metastable composition. The measured particle size at peak hardness, corresponding to an equivalent sphere radius of 17 nm, provides in important calibration point for quantitative modeling of precipitation strengthening for alloy design. The interfacial energy is also determined based on the capillarity effect.

10:10 AM Break

10:30 AM
Aging Behavior of Die-Cast MRI230D: *Jessica TerBush*[1]; J. Wayne Jones[1]; Tresa Pollock[1]; [1]University of Michigan

The aging behavior of die-cast MRI230D magnesium alloy has been investigated. Samples were aged for times ranging from 10s to 500h for temperatures between 175 and 350°C. The hardness of the aged specimens was measured using a micro-Vickers test with 500 gf load. After aging for 100h at 250°C, the hardness increased from 68.7 Hv in the as-cast state to 74.7 Hv. Microstructure of the aged specimens was examined using transmission electron microscopy (TEM). Precipitates were observed in the as-cast and aged conditions lying parallel to the basal plane. The results will be compared to a similar study previously conducted on die-cast AXJ530.

10:50 AM
Effect of Pre-Aging on Age Hardening and Microstructure in Mg-Zn and Mg-Zn-Al Alloys: *Keiichiro Oh-ishi*[1]; Kazuhiro Hono[1]; Kwang Seon Shin[2]; [1]National Institute for Materials Science; [2]Seoul National University

Mg-Zn alloys are well known as an age-hardenable alloy. The tensile strength of Mg-Zn alloys were found to increase further by Al additions and two-step aging. The age-hardening response and microstructural variation of Mg-Zn and Mg-Zn-Al alloys were examined by hardness test, transmission electron microscopy (TEM) and three-dimensional atom probe. The two-step aged samples exhibit enhanced age hardening response at an earlier stage compared to the single-aged ones. TEM observations exhibited that the peak aged Mg-Zn samples had rods along the c-axis of the matrix phase and plates lying on the basal plane. The Mg-Zn-Al samples had rods and cuboidal precipitates. After two-step aging, the microstructure becomes finer for both the Mg-Zn and Mg-Zn-Al alloys. Atom probe analyses for the samples pre-aged at 70°C clearly showed the formation of Zn-rich zones.

11:15 AM
The Influence of Zn on the Precipitation Path in Mg-Sn Alloys: *Shaul Avraham*[1]; Tomer Leviatan[1]; Yael Maoz[1]; Alex Katsman[1]; Menachem Bamberger[1]; [1]Technion, Israel Institute of Technology

Intermetallic phases in Mg alloys determine the elevated temperature mechanical properties. Dissolved alloying elements at low temperatures and thermally stable intermetallics can hinder plastic deformation mechanisms (dislocation sliding). The presence of Sn in Mg alloys can result in the high melting Mg_2Sn intermetallic. Zn is a common alloying element in Mg alloys, it can form several Mg-Zn intermetallics. This work is focused on the mechanisms by which Zn modifies the age hardening response of Mg-Sn alloys. A binary Mg-Sn and a ternary Mg-Sn-Zn alloys were designed according to computational thermodynamic simulations. The two alloys were solution treated and aged (t< 32 days, 175°C). Micro hardness measurements were used to follow microsctuctural changes. Thermal analysis and electron microscopy methods were used to analyze the influence of Zn on the Mg_2Sn precipitation.

11:35 AM
Age Hardening Response and Precipitate Microstructures of ZK60 Alloy Containing Trace Additions of Ag and Ca: *Chamini Mendis*[1]; Keiichiro Oh-ishi[1]; Kazuhiro Hono[1]; [1]National Institute for Materials Science

Mg-Zn based ZK60 alloy is a widely used wrought magnesium alloy that has a good combination of strength and ductility but only minor increments in strength is achieved through precipitation hardening due to the very limited hardening response. The low hardening response has been attributed to formation of coarse precipitates of $MgZn_2$. Trace additions of 0.1at.%Ag and 0.1at.%Ca to Mg-2.4Zn and Mg-2.4Zn-0.16Zr (at.%) alloys have been shown to increase the peak hardness in the temperature range 160-200°C. With the combined additions of Ag and Ca the hardening response doubles that observed in the Mg-2.4Zn and Mg-2.4Zn-0.16Zr (at.%) alloys. The transmission electron microscopy examinations revealed that the increment in hardening response was achieved by refining the precipitate distribution observed in the binary alloy. In the presence of Zr, these alloys also show significant refinement of grain structures in addition to the fine scale distribution of precipitates within the grains.

11:55 AM
Thermodynamic Investigation of Alkali-Metal-Induced High Temperature Embrittlement in Mg-Li Alloys: *Shengjun Zhang*[1]; Qingyou Han[2]; Zi-Kui Liu[3]; [1]Northwestern University; [2]Oak Ridge National Laboratory; [3]Pennsylvania State University

Alkali metals are undesirable impurity elements in magnesium-lithium alloys. Despite their trace amount, they lead to high temperature embrittlement (HTE) due to intergranular fracture which is prone to edge cracking during thermal processing. In the present work, the results of a thermodynamic investigation are presented to elucidate its mechanism and compared with available experimental data. It is demonstrated that HTE arises from an intergranular alkali-metal-rich liquid phase that segregates into grain boundaries from the matrix and significantly weakens their strength. In order to suppress HTE, a proper processing temperature should be chosen to locate an alloy in the HTE safe zone, according to the alloy composition. There exists a maximum alkali-metal content above which HTE cannot be avoided for a given Mg-Li alloy. A new model is developed to describe the tendency of HTE, which shows grain refinement can decrease the tendency.

Magnesium Technology 2008: Wrought Alloys II

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Mihriban Pekguleryuz, McGill University; Neale Neelameggham, US Magnesium LLC; Randy Beals, Chrysler LLC; Eric Nyberg, Pacific Northwest National Laboratory

Tuesday AM — Room: 291
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Ravi Verma, General Motors Corporation; Sean Agnew, University of Virginia

8:30 AM
Direct Chill Casting and Plastic Deformation of Magnesium Alloys: *Gady Rosen*[1]; Menachem Bamberger[2]; Galia Harel[2]; Enrico Evangelista[3]; Mohamad El Mehtedi[3]; Sandrine Sereni[4]; [1]Alubin Ltd; [2]Technion Israeli Institute of Technology; [3]Università Politecnica Delle Marche; [4]Centro Ricerche Fiat-Societa Consortile Per Azioni

Magnesium alloys stimulate growing interest for use in structural and functional components (aeronautics, automotive, electronics, etc.). Due to their attractive mechanical properties the market share of wrought products is rapidly increasing. In order to produce sound extruded products, high quality billets (feed stock) are required. Therefore, in-depth understanding of the influence of casting conditions on the production quality, and on micro-structure, properties and workability are of high importance. Efficiency of the production can be improved by utilizing advanced process-simulation. Modelling the underlying physics aspects so that process improvement variables can be identified and controlled, resulting in significant benefits. Thus, modelling the solidification of a casting yields: Temperature profiles for a more accurate solidification analysis. Predict solidification time and the effect of cooling system on the solidification. Estimate billets micro-structure, and, mechanical properties in as cast state. Complimentary work is conducted on torsion tests to produce constitutive constants for FEM models.

8:50 AM
Effect of Reheating and Warm Rolling on Microstructure and Mechanical Properties of Twin Roll Strip Cast Mg-4.5Al-1Zn-0.3Mn-0.3Ca Alloy Sheet: *Suk-Bong Kang*[1]; Hongmei Chen[1]; Hyoung Wook Kim[1]; Jae Hyoung Cho[1]; [1]Korea Institute of Materials Science

Twin roll strip cast magnesium sheet was prepared to evaluate the characteristic of microstructure induced by ralatively higher cooling rate. The segregation of aluminum elements was hardly seen to exist after more than 60 % rolling reduction at 300°C. In order to study the effect of reheating and warm rolling on twin roll strip cast magnesium alloy sheet, the thickness of sheet was reduced from 3.2 mm to 0.6 mm through 5 sequential reheating at 350°C for 10 min and warm rolling at about 300°C. Tensile test was performed after each warm rolling pass and following reheating. The microstructure was also observed along the through thickness of cast and rolled sheets together with following reheated ones. The grain refinement during reheating and warm rolling is attributed to the formation of heavy shear band partly from the micro-sized precipitates of Al2Ca and/or Al6Mn.

9:10 AM
Texture Randomization of Magnesium Alloys Containing Rare Earth Elements: Sean Agnew[1]; *Jeremy Senn*[1]; [1]University of Virginia

Rare earth (RE) element-containing Mg alloys were thermomechanically processed and characterized to understand the mechanisms by which their deformation texture is randomized during recrystallization. Two conventional Mg-Zn-Zr alloys were analyzed in the same fashion, because they are known not to randomize. The deformation textures of all the alloys examined were similar to one another after plane strain compression. However, the texture radically decreased during recrystallization and subsequent grain growth in the Mg-RE alloys. It was found that these alloys generate randomly oriented nuclei during recrystallization in shear bands, and further reduced their textures during subsequent grain growth. For the Zn-containing materials, grain boundary nucleation and bulging were observed during recrystallization, and the textures actually increased during grain growth. It is believed that the texture increases in these conventional alloys due to an oriented growth mechanism, which may be shut down in the case of Mg-RE alloys due to solute drag.

9:30 AM
Constitutive Behavior of Four Wrought Magnesium Alloys under Warm Forming Conditions: *Sean Agnew*[1]; Cyrus Dreyer[1]; John Polesak III[1]; William Chiu[1]; Charles Neil; Marcos Rodriguez[2]; [1]University of Virginia; [2]Universidad Plitécnica de Madrid, Ciencia de los Materiales, Centro Nacional de Investigaciones Metalúrgicas-Consejo Superior de Investigaciones Cientificas

The constitutive response of traditional wrought magnesium alloys, AZ31 and ZK10, is compared with two new rare earth element-containing alloys. The alloys were tested in tension over a range of strain rates and temperatures relevant to warm forming technology. The alloy denoted ZW41, which contains 4 wt% Zn and 1 wt% Y, exhibits superior elongation to failure within specific rate and temperature regimes. A proprietary alloy designated 'X' was shown to exhibit superior strength, particularly at the highest temperatures explored. All the alloys exhibited a temperature regime over which their elongation to failure decreased or at least remained constant with temperature and this was understood in terms of their strain hardening and rate hardening responses

9:50 AM
Microstructure, Mechanical Properties and Bendability of AM60 and AZ61 Magnesium Alloy Tubes: Yingxin Wang[1]; *Alan Luo*[2]; Xiaoqin Zeng[1]; Anil Sachdev[2]; Li Jin[1]; Raj Mishra[2]; Wenjiang Ding[1]; [1]Shanghai Jiao Tong University; [2]General Motors Corporation

Microstructure, mechanical properties and bendability of AM60 and AZ61 magnesium alloy tubes have been investigated. The AM60 tubes were successfully extruded at the temperatures of 370°C and 420°C and average strain rates of 0.1 s-1 and 0.6 s-1; while the AZ61 tubes were extruded at 410°C and 0.1 s-1. The lower extrusion temperatures resulted in higher yield strength for the AM60 tubes, and the strain rate had no significant effect on the mechanical properties. The bendabililty of both AM60 and AZ61 alloy tubes improved at elevated temperatures. For AM60 alloy tubes, a moderate temperature range (100 - 200°C) was sufficient since twinning and possible non-basal slip enhanced the formability. Higher bending temperatures (200 – 250°C) were needed for bending AZ61 alloy tubes where extensive non-basal slip was facilitated by the CDRX (continuous dynamic recrystallization) mechanism.

10:10 AM Break

10:30 AM
Study of Deformation Modes in AZ80 Magnesium Alloy: *Jayant Jain*[1]; Warren Poole[1]; Chad Sinclair[1]; [1]University of British Columbia

This work represents an effort to examine the basic deformation modes in high aluminium bearing magnesium alloy (AZ80, 8 wt% Al) at low temperature with the motivation being that these results will provide insight into room-temperature deformation mechanisms. The alloy was deformed in solution-treated condition where the material had a grain size of 32 µm and there was a random orientation of grains. Uni-axial compression was applied at 77K by immersing the compression rig in liquid nitrogen. The deformed surface of the sample was examined for slip and twin identification using Nomarski interference microscopy and electron backscattered diffraction, respectively. The activation of different slip modes (basal, prismatic, pyramidal ([a] type) and second order pyramidal ([c+a] type)) and twin modes ({10-12}, {10-11}, {10-11}–{10-12}) were considered during trace analysis. Preliminary results suggest the importance of non-basal slip (both [a] and [c+a]) and tensile twinning ({10-12}) during low-temperature deformation behaviour of AZ80.

10:50 AM
Development of a Strip-Rolling Technology for Mg Alloys Based on the Twin-Roll-Casting Process: *Rudolf Kawalla*[1]; Matthias Oswald[1]; Madlen Ullmann[1]; Christian Schmidt[1]; Hans-Peter Vogt[2]; Nguyen Duc Cuong[2]; [1]Freiberg University of Mining and Technology; [2]MgF Magnesium Flachprodukte GmbH

The Institute of Metal Forming, Freiberg and the MgF, Freiberg are currently working on a new production technology for magnesium strip, based on twin-roll-casting and strip rolling. By means of this economic method it is possible to produce strips in deep drawing quality with good forming properties in order to satisfy the request for low cost Mg sheets in the automotive and electronic industry. Strips of magnesium wrought alloy AZ21 and AZ31, which have been produced by twin-roll-casting technology, feature a good forming ability. In order to develop an optimal strip rolling technology it is important to discover the influence of the different technological factors. Therefore, the project aims at determining the property development depending on several influencing factors. Consequently, conclusions regarding the technology can be made. Besides, fundamental research in terms of microstructure and texture development of twin-roll-casted and rolled magnesium strips has been conducted.

11:10 AM
The Flow Stress during Hot Compression Deformation of Mg-Gd-Y-Zr Magnesium Alloy: Experiments and Numerical Simulations: Xinping Zhang[1]; *J. Wang*[1]; [1]Department of Materials Science and Engineering, Nanjing University of Science and Technology

The hot deformation behaviour of Mg-Gd-Y-Zr alloy was investigated by compression test and Finite Element analysis method. Compression tests were conducted in the temperature range 350 to 500°C and strain rate range 0.1 to 0.4 per second. The load-displacement data obtained from the compression tests were used to obtain stress-strain data for hot compression by inversed Finite Element method. As expected, the flow stress increases with the increase of strain rate, and decreases with the increase of the deforming temperature. The flow stress of the Mg-Gd-Y-Zr magnesium alloy during hot compression deformation can be described by using Zener-Hollomon parameter including Arrhenius item, and the deformation activation energy is estimated to be 230.098kJ/mol.

11:30 AM
Weld-Seam Quality of Hollow Magnesium Alloy Extrusions: *Wim Sillekens*[1]; Daniël van der Linden[1]; Andrew den Bakker[2]; [1]TNO Science and Industry; [2]Nedal Aluminium

Extrusion technology for wrought magnesium alloys is still maturing, with research efforts being directed to bringing production efficiency as well as product quality to a level matching up to aluminum. For hollow extrusions, one particular aspect concerns the mechanical integrity of the so-called longitudinal weld seams (that form when the metal streams flowing around the mandrel in the die are rejoined through a process of solid-state bonding).This paper presents the results of laboratory trials in which tubular extrusions were produced using a porthole die. Several magnesium alloys (ZM21, AZ31, AZ61, ZE10) were investigated along with some corresponding aluminum alloys (AA6060, AA6082). Tube expansion tests and light-optical microscopy were carried out to study the quality of the weld seams in relation to the parent material. The results show that weld-seam quality in magnesium alloys is much less critical than in aluminum alloys (notably AA6082).

11:50 AM
Grain Refinement Studies in Two Different Mg Alloys by Severe Deformation Process and by Rolling: Bilal Mansoor[1]; *Amit Ghosh*[1]; Raymond Decker[2]; [1]University of Michigan; [2]Thixomat, Inc.

Significant grain refinement of Mg alloys has been achieved to produce low temperature superplastic forming capability in these alloys. Severe plastic deformation is imparted to two different magnesium alloys, AZ31C and AM60. The AZ31C Mg alloy plate is processed via alternate biaxial reverse corrugation(ABRC) process followed by flattening and hot rolling. The initial bimodal grain structure is progressively subdivided during repeated deformations followed by flattening at 250°C and hot rolling at 350°C to nearly uniform microstructure of average size 1:5 micron. Thixomolded AM60 samples showed some cracking under the ABRC processing regime and was processed in by hot rolling at 350°C. It was shown that ultrafine grain size structure could be obtained for AZ31C and AM60 magnesium alloys. Comparison of mechanical properties and formability properties of the two alloys is provided in this article (work supported under grant from NSF).

12:10 PM
Texture Modification to Reduce Yield Anisotropy in Wrought Mg Alloys: *Toshiji Mukai*[1]; Hidetoshi Somekawa[1]; Alok Singh[1]; Tetsuya Shoji[2]; Akira Kato[2]; [1]National Institute for Materials Science; [2]Toyota Motor Corporation

As is well known from previous literatures, wrought magnesium alloys often show yield anisotropy; the yield stress in tension shows a higher value than that in compression. This yield anisotropy results from the formation of strong basal texture in wrought magnesium alloys. In this study, texture modification has been carried out to reduce the yield anisotropy. For a conventional AZ31 alloy, the basal texture was modified to make it relatively random by applying ECAE which can change the texture by reiterative shear loading. Another method used for texture modification was by changing the recrystallization behavior with a combination of adding a rare earth element and severe plastic working. Microstructure analysis by SEM/EBSD revealed that both the examined alloys possessed a relatively random texture. Yield stress in compression was found to be comparable to that in tension in a wide range of strain rate. The deformation mechanism will be also addressed.

Materials in Clean Power Systems III: Fuel Cells, Hydrogen-, and Clean Coal-Based Technologies: Solid Oxide Fuel Cells: Metallic Interconnects

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee
Program Organizers: Zhenguo “Gary” Yang, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; K. Scott Weil, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University; Ayyakkannu Manivannan, National Energy Technology Laboratory

Tuesday AM	Room: 392
March 11, 2008	Location: Ernest Morial Convention Center

Session Chairs: James Rakowski, Allegheny Ludlum; Richard Smith, Montana State University

8:30 AM Invited
Evaluation of a Ferritic Stainless Steels for SOFC Interconnect Application: *Paul Jablonski*[1]; Christopher Cowen[1]; David Alman[1]; [1]National Energy Technology Laboratory

Ferritic stainless steels have many characteristics that are attractive for application as an interconnect in SOFC. These characteristics include low cost, good thermal expansion matching with the ceramic components and slow growth of adherent oxide scales in service. We have developed a surface treatment which greatly enhances the oxidation resistance of this class of alloys in SOFC relevant conditions. We report on the performance of this surface treatment on several stainless steels. We also discuss a probable mechanism for the enhanced performance and the likelihood for long term effectiveness of treated stainless steels.

9:00 AM Invited
Metallic Alloys for Solid Oxide Fuel Cell Interconnects: *James Rakowski*[1]; [1]Allegheny Ludlum

Ferritic stainless steels are viable candidates for solid oxide fuel cell interconnect substrates due to attractive elevated temperature properties. Commercially available alloys with 16-30 wt. % chromium have been used as interconnects with varying degrees of success. Commonly encountered issues include oxide scale resistivity, spallation, and evaporation. Fe-Cr alloys with optimized compositions have been melted, processed, and tested in environments simulating SOFC operating conditions, with the results compared to those obtained for commercially available ferritic stainless. In addition to alloy development efforts, novel surface modifications applied to off-the-shelf materials have been investigated with promising results.

9:30 AM
Investigation and Development of Cost-Effective Ferritic Stainless Steel Interconnects for SOFC Applications: *Zhenguo “Gary” Yang*[1]; Guanguang Xia[1]; Zimin Nie[1]; Joshua Templeton[1]; Sharri Li[1]; Jeff Stevenson[1]; Prabhakar Singh[1]; James Rakowski[1]; [1]Pacific Northwest National Laboratory

Ferritic stainless steels are among the most promising candidate materials for interconnect applications in intermediate-temperature planar SOFC stacks due to their good oxidation resistance and excellent thermal expansion match to the cell components. In particular newly developed compositions demonstrate improved properties relevant to the interconnect applications over traditional ones. However there is still a need of further reduction of chromium volatility, long term-stability and foremost cost-reduction. Thus efforts have been initiated to search and develop ferritic stainless steel interconnects that are made from more cost-effective ferritic substrates, while being protected with conductive oxide coatings. This paper will present details of this work and discuss the suitability of the developed interconnects.

9:55 AM
The Glass-Metal Joint Strength Improvement in SOFC Interconnect Stacks by Reactive Air Coating Process: *Sung-tae Hong*[1]; Jung Pyung Choi[1]; K. Scott Weil[1]; [1]Pacific Northwest National Laboratory

One of the keys to making glass-ceramic sealants viable as a long-term sealing solution for SOFCs is to control their reactivity with the metal components. For example, the barium aluminosilicate class of sealants generally adhere well to YSZ with little chemical interaction, but tend to form interfacial reaction products such as barium chromate ($BaCrO_3$) and monocelsian ($BaAl_2Si_2O_8$) with the oxide scales of the various candidate stainless steel alloys. With long-term exposure at the stack operating temperature, it has been previously shown that these phases thicken and become porous yielding interfaces that are often weak and susceptible to thermomechanically induced cracking. This correlates with observations of the glass-ceramic failures along the metal/sealant interface observed in full-scale stacks operated for 1000hrs or longer. The study presented here examined a new process to modify the surface of the metal component. Simple mechanical tests performed to quantitatively evaluate the effect of modification on the joint strength showed an improvement of 350% relative to conventional glass joints. The reasons for this can be explained in part based on findings from microstructural analysis, which will also be presented and discussed.

10:20 AM Break

10:30 AM
The Catalytic Property of Ni-Fe Alloys towards Methane Oxidation in SOFC: *Xiaochuan Lu*[1]; Jiahong Zhu[1]; [1]Tennessee Technological University

SDC + Ni-Fe composites have been investigated as anode for SOFC running on both H_2 and methane. Single cell with $Ni_{0.8}Fe_{0.2}$ + SDC anode and Pt current collector possessed the highest maximum power density of 1.43 and 1.03 W/cm^2 in wet H_2 and CH_4 at 800°C, respectively. Only 3.3 % loss in power density was observed during 100 hours operation in methane at 800°C. To eliminate the possible contribution of Pt towards methane oxidation, the same single cell with Au current collector was also run in H_2 and methane. The cell exhibited a maximum power density of 0.94 W/cm^2 in H_2 and a much lower power density of 0.36 W/cm^2 in methane. To clarify the catalytic property of Ni-Fe alloys towards methane oxidation, composite anodes of YSZ + $Ni_{1-x}Fe_x$ were exposed to methane at 800°C for 10 h. Additional single-cell testing with the anodes was conducted to confirm the results.

10:50 AM
Microstructure Modeling and Design of Solid Oxide Fuel Cells: Kei Yamamoto[1]; *Edwin Garcia*[1]; [1]Purdue University
With soaring oil prices and diminishing fossil fuel reserves, the development of new ways to produce electricity has become a priority. One such technology is Solid Oxide Fuel Cells (SOFC), whose elevated operating temperatures allow them to use a wide variety of fuels. Fuel cell performance is intricately related to material properties and thus a correctly engineered microstructure will increase the efficiency of the device. In this paper, the effect of particle size, crystallographic orientation, morphological anisotropy, porosity, etc., are engineered to maximize the delivered power density of the device. Moreover, the details and spatial distribution of triple phase boundaries are tailored by implementing a novel numerical approach that accounts for the local electrochemical particle-particle interactions. Alternate, non-conventional device architectures are proposed.

11:15 AM
Performance Evaluation of SOFC with Different Interconnect Alloys/ Coatings: *John Batey*[1]; [1]Tennessee Technological University
The performance stability of a solid oxide fuel cell (SOFC) was studied with different interconnect materials. The cell configuration included a porous Ni/YSZ anode substrate as support, a Ni/YSZ anode interlayer, a thin YSZ electrolyte layer, a LSM/YSZ cathode interlayer, and a LSM cathode, and a Pt layer for cathode-interconnect contact, and a channeled alloy interconnect. The cell testing was conducted at 1073K using H2+3%H2O gas as fuel and the current density of the cell was monitored as a function of time at a fixed cell voltage of 0.7V. Several alloy interconnects, such as Crofer 22 APU and low-Cr Fe-Ni-Co alloys, were selected for evaluation. The spinel coatings evaluated included both thermally-grown spinel phases and spinel layers synthesized via eletroplating. The effectiveness of surface spinel composition/quality on mitigating Cr migration from the interconnect to the cathode was assessed.

11:40 AM Invited
Carbon Tolerant Electrocatalysts Based on Ni/YSZ Modified with Au or Ag: *Stelios Neophytides*[1]; Ilias Gavrielatos[1]; Dario Modinaro[2]; Nicos Triantafylopoulos[3]; [1]Institute of Chemical Engineering and High Temperature Processes; [2]SOFCpower S.r.l.; [3]ADVENT Technologies
Carbon tolerant Ni/YSZ anode modified with 1%at Au or Ag with respect to Ni was tested under methane rich steam reforming conditions. The electrodes had shown high tolerance to C deposits, even at methane to water ratios as high as 3, at temperatures ranging between 700°C-900°C. The Ni based bimetallic anodes were prepared by combustion synthesis at 600°C and sintered under air at 850°C. The SEM images have shown that the Au or Ag modified Ni/YSZ electrode were nanostructured with particle size ranging between 50-100nm. XRD and XPS measurements provide a strong evidence for the formation of surface alloy. Temperature programmed reaction experiments, kinetic and electrokinetic measurements, under CH4 steam reforming conditions in combination to physicochemical characterization of the electrocatalysts, had shown that the presence of Au or Ag plays a vital role in the tuning of CH4 sequential dehydrogenation and oxidation rates of the hydrogenated adsorbed carbon species.

12:05 PM
Interdiffusion in γ (Face-Centered Cubic) Fe-Ni-Cr-X (X = Al, Si or Ge) Alloys at 700°C and 900°C: *Narayana Garimella*[1]; Michael Brady[2]; Yong-ho Sohn[1]; [1]University of Central Florida; [2]Oak Ridge National Laboratory
Effect of minor alloying additions on the interdiffusion behavior of quaternary Fe-Ni-Cr-X (X = Al, Si or Ge) alloys were investigated using solid-to-solid diffusion couples. Fe-Ni-Cr-X alloys (fcc γ-phase) having compositions of Fe-25wt.%Ni-20wt.%Cr-3 wt.%Al, Fe-25wt.%Ni-20wt.%Cr -2.0wt.%Si and Fe-25wt.%Ni-20wt.%Cr -2wt.%Ge were arc-melted and chill-cast. Solid-to-solid diffusion couples were assembled in Invar steel jig, encapsulated in Ar after several hydrogen flushes, and annealed at 700° and 900°C in a three-zone tube furnace for 168 and 720 hours, respectively. Experimental concentration profiles were determined by using electron probe microanalysis with pure standards. Interdiffusion fluxes of individual components were determined directly from the experimental concentration profiles, and integrated to determine average interdiffusion coefficients. Effects of alloying additions on the diffusional behavior of Fe-Ni-Cr-X alloys are examined in the light of the formation and the growth kinetics of protective Cr_2O_3 scale.

Materials Informatics: Enabling Integration of Modeling and Experiments in Materials Science: Informatics and Materials Property Design

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizer: Krishna Rajan, Iowa State University

Tuesday AM — Room: 271
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: To Be Announced

8:30 AM
Advanced Computational Modeling of Titanium Alloys: *Jaimie Tiley*[1]; [1]US Air Force
Three dimensional analysis of titanium alloys using new electron microscopy techniques provides incredible potential for incorporating texture information into mechanical behavior models. The use of crystallographic data provides vital information on slip lengths and three dimensional shapes of Widmanstatten alpha and beta structures that is used in property-microstructure models including fuzzy logic and neural network models. Results using the slip length data are compared to more traditional regression models to determine relationships between microstructure features and properties. In addition, probability distribution functions are used in place of mean values to improve accuracy and determine interparameter relationships between features.

9:10 AM
Application for Pre-Calculation Global Cost of Heat Treatment: Szota Michal[1]; Jozef Jasinski[1]; *Grzegorz Walczak*[1]; [1]Czestochowa Technical University
At present, different thermo-chemical techniques are used in the thermo chemical treatment practice. One of this is heat treatment in fluidized bed. This is characterized by high heat and mass transfer. These techniques are very often used in the research institutes and small industrial plants. In this processes is very important predicted global cost. These research are conducted in order to introduce analysis cost of thermo-chemical process, before it will be lead. Special prepared software connected with this system may be using in pre-calculated process cost. The cost pre-calculation is very important for process designer, because at present prices of every thing rise continuously every day. The customer usually wants get to know of heat treatment, before he commissions workmanship of it.

9:35 AM
Computational Design and Prototype Evaluation of Aluminide-Strengthening bcc-Based Superalloys for Elevated Temperature Applications: *Zhenke Teng*[1]; Shenyan Huang[1]; Peter K. Liaw[1]; Chain T. Liu[1]; Morris E. Fine[2]; Gautum Ghosh[2]; Mark D. Asta[3]; Gongyao Wang[1]; Daniel Worthington[3]; [1]University of Tennessee; [2]Northwestern University; [3]University of California, Davis
Strengthening through a homogeneous distribution of a second phase is widely employed in the design of high-temperature materials. Nickel-based γ/γ' superalloys owe their excellent creep strength to the presence of a high volume fraction of coherent-coplanar ordered fine γ' precipitates. Like γ/γ' systems, the body centered cubic (bcc) Fe-matrix with NiAl-type precipitates exhibit a cube-on-cube orientation relationship. However, ferritic steels with ß/ß' phases are brittle at room temperature and they are only used at temperatures below 893K due to the limited creep resistance. In our study, computational thermodynamic modeling is employed for the phase-stability study and microstructure design. Prototype Fe-based alloys with microstructures analogous to the classical Ni-based γ/γ' superalloys are obtained. The creep resistance can be improved by adding small amounts of alloying elements. This project is acknowledged by the Department of Energy (DOE) Office of Fossil Energy Program, Dr. Patricia Rawls.

10:00 AM
Neural Networks Modeling Used for Improving Mechanical Property of Cars Elements after Heat Treatment in Fluidized Bed: *Szota Michal*[1]; Jozef Jasinski[1]; [1]Czestochowa Technical University

This paper presents neural network model used for improving mechanical property of cars elements after heat treatment in fluidized bed. Thermo-chemical process in fluidized bed is very complicated and difficult. The multi-parameters changes during this process are non linear characteristic. This fact and lack of mathematical algorithms complex describing processes in fluidized bed makes modeling by traditional numerical methods difficult or even impossible. In this case it is possible to try using artificial neural network. Such prepared neural network model, after putting expected values of mechanical property of steel in output layer, can give answers to a lot of questions about leading thermo-chemical process in fluidized bed. Neural network simulation allowed to carried out heat treatment with suitable process parameters, which allowed to obtain better mechanical property of cars elements after heat treatment.

10:25 AM
Scaling Techniques as Part of the Materials Informatics Toolbox: *Patricio Mendez*[1]; [1]Colorado School of Mines

Scaling is a proven technique for the synthesis, generalization, and extrapolation of knowledge across systems in physics, applied mathematics, and many engineering disciplines. Judging by the impact of scaling on those other disciplines, their application to the field of materials science and engineering holds enormous potential. Paradoxically, scaling techniques are seldom applied in the materials field, and the results in those cases are occasionally unreliable. This presentation will discuss the reasons behind this paradox, promising efforts and techniques aimed at overcoming the challenges, and the type of results that could be accomplished by using scaling techniques as a tool of materials informatics.

11:05 AM
Titanium Based Alloy Chemistry Optimization for Maximum Strength, Minimum Weight and Minimum Cost Using JMatPro and IOSO Software: *George Dulikravich*[1]; Amit Kumar[1]; Igor Egorov[2]; [1]Florida International University; [2]IOSO Technology Center

Chemical concentrations of Ti-Al-Cr-V alloys were predicted computationally so that they maximize Young's modulus of elasticity while minimizing alloy density and cost of raw materials. JMatPro software calculated the properties and IOSO software performed multi-objective evolutionary optimization. The method was applied at temperatures up to 1500°C. The intention was to demonstrate a possibility to computationally create alloys having multiple superior properties without a need for expensive experimental verifications. The resulting alloys at each temperature had significantly reduced cost and lower density while increasing the Young's modulus. This method is applicable to alloys with a large number of alloying elements and significantly more properties. The accuracy of the method depends on the accuracy of the software packages used. [1]http://www.jmatpro.com [2]I.N. Egorov, "Indirect Optimization Method on the Basis of Self-Organization," Proceedings of Optimization Techniques and Applications (ICOTA'98), Curtin University of Technology, Perth, Australia, 2 (1998), 683-691.

Materials Processing Fundamentals: Powders, Composites, Coatings and Measurements

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Process Technology and Modeling Committee
Program Organizer: Prince Anyalebechi, Grand Valley State University

Tuesday AM — Room: 283
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Prince Anyalebechi, Grand Valley State University

8:30 AM
Preliminary Investigation into the Effect of Composite Reinforcement Particle Size on the Sintering of Ti-Titanium Boride Dual Matrix Composites: *Khaled Morsi*[1]; V.V. Patel[1]; Kee Moon[1]; Ahmed El-Desouky[1]; Javier Garay[2]; [1]San Diego State University; [2]University of California-Riverside, Department of Mechanical Engineering

Titanium-titanium boride whisker (TiBw) composites are emerging as strong candidate materials for advanced applications within the automotive, aerospace and defense industries. Although increasing TiBw volume fraction improves the specific stiffness and wear resistance of titanium, it is usually on the expense of fracture toughness and ductility especially at the higher volume fractions of reinforcements where whiskers interlock. This research addresses strategies for microstructural design that may overcome these shortcomings, by generating titanium composites that are locally reinforced with TiBw through in-situ reaction in segregated regions of the microstructure and separated by ductile Ti matrix. The effect of varying composite reinforcement particle sizes (i.e. size of the reinforced regions)on the pressure-less sintering of these dual matrix composites is discussed. Electric current activation was also used to generate high density dual matrix composites and a new simultaneous imaging and modulus mapping technique was used to characterize these unique microstructures.

8:50 AM
Biomorphic Cellular TiC/C Ceramics from Woods: *Sutham Niyomwas*[1]; [1]Prince of Songkla University

Biomorphic Cellular TiC/C ceramics were produced by vacuum-infiltrating carbon preforms with TiO_2 sol and subsequently synthesized by carbothermal reduction process. Carbon preforms were pyrolyzed from para-rubber wood, sadao-chang wood and coastal pine wood. The effect on holding infiltrated time of TiO_2 sol to the TiC/C products was analyzed. By observing the resulted products by SEM and XRD found that TiC/C was formed with retaining morphology of the original template structure.

9:10 AM
An Investigation of TiB_2 Synthesis Using TiO_2/B_4C/C Powder Mixture: *Filiz Sahin*[1]; Kutluhan Kurtoglu[1]; Bora Derin[1]; Onuralp Yucel[1]; [1]Istanbul Technical University

This paper presents a synthesis of TiB_2 via carbothermal reduction methodusing a mixture of TiO_2, B_4C and C prepared in molar ratio of 2:1:3. Reduction experiments were carried out in a high temperature Tammann Nernst furnace in a graphite crucible under argon atmosphere at different time (0-15-30-60 min.) and temperatures (1400-1500-1600-1700°C). The products were ground and investigated using XRD, SEM and BET techniques. Results were also compared with the literature about the carbothermally produced TiB_2 using mixture of TiO_2, B_2O_3 and carbon black powder.

9:30 AM
Periodic Technique for Measurement of the Thermal Properties of Nanocomposite Materials: *Garrett May*[1]; Donald Swart[1]; Kevin Stokes[1]; [1]University of New Orleans

We present a periodic technique for measuring the thermal conductivity of thin, disk-shaped samples. In samples of this type, temperature measurements must be made across the sample faces and are therefore subject to large error due to the interface resistance between the temperature sensor and the sample. The technique uses measurements of the amplitude and phase of the periodic temperature across both a reference sample and the unknown material at several different frequencies. Modeling of the heat flow in the sample allows the simultaneous determination of the thermal parameters of the sample as well as the interface resistance. The technique is demonstrated on nanocomposite samples less than 1.0 mm thick.

9:50 AM
Preliminary Investigations into the Aqueous Processing of Alumina-Carbon Nanotube Composites: *Roberto Angulo*[1]; F. Büyüksönmez[1]; Khaled Morsi[1]; [1]San Diego State University

Carbon nanotubes(CNTs) have recently emerged as materials with outstanding properties. This has promoted their consideration as reinforcements for other material systems, such as ceramics. However, one of the fundamental problems with producing CNT-reinforced ceramics is the effective dispersion of CNTS during processing. This has been successfully carried out recently using organic solvents, albeit with negative implications for the environment. This paper investigates the rheological behavior of aqueous CNT-alumina slips, with the objective of producing an environmentally friendly aqueous tape casting process for the production of alumina-CNT composites. The effect of CNT (and surface modified CNT) addition on the rheology of the slip and tape casting is presented.

10:10 AM Break

10:20 AM
Titanium Diboride Synthesis by Carbothermal Reduction of TiO_2 and B_2O_3: Kutluhan Kurtoglu[1]; *Bora Derin*[1]; Filiz Sahin[1]; Onuralp Yücel[1]; [1]Istanbul Technical University

The synthesis of titanium diboride (TiB_2) by carbothermal reductions of TiO_2 and B_2O_3 mixed with carbon black was investigated. For this aim, TiO_2, B_2O_3 and carbon black powders were dried, weighed out in stoichiometric proportions, ball-milled, and then charged into graphite crucibles. The experiments were carried out in a graphite resistance furnace (Tammann furnace) in argon media using different range of temperature (1400-1700°C) and time (0-60 min). The phase analyses and the microstructural observation of the products were examined by using XRD and SEM. Results were also compared with the TiB_2 products previously obtained with self-propagating high-temperature synthesis (SHS) method using mixture of TiO_2, B_2O_3 and Mg followed by acid leaching.

10:40 AM
Activity Measurement of REs and Their Alloy System Using Multi-Knudsen Cell Mass Spectrometry: *Woong-hee Han*[1]; Masao Miyake[2]; Hisao Kimura[1]; Masafumi Maeda[1]; [1]University of Tokyo; [2]University of Illinois

Although development of the production process with rare earth (RE) metals requires thermodynamic data of alloys, available experimental and theoretical data in the literature aren't sufficient yet. Those can be attributed to the experimental difficulties brought by the high reactivity of rare-earth metals. In this study, vapor pressure of RE metals and activities of RE in alloys were determined by Knudsen cell mass spectrometry. The Knudsen cell method was developed to measure vapor pressure of species by weight loss per evaporated unit area. After then, combination of a single Knudsen cell with mass spectrometry could determine activity in metal alloy by measured ion current of evaporated species from a substance. In this study, Multi Knudsen cell was used. The method of multi Knudsen cells mass spectrometry allow measurement of ion currents of evaporated species from substances, which are pure metal taken as reference, and RE alloy, in a single experiment.

11:00 AM
Transportation of a Liquid Droplet on a Substrate Having a Spatial Gradient: Itaru Jimbo[1]; *Minehiro Arai*[1]; Norimasa Okada[1]; [1]Tokai University

A surface having a spatial gradient in its surface free energy is promising for its applicability. The transportation of a liquid droplet on such surfaces does not need complicated mechanisms or energy sources. This kind of surface can be applied to form various shapes to transport liquid or even bubbles. The idea was first documented in the study of immunochemistry by Swedish scientists in 1984, where a spatial gradient of surface free energy was made on a silicon substrate by using methylsilane. In this study, the concept and various schemes of droplet and bubble transportation will be reviewed and the capability of the surface properties to realize the motion of a droplet will be discussed on several key parameters such as advancing and receding contact angles and surface free energy gradient. Microscopic observation will also be introduced in order to clarify the relationship with the surface wettability.

11:20 AM
Synthesis of Nanosized Tungsten Carbide Powder by Thermal Plasma Process: *Taegong Ryu*[1]; Kyu Sup Hwang[1]; Hong Sohn[1]; Zhigang Fang[1]; [1]University of Utah

Thermal plasma has been applied to the preparation of nanosized tungsten carbide powder. The reduction and carburization of the vaporized tungsten chloride by methane-hydrogen mixtures produced nanosized WC_{1-x} powder, which sometimes contained the WC, W_2C and/or W phase. The products were characterized using XRD, carbon analyzer, and TEM. The effects of plasma torch power, flow rate of the primary plasma gas (Ar) to generate plasma flame, CH_4/WCl_6 molar ratio, and H_2/CH_4 molar ratio on the product composition were investigated. The tungsten carbide (WC_{1-x}) powders showed particle sizes of less than 30 nm. The powder was treated under hydrogen atmosphere to fully carburize the WC_{1-x}, W_2C and W and to remove excess carbon, from which essentially pure WC powder was obtained.

11:40 AM
Rapid Synthesis of Ultrafine WC-Co Cemented Carbides by In-Situ Reactions and Spark Plasma Sintering: *Xiaoyan Song*[1]; Jiuxing Zhang[1]; Wenbin Liu[1]; Kai Wang[1]; Shixian Zhao[1]; Mingsheng Wang[1]; Xuemei Liu[1]; [1]Beijing University of Technology

A novel route for rapid synthesis of ultrafine WC-Co cemented carbides with homogeneous binder phase distribution is reported. The composite powder is produced by in situ reduction-carbonization reactions by using $WO_{2.9}$, Co_3O_4 and carbon black as raw powders. The reaction equilibrium and phase stability are studied by thermodynamic calculations. The WC-Co composite powder is condensed by spark plasma sintering (SPS) to obtain the cemented carbides bulk. The sintering temperature is lower than that in the conventional sintering methods by 150-300°C, while the sintering time is remarkably reduced by 3-5 times. The high performances of the hardness and the fracture toughness are obtained. The experimental results confirm that the present processing scheme of *in situ* reduction-carbonization reactions and subsequent SPS sintering is an efficient way to prepare WC-Co cemented carbides, and is apparently superior to the conventional long-period techniques of preparing composite powder and cemented carbides bulk.

Mechanical Behavior, Microstructure, and Modeling of Ti and Its Alloys: Phase Transformation and Microstructure Development II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
Program Organizers: Ellen Cerreta, Los Alamos National Laboratory; Vasisht Venkatesh, TIMET; Daniel Evans, US Air Force

Tuesday AM — Room: 384
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Ellen Cerreta, Los Alamos National Laboratory; Michael Mills, Ohio State University

8:30 AM Invited
Constitutive Based Modelling of the Alpha Phase Lath Width and Spherodisation in Titanium-6-4: *Jeffery Brooks*[1]; M. Mulyadi[2]; M. Rist[2]; [1]Qinetiq Ltd; [2]Open University

The control of alpha lath width during hot working and the subsequent formation of a uniform spherodised alpha structure during thermo-mechanical processing are essential for the development of the properties required for Titanium-6-4 critical aero-engine parts. A quantitative description of the evolution of flow stress and microstructure is used to predict the alpha lath width and degree of spherodisation of the alpha phase during the hot deformation of Titanium-6-4. The approach is validated using quantitative metallographic analysis data obtained from uniaxial compression tests carried out in the alpha-beta temperature range (850-1000°C) for strains (0-1) and strain rates (0.003-1/s) relevant to primary processing. The validation of the models is dependent on the quality of the microstructural data and as a consequence a number of approaches to the quantification of the structures are discussed together with the errors that are associated with the different measurement techniques and assumptions.

9:00 AM
Characterization of Ti-TiC and Ti-TiN Composites Produced by "Reactive LENS®": *Junyeon Hwang*[1]; Anantha Puthucode[1]; Alderson Neira[1]; Rajarshi Banerjee[1]; Michael Kaufman[2]; [1]University of North Texas; [2]Colorado School of Mines

Ti-TiC and Ti-TiN composites were prepared by a reactive LENS® (laser engineered net shaping) process. It is shown that the as-deposited structures contain primary TiC and primary TiN dendrite cores surrounded by α-Ti grains. Further the TiC and TiN dendrite cores are shown to contain α-Ti precipitates in the form of Widmanstatten plates that lie on {111} planes. In all cases, the α-Ti grains and precipitates have an orientation relationship with the TiC and TiN where the close-packed planes and directions: $\{111\}_{fcc}//(0001)_{\alpha\text{-Ti}}$ and $<011>_{fcc}//<1120>_{\alpha\text{-Ti}}$. In the case of the Ti-TiN composite material, the precipitation of metastable α-Ti precipitates instead of Ti_2N in the primary TiN appears to be related to the mechanism of nucleation of this phase on stacking faults in the TiN. Finally, the similarities and differences in these two systems will be discussed and related to the potential of using reactive LENS for producing structural Ti-matrix composites.

9:20 AM
Effect of Initial Structure on Dynamic Recrystallization in a Ti-Fe Alloy: *Tadashi Furuhara*[1]; B. Poorganji[1]; G. Miyamoto[1]; M. Yamaguchi[2]; Y. Itsumi[3]; K. Matsumoto[3]; T. Tanaka[3]; Y. Asa[3]; [1]Tohoku University; [2]Sumitomo Titanium Corporation; [3]Kobe Steel, Ltd.

Deformation behavior and microstructure evolution of a Ti-1.5Fe alloy in (alpha+beta) two-phase region is studied with focusing on effect of initial microstructure at different deformation temperatures and strain rates. Quenching to room temperature after beta solutionizing and reheating to the deformation temperature causes a fine interlamellar spacing and colony size in the (alpha+beta) lamellar microstructure whereas direct cooling to the deformation temperature results in a coarser lamellar structure. Decrease in interlamellar spacing of initial microstructure results in promotion of DRX at a given deformation condition. At a high temperature where alpha volume fraction is lower, dynamic recovery is a major deformation mechanism while at a lower temperature, i.e., a higher fraction of alpha phase, DRX of alpha phase occurs predominantly. It is concluded that critical needed strain for initiation and completion of dynamic recrystallization is decreased by refining the initial microstructure.

9:40 AM
Finite Element Analysis of the Primary Processing of TIMETAL 555: *Manish Kamal*[1]; Vasisht Venkatesh[1]; [1]Titanium Metals Corporation

TIMETAL 555 (Ti-5Al-5Mo-5V-3Cr), a near beta titanium alloy, is currently under evaluation for several major airframe applications. TIMET has recently started processing Ti-5553 as production heats. Being a relatively new material the understanding of forging routes and their effects on material properties is still in the early stages. Numerical modeling techniques were used to study the cogging process to evaluate the hot workability of Ti-5553. Finite element analysis codes were used to determine the local state of stress, strain, strain rate, and temperature within the workpiece. These variables were then utilized to predict damage within the workpiece using an appropriate ductile fracture criterion. Cogging routes for the primary processing of TIMETAL 555 were designed based on the model. Simulation results show that strain induced porosity should not be an issue with the proposed forging route. However, pass schedules need to be optimized to yield a more uniform final structure.

10:00 AM Invited
Some Aspects of Phase Transformations in Ti and TiAl-Based Alloys: *Michael Loretto*[1]; H. Saage[1]; D. Hu[1]; H. Jiang[1]; N. Wain[1]; J. Mei[1]; X. Xu[1]; [1]University of Birmingham

Some factors which affect the precipitation of alpha in Ti-15-3 and Ti5553 have been investigated with the aim of understanding the factors that control the extent and the nature of the alpha which is precipitated from solution. The influence of ageing temperature and of the addition of small additions of carbon on both grain boundary alpha and of the scale of the alpha precipitated in the matrix in Ti-15-3 has been investigated. These results will be discussed in terms of the role of oxygen in enhancing precipitation of alpha. Recent work has shown that the amount of alpha which is precipitated during heat treatment of TiAl-based alloys in the two phase field can be strongly influenced by the pressures used during HIPping and similar results have been obtained in Ti5553. These observations will be discussed in terms of the control of phase transformations by HIPping and by post-HIP heat treatments.

10:30 AM Break

10:50 AM Invited
Phase Formation and Decomposition in IMI 550 during Cooling from Elevated Temperature: *Henry Rack*[1]; L. Allred[2]; J. Hudson[1]; Javaid Qazi[3]; [1]Clemson University; [2]Oak Ridge National Laboratory; [3]KEMET Electronics Corporation

This investigation has examined the formation and decomposition of phases formed during cooling from elevated temperature in IMI550.Decomposition at the fastest cooling rate examined is initially controlled by the solution treatment temperature, i.e the composition of the parent beta phase, higher temperatures within the alpha plus beta and beta phase field resulting in alpha prime martensite, with lower solution treatment temperatures, where the parent beta phase becomes increasing higher in beta stabilizing content, resulting in orthorhombic martensite formation.Reduction in cooling rate have been found to result in short range diffusion within both the martensitic platelets and increasing within the retained beta matrix.The mechanisms of the alpha phase formation at slower rates will be discussed and related to the commonly observed Widmanstatten, basketweave and colony microstructures observed in titanium alloys after cooling from elevated temperature.

11:20 AM
Modeling and Experimental Validation of Hot Rolling Ti-6-4 Plates: *Vasisht Venkatesh*[1]; [1]TIMET

DEFORM-3D, a commercial finite element analysis (FEA) software, was used to model multi pass hot rolling of Ti-6-4 plates. In order to validate the FEA model appropriately designed small scale rolling experiments were conducted, at alpha+beta temperatures, on 2-high rolling mill using 25 mm thick plates. The effect of key rolling parameters such as temperatures, heat transfer coefficient, friction and pass line on mill load, strain distribution and final plate shape were investigated. In addition, predictions from relevant alpha spherodization models were compared to final rolled microstructures.

11:40 AM Invited
Probing the Early Stages of Phase Separation and Second Phase Nucleation in Complex Beta Titanium Alloys: *Rajarshi Banerjee*[1]; Soumya Nag[2]; Anantha Puthucode[1]; Arda Genc[2]; Hamish Fraser[2]; [1]University of North Texas; [2]Ohio State University

The solid-state decomposition of the beta phase of titanium alloys is a rather complex phenomenon involving multiple competing compositional and structural instabilities including, phase separation in the beta phase and precipitation of the omega and alpha phases. The influence of these beta phase instabilities on the microstructural evolution and resultant mechanical properties of these alloys are rather poorly understood. Furthermore, recent developments in advanced characterization techniques such as high-resolution scanning transmission electron microscopy and 3D atom probe tomography allow for unprecedented insights into the true atomic scale structure and chemistry changes associated with the instabilities in the beta phase of these complex alloys. Such detailed studies are being carried out on two different beta titanium alloys, the Ti-5Al-5Mo-5V-3Cr-0.5Fe (TIMETAL-5553 or Ti-5553) alloy, used in aerospace applications, and the Ti-35Nb-7Zr-5Ta (or TNZT) alloy, used in orthopedic implant applications, and will form the basis of this presentation.

Mechanics and Kinetics of Interfaces in Multi-Component Materials Systems: Mechanical Properties of Interfaces

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee, TMS: Thin Films and Interfaces Committee
Program Organizers: Bhaskar Majumdar, New Mexico Tech; Rishi Raj, University of Colorado, Boulder; Indranath Dutta, US Naval Postgraduate School; Ravindra Nuggehalli, New Jersey Institute of Technology; Darrel Frear, Freescale Semiconductor

Tuesday AM   Room: 279
March 11, 2008   Location: Ernest Morial Convention Center

Session Chairs: Darrel Frear, Freescale Semiconductor; Carl Boehlert, Michigan State University

8:30 AM Keynote
Ductile-to-Brittle Transition in Solder Joints Measured by a Mini Charpy Impact and Drop Machine: *King-Ning Tu*[1]; Yuhuan Xu[1]; Alan Wang[1]; Masaru Fujiyoshi[2]; [1]University of California, Los Angeles; [2]Hitachi Metals

The most frequent failure of wireless, handheld, and portable consumer electronic products is an accidental drop to the ground. The impact may cause interfacial fracture of ball-grid-array solder joints. In this talk, a mini impact and drop tester based on the classic Charpy impact machine was built and utilized to measure the impact toughness and to characterize the impact reliability of both eutectic SnPb and SnAgCu solder joints. The annealing effect on the impact toughness was investigated and the fractured surfaces were examined. A ductile-to-brittle transition for the Pb-free solder joints has been observed. By cross-sectional SEM analysis, it is found that the annealing has allowed more growth of intermetallic compound at the interface, which may have contributed to the ductile-to-transition of the SnAgCu solder joints.

9:10 AM Invited
Characterizing Interfacial Delamination in Layered Thermal Barrier Coatings: *Kevin Hemker*[1]; [1]Johns Hopkins University

Multilayered thermal barrier systems offer enhanced temperature capabilities but are susceptible to interfacial delamination and global spallation. Important phenomena that affect delamination occur in each layer and at the interfaces, and mechanics-based models of these events require a detailed understanding of the properties of each layer. The system investigated in the current study is comprised of: a EBPVD 7YSZ top coat, a thermally grown oxide (TGO), a low-pressure plasma sprayed NiCoCrAlY two-phase bond coat and a Ni-base superalloy substrate. This talk will outline experimental techniques developed to capture the salient material properties of these disparate layers and introduce the physics-based model developed to describe delamination in this system.

9:40 AM
The Strength of Adhesive Joints as Influenced by the Interface Structure and Stress States: *Bhaskar Majumdar*[1]; Michael Kent[2]; Earl Reedy[2]; [1]New Mexico Tech; [2]Sandia National Laboratory

The strength of adhesive joints depends strongly on the interface, with failure shifting from ductile cohesive failure inside the adhesive to brittle interfacial failure, depending on the local chemistry and interface morphology. In this work, self assembled monolayers (SAMs) were used to alter the interface conditions between the substrate and an epoxy based adhesive. Results show a highly non-linear dependence of joint strength on the concentration of interacting sites, where weak sites were obtained with methyl terminated SAMs and strong sites were obtained with bromine terminated SAMs. The non-linear behavior is shown to depend on yielding of the polymer, as also influenced by local stress states. Lap shear tests as well as toughness tests exhibited slightly altered behavior, and the roles of stress concentration and stress states are discussed. These results highlight the importance of developing appropriate design parameters to characterize the strength and durability of joints.

10:05 AM Break

10:20 AM
Complementary Testing Techniques for Mechanical Properties in Metal-Polymer Systems: Peter McNabb[1]; John Yeager[1]; David Bahr[1]; *Marian Kennedy*[2]; [1]Washington State University ; [2]Clemson University

Testing the mechanical properties and fracture resistance of metal-polymer systems is important for reliability in flexible electronics. Using a model system of Au/Kapton, this study focuses on the development of a method to measure interfacial fracture energy using bulge testing, and the toughness is compared to previous four point bending results. Initial results show that this technique, combined with nanoindentation, can be used to study interfacial fracture energies, fatigue lifetimes and creep of metal-polymer systems. Fracture within the metals films was shown to occur during tensile strain of the film system with tensile stress. The interfacial energiescan then be predicted as the substrates is depressurized and the film delaminates due to compression. These fracture energies are shown to be a function of both interlayer chemistries, TiW and W, as well as interphase regions formed by reactive ion etching within the kapton substrate.

10:45 AM
Thermocompression Bonding of Gold Coated Carbon Nanotube Turfs for Heat Tranfer Applications: *Ryan Johnson*[1]; David Bahr[1]; Cecilia Richards[1]; Robert Richards[1]; Jeong-Hyun Cho[1]; Ali Zbib[1]; Amer Hamdan[1]; Devon McClain[2]; Jun Jiao[2]; [1]Washington State University; [2]Portland State University

Vertically aligned carbon nanotubes turfs (VACNTs), grown using chemical vapor deposition, have been proposed as heat transfer media in thermal applications. Mechanical contact allows conduction, making the thermal resistance of the interface between the VACNTs and the contact surface a critical parameter. To normalize the contact surfaces for heat transfer measurements, VACNTs were coated in a thin layer of sputtered gold and then mechanically transferred to a gold film on silicon using thermal compression bonding. Controlling the applied pressure allows clean transfer while avoiding CNT buckling, incomplete transfer, or transfer of the catalyst. A stress of 0.5 MPa has produced a clean transfer of 200 micrometer tall turfs. Electron microscopy and mechanical measurements of interfacial strength are used to characterize the structural implications for thermal conductivity applications. We present a discussion of bonding mechanisms between the separate materials in the structure: the gold, CNTs, and the oxide containing the catalyst.

11:10 AM
Debond Strength Comparisons of Microscopic Evaluations of Ti-24Al-17Nb-0.66Mo (at%), Ti-24Al-17Nb-1.1Mo and Ti-24Al-17Nb-2.3Mo SiC Fiber-Reinforced Composites: Jeffrey Quast[1]; *Carl Boehlert*[1]; [1]Michigan State University

The debond strength is a critical design parameter for continuously-reinforced metal matrix composites (MMCs) as the use of such materials is dependent on the ability of the interface to transfer load from the matrix to the fiber. Research on MMCs containing first generation orthorhombic-based matrix alloys based on Ti-25Al-17Nb-xMo(at.%) has shown promising results. Powder-processed and tape cast four-ply, unidirectional-reinforced MMC samples, possessing Ti-24Al-17Nb-xMo(at.%) matrices with Ultra SCS-6 continuous fibers (approximately 36% vol. pct.), were room-temperature tensile tested with the fibers transverse to the loading direction using a cruciform specimen geometry, which has been previously used for transverse interfacial property evaluation of high volume fraction MMCs. The effects of processing conditions and small Mo additions (0.66, 1, and 2.3at.%) on the debond strength of Ti-24Al-17Nb-xMo(at%) MMCs were investigated. Debonding events were captured by the stress-strain curves along with metallographic examination. The greatest debond strengths were observed in the Ti-24Al-17Nb-1.1Mo MMC.

11:35 AM
Evaluation of Variability on the Thermomechanical Response of Nitride Nuclear Fuels through Microstructurally Explicit Models: *Manuel Parra Garcia*[1]; Sung-Ho Park[2]; Kirk Wheeler[1]; Pedro Peralta[1]; Ken McClellan[3]; [1]Arizona State University; [2]Gyeongsang National University; [3]Los Alamos National Laboratory

A two-dimensional (2D) thermo-mechanical finite element model of a cylindrical fuel pellet seen from the longitudinal plane has been run to investigate variability of the thermo-mechanical response (stress field, strain

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field, grain boundary interaction, temperature distribution) due to microstructure heterogeneity within a Representative Volume Element (RVE). Microstructural information was obtained from sintered ZrN, as a surrogate for PuN, processed under conditions similar to those used in actinide bearing fuels. The 2-D RVE obtained from the microstructural characterization, which includes pore and grain geometry as well as grain orientation, was surrounded by "effective material" and was located at different positions in the model to evaluate the effect of stress and temperature gradients on the local fields. This effort is directed towards the formulation of a framework that can be translated into characterization and modeling of actual fuels to improve simulations of fuel performance. Work supported under DOE/NE Agreement # DE-FC07-05ID14654.

Minerals, Metals and Materials under Pressure: Electronic, Magnetic and Optical Properties of Materials under High Pressure

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Richard Hennig, Cornell University; Dallas Trinkle, University of Illinois; Ellen Cerreta, Los Alamos National Laboratory

Tuesday AM — Room: 385
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Richard Hennig, Cornell University; Dallas Trinkle, University of Illinois

8:30 AM Invited
Exotic Pressure Effects in Silane to 75 GPa: *Arthur Ruoff*[1]; Chandrabhas Narayana[1]; Raymond Greene[2]; [1]Cornell University; [2]Corning, Inc.

Two groups of theorists have calculated the crystal structures of silane at high pressures and have predicted that those present at the highest pressure will become metallic. Their lowest enthalpy structures (infinite mass approximation) do not agree with each other or with experiment. It has also been suggested that metallic silane may be a high Tc superconductor. Our experimental work finds that at pressures from 7 to 40 GPa silane has a monoclinic structure (point group 14). Silane at 298K is thermodynamically unstable with deltaG super zero = 56,900 J/mole. We have obtained deltaG(P) which becomes negative at 4 GPa but reverts to being positive at 16 GPa. When using stainless steel gaskets the samples become black beginning next to the gasket at 20 GPa (with no raman signal of H2). No Si-H or H2 vibrons are present. This phase is an excellent shiny metal based on reflectivity studies.

9:00 AM Invited
Theoretical Aspects of Light-Element Alloys under Pressure: *Ji Feng*[1]; Richard Hennig[2]; Neil Ashcroft[2]; Roald Hoffmann[2]; [1]Cornell University, Harvard University; [2]Cornell University

Nothing is more fundamentally chemical than compound formation. Yet some elements are quite reluctant to bind, at least under normal conditions of temperature and pressure. An archetypical example is the beryllium (Be) and lithium (Li) system; these two lightest of all metallic elements in the condensed state are immiscible, and form no binary alloy. Here we report a computational study indicating that the reactivity of Be and Li can be fundamentally altered by pressure, leading to the formation of ordered alloys. We also show that in these compounds, the valence electrons reside in unexpected and quite striking 2-dimensional free-electron-gas-like states embedded in a 3-dimensional crystalline environment. Along with other examples, a general electronic structure viewpoint, which unifies some important aspect of the reactivity, electronic structure and geometric structure concerning light-element alloys under pressure, will be discussed.

9:30 AM Break

9:45 AM Invited
Recent Studies in Superconductivity at Extreme Pressures: *James Schilling*[1]; [1]Washington University

High pressure experiments play an important role in the field of superconductivity in three primary ways:[1] (a) increase the number of known superconductors and enhance their transition temperatures to record values, (b) serve as a guide in the synthesis of materials with superior superconducting properties at ambient pressure, and (c) give information on the pairing interaction and allow a quantitative test of theory. In this talk I will discuss recent experimental results on the alkali metals, Y, CaC6, and high-temperature superconductors which illustrate these possibilities. For example, the number of elemental superconductors has been increased by 22 through the application of high pressure from 30 to 52, including Li, Y, Fe, O, and Si. Recently the transition temperature of Y metal has been pushed by 1.2 Mbar pressure to 20 K and that of Ca by 1.6 Mbar to 25 K, a record value for an elemental superconductor. Such enormous pressures are sufficient to even destroy the free-electron character of the conduction electrons in the alkali metals, as predicted by Neaton and Ashcroft,[2] which is why both Cs and Li become superconducting. Comparison of parallel studies on the trivalent d-electron elements Sc, Y, La, and Lu reveals an interesting correlation between Tc and the free volume available to the conduction electrons outside the ion cores, as suggested many years ago by Johansson and Rosengren.[3] [1]J. S. Schilling, Ch. 11 in "Handbook of High Temperature Superconductivity: Theory and Experiment", editor J. R. Schrieffer, associate editor J. S. Brooks (Springer Verlag, Hamburg, 2008), p. 427-462. [2]J. B. Neaton and N. W. Ashcroft, Nature 400, 141 (1999); ibid., Phys. Rev. Lett. 86, 2830 (2001). [3]B. Johansson and A. Rosengren, Phys. Rev. B 11, 2836 (1975).

10:15 AM
First-Principles Calculations of High-Coordinated Group IV Metal Dioxides: *Varghese Swamy*[1]; Barry Muddle[1]; [1]Monash University

High-coordinated Ti, Zr, and Hf dioxides have received significant attention as potential ultrastiff/ultrahard substances. Cotunnite-TiO_2 with nine-fold metal-oxygen coordination synthesized at ~60 GPa represents the stiffest and hardest of the oxides identified to date. Other proposed high-density structures (stable at high-pressures) include an orthorhombic phase, a pyrite phase, and a fluorite phase. Good agreements have been obtained between experimental and ab initio results on the elastic properties of most of the phases; however, a major discrepancy has emerged between the available experimental and theoretical elastic data on pure and Zr-substituted cubic TiO_2. Here we present the results of our first-principles calculations with a focus on the elastic properties of the cubic phases. We used density-functional and hybrid density-functional –Hartree-Fock approaches. A detailed comparison of the effects of the functionals used on the computed elastic properties as well as that between the theoretical and experimental data will be presented.

10:35 AM Break

10:50 AM Invited
Ex Nihilo Determination of Structures by Random Searching: Richard Needs[1]; *Chris Pickard*[2]; [1]University of Cambridge; [2]University of St. Andrews

We introduce a search strategy for predicting structures based on relaxation of randomly chosen structures under the forces and stresses obtained from ab initio density functional theory (DFT) computations. By starting from random structures we aim to obtain a wide coverage of the "structure space", which allows for the possibility of finding radically new structures which have not been considered previously. Our search strategy is remarkably straightforward and does not require the selection of highly-system-specific parameter values. Tests indicate that our strategy works very well for systems with up to of order 10 atoms, which allows us to address many interesting problems. We illustrate the performance of the method with applications to various systems under high pressures, including solid hydrogen[1] where we find a new candidate structure for phase III,[2] silane where we find electron deficient bonds at high pressures, and aluminium hydride where we find high-pressure insulating and metallic phases.[3] [1]"Structure of phase III of hydrogen", C. J. Pickard and R. J. Needs, Nature Physics 3, 473 (2007). [2]"High-pressure phases of silane", C. J. Pickard and R. J. Needs, Phys. Rev. Lett. 97, 045504 (2006). [3]"Metallisation of Aluminium Hydride at High-Pressures", C. J. Pickard and R. J. Needs, submitted for publication.

11:20 AM Invited
Superconductivity in Hydrogen Dominant Materials: Silane: *Mikhail Eremets*[1]; I. Trojan[1]; S. Medvedev[1]; [1]Max Plank Institute fuer Chemie

The fundamental problem of metallization of hydrogen requires currently experimentally inaccessible pressures of P>400 GPa. The dense group IVa hydrides has recently attracted considerable attention because hydrogen in these compounds is chemically precompressed. We report experimental observation of the transformation of SiH_4 to the metallic state with P63 structure with high density of hydrogen atoms likely creating a three-dimensional network. In addition, we found that silane becomes superconducting with Tc=17 K at 96-120 GPa. These findings support the idea of modeling metallic hydrogen by hydrogen-rich compounds.

Neutron and X-Ray Studies for Probing Materials Behavior: Phase Transitions and Beyond

Sponsored by: National Science Foundation, The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter K. Liaw, University of Tennessee

Tuesday AM — Room: 391
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Yang Ren, Argonne National Laboratory; Donald Brown, Los Alamos National Laboratory

8:30 AM Keynote
Dynamic PDF Method: A Novel Technique to Study Local Dynamics: *Takeshi Egami*[1]; Wojtek Dmowski[1]; [1]University of Tennessee

Neutron scattering is a powerful method to study excitations in solids, such as phonons and spin waves (magnons). But when the solid is disordered, as in liquids and glasses, these excitations are scattered by the structure, and have short coherence. Thus the peaks in the dynamic structure factor, S(Q, E), become broad, and it is difficult to determine their dispersion. We propose a new technique of dynamic pair-density function (DPDF), which is obtained by Fourier-transforming S(Q, E) over Q. DPDF is a two-dimensional function of r (atomic distances) and E (frequency), and registers only localized or semi-localized phonons and magnons, while propagating phonons and magnons are not observed. Thus it is a complementary technique to the conventional approach. Using the Pharos spectrometer of Los Alamos NL to obtain S(Q, E), we demonstrate how the DPDF method captures the saddle-point phonons in nickel, and local phonons in relaxor ferroelectric Pb(Mg1/3Nb2/3)O3 (PMN).

9:00 AM Invited
High-Energy Synchrotron X-Ray Study of Magnetic-Field-Driven Structural Transformation and Its Role in the Colossal Magneto-Electric, -Elastic and -Caloric Effects: *Yang Ren*[1]; Yandong Wang[2]; Sergio Gama[3]; Feng Ye[4]; Peter Liaw[5]; Dennis Brown[6]; [1]Argonne National Laboratory; [2]Northeastern University; [3]University of Campinas-UNICAMP; [4]Oak Ridge National Laboratory; [5]University of Tennessee; [6]Northern Illinois University

One of the emerging features of many materials with interesting properties, like high temperature superconductivity, relaxor ferroelectricity, colossal magneto-resistivity, magnetic shape-memory effect, and multiferroics, is a propensity to form inhomogeneous structures on a variety of length scales, due to micro- or macro-scopic phase separation. It is of fundamental importance to study those structural inhomogeneities and their real-time responses to the external stimuli. Exploiting advantages of synchrotron high-energy photons, we have been developing experimental facilities for high-energy x-ray study of structural changes of samples under multiply environments combining high/low temperature, high pressure, high magnetic field and electric field. In this talk, we will present some recent results on the high-energy synchrotron x-ray study of materials under high magnetic field. We demonstrate that the magnetic-field-driven structural transformation plays a key role in the physical and mechanical properties of the materials including correlated electron systems, inter-metallic compounds, and magnetic shape memory alloys.

9:25 AM
In-Situ Study on Pressure-Induced Microstructure Evolution of Ferromagnetic Shape-Memory Alloys: *Gang Wang*[1]; Yandong Wang[1]; Yang Ren[2]; Yandong Liu[1]; Liang Zuo[1]; [1]Northeastern University; [2]Argonne National Laboratory, X-Ray Science Division

Full information on crystallographic aspects during the phase transformation is essential for understanding the 'memory' characteristics in the ferromagnetic shape-memory alloys (FSMA) related to texture and stress. In the present paper, the detailed local information of microstructure of Ni_2MnGa alloy under different pressure was observed in-situ on the high energy synchrotron beam line 11-ID-C of APS. The transformation between parent phase and martensite phase was traced by means of the instantaneously recorded 2D images of diffraction results. The characteristics of orientation variation are analyzed to give a detailed description of the pressure-induced phase transformation process of Ni_2MnGa. According to these results, the mechanism of phase transformation of Ni2MnGa induced by pressure is illuminated, which can enrich our knowledge for controlling the microstructure and performances of FSMA.

9:45 AM Invited
X-Ray Scattering Studies of Semiconductor Nanoclusters in Zeolites: *A. M. Milinda Abeykoon*[1]; Miguel Castro-Colin[2]; M. N. Iliev[1]; W. Donner[1]; A. J. Jacobson[1]; S. C. Moss[1]; [1]University of Houston; [2]University of Texas

When electrons and holes in a semiconductor are confined to ultra-small regions of space (typically 1-25 nm), the optical and electronic properties become strongly size-dependent. Such structures are called quantum dots, nanowires or nanoclusters, depending upon their shape and dimensionality. These nanostructures are of great interest for a variety of potential electronic, photochemical and nonlinear optical applications and are necessary for an analysis of the transition from molecular to bulk semiconductor properties. This talk will discuss the structure of HgSe and Se semiconductor nanoclusters encapsulated in both Nd-Y (spherical pore) and LTL (tubular pore) zeolites. The molecular structure of these systems was modeled by performing a Rietveld refinement on X-ray Bragg data. A remarkable feature in our X-ray diffraction patterns, continuous diffuse scattering under the Bragg peaks, will also be discussed along with forthcoming PDF results. We use the results of optical studies to complement our X-ray structural work.

10:10 AM Invited
The Role of Chemical and Displacement Pair Correlation in the Determination of Higher Order Correlation: *Donald Nicholson*[1]; R. Barabash[1]; Y. Puzyrev[1]; Gene Ice[1]; [1]Oak Ridge National Laboratory

Scattering experiments are routinely used to measure atomic pair correlation. Only in special cases can model structures that reproduce the pair correlation be used to unambiguously determine higher order correlations. However, the requirement that measured chemical and displacement short range order be reproduced by alloy models constrains the possible higher order correlations. A strategy aimed at further specification of higher correlations by augmenting measured pair correlations with first principles results will be discussed. This work sponsored by DOE-OS through the Office of Basic Energy Sciences (BES) was performed at Oak Ridge National Laboratory, which is managed by UT-Battelle, LLC under Contract No. De-AC05-00OR22725.

10:30 AM Break

10:40 AM
Strain-Induced Phase Transformation in a Cobalt-Based Superalloy during Different Loading Modes: *Michael Benson*[1]; Peter K. Liaw[1]; Hahn Choo[1]; Don Brown[2]; Mark Daymond[3]; Dwaine Klarstrom[4]; [1]University of Tennessee; [2]Los Alamos National Laboratory; [3]Queens University; [4]Haynes International Inc

The strain-induced face-centered-cubic (FCC) -> hexagonal-close-packed (HCP) phase transformation in a cobalt-based superalloy was investigated with four in-situ loading neutron-diffraction experiments: monotonic tension, monotonic compression, high-cycle fatigue, and low-cycle fatigue. The experiments revealed relevant characteristics about the phase transformation. The transformation onsets for the four respective cases were 685 MPa, 700 MPa, 1 cycle, and 3 cycles. The accumulation rates for the tension and

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compression cases were 0.1 g-MPa^{-1} and 0.05 g-MPa^{-1}, respectively, throughout the experiment. For the cyclic-loading cases, the accumulation rates were found to be inversely proportional to the number of fatigue cycles. A criterion of tensile plastic work is proposed to describe the observed results.

10:55 AM
New Strain Path to Inducing Phase Transition in PTFE Measured by Neutron Diffraction: *Eric Brown*[1]; Philip Rae[1]; Dana Dattelbaum[1]; Donald Brown[1]; Bjorn Clausen[1]; [1]Los Alamos National Laboratory

A novel application of in situ neutron diffraction under applied uniaxial strain is presented; measuring the crystalline domain evolution in a semi-crystalline polymer under bulk deformation. PTFE is shown to respond to uniaxial deformation by undergoing a crystalline phase transition previously believed to occur only at very high hydrostatic pressure. Discovery of this phase transition under applied uniaxial-strain fundamentally changes our understanding of the deformation mechanisms in semi-crystalline polymers and how they need to be modeled. Under compression parallel to the basal plane normal (i.e., parallel to the molecular axis) the modulus is ~1000× bulk dominated by intra-polymer chain compression, providing experimental validation of theoretical predictions. Deformation parallel to the pyramidal plane normal exhibit both axial and transverse strains of the opposite sign as the applied load, suggesting the crystalline lattice is accommodating deformation by shearing along the prismatic planes.

11:15 AM
Analysis of Chemical Segregation in Nickel Based Super Alloys Using Neutron Diffraction: *Jaimie Tiley*[1]; [1]US Air Force

Neutron Diffraction techniques were used to determine chemical segregation and crystal parameters in several nickel super alloys. The results validate crystal models used for determining occupancy sites and amounts within both gamma and ordered gamma prime systems with as many as 8 elements. Combining the techniques with advanced three dimensional analysis tools including atom probe tomography and transmission electron microscopy provides powerful tools for characterizing microstructures and atomic behavior. In addition, neutron diffraction techniques were used to determine volume fractions, thermal expansion terms and chemical segregation/phase changes for ordered gamma prime as a function of temperature.

11:35 AM
Texture Evolution in the Ni-Mn-Ga Ferromagnetic Shape-Memory Alloys Studied by Neutron Diffraction Technique: *Z.H. Nie*[1]; Y.D. Wang[1]; G.Y. Wang[2]; J.W. Richardson[3]; G. Wang[1]; Y.D. Liu[1]; P.K. Liaw[2]; L. Zuo[1]; [1]Northeastern University, Key Laboratory for Anisotropy and Texture of Materials (MOE); [2]University of Tennessee, Department of Materials Science and Engineering; [3]Argonne National Laboratory, Intense Pulsed Neutron Source

The initial and transformation textures in the as-cast Ni-Mn-Ga ferromagnetic shape-memory alloys were studied by the time-of-flight (TOF) neutron diffraction technique. The neutron diffraction experiments were performed on General Purpose Powder Diffractometer (Argonne National Laboratory). Inverse pole figures were extracted from the neutron data for determining the orientation distributions of polycrystalline Ni-Mn-Ga rods before and after the uniaxial compression deformation. Texture analysis reveals that the initial texture in the as-cast Heusler state composed of {110}<001> and {110}<011>, which was weakened after the deformation. Moreover, a strong preferred selection of martensitic variants ({110}<001> and {100}<001>) was observed in the transformed martensite after a compression stress was imposed along the axial direction on the Heusler phase. The preferred selection of martensitic variants can be well explained by considering the grain/variant-orientation-dependent Bain distortion energy under the stress field applied during the phase transition.

11:50 AM
Defects Induced Conversion of a First-Order Bulk Phase Transition to a Tricritical in the Near Surface Layers of V_2H: A. Korzhenevskii[1]; *Rozaliya Barabash*[2]; J. Trenkler[3]; K. E. Bassler[4]; G. F. Reiter[4]; S. Moss[4]; [1]Russian Academy of Science, Institute of Problems of Mechanical Engineering; [2]Oak Ridge National Laboratory; [3]Carl Zeiss SMT AG; [4]University of Houston, Department of Physics and Texas Center for Superconductivity

In our pursuit of the "two-length-scale" issue measured in a single domain single crystal of V_2H, a conversion from a strong 1-st-order phase transition in the bulk to a continuous transition in a defective near-surface region ("skin layer") we have employed high- and low-energy x-ray diffraction in transmission and reflection geometry, respectively. The conversion appears to be driven by the depth-dependent change in the mosaic spread associated with the surface-enhanced dislocation walls, which act to nucleate an ordered phase, yielding a depth-dependent decay of the correlation length and effective local critical temperature TC. Related aspects of the data were also measured, such as strongly anisotropic diffuse scattering in the defective "skin layer" but isotropic scattering in the bulk, and a "crossover behavior" from mean field (tricritical) exponents (susceptibility and correlation range) closer to TC, to larger, non-universal values, further from TC. Supported in Houston by the DOE/BES—DMS.

12:10 PM
***In Situ* Neutron Diffraction Study of B_2 Ordered FeCo under Compressive Loading**: *Saurabh Kabra*[1]; Donald Brown[1]; Easo George[2]; [1]Los Alamos National Laboratory; [2]Oak Ridge National Laboratory

FeCo magnetic alloys are limited in their applicability because of brittleness under tensile stress. The brittleness of B_2 ordered alloys in general and ordered FeCo in particular is an unexpected phenomenon given the high symmetry of the B_2 crystal structure and the abundance of <111> type directions and {110} type planes for ½ <111>{110} slip. In order to study the deformation mechanism in these alloys, *in situ* neutron diffraction experiments were performed at the SMARTS diffractometer at Los Alamos Neutron Science Center. The diffraction pattern showed many anomalous changes during loading. Along with unusual changes in peak positions, the most notable changes occurred in the intensities of FeCo peaks. As a general trend, the fundamental peaks increased in intensity while the superlattice peaks decreased. These intensity changes along with supporting experiments (texture on HIPPO diffractometer and metallography) show that APB induced disorder is introduced as the sample is loaded.

Particle Beam-Induced Radiation Effects in Materials: Metals III, RIS and Multilayers

Sponsored by: The Minerals, Metals and Materials Society, American Nuclear Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee
Program Organizers: Gary Was, University of Michigan; Stuart Maloy, Los Alamos National Laboratory; Christina Trautmann, Gesellschaft fur Schwerionenforschung; Maximo Victoria, Paul Scherrer Institute and Lawrence Livermore National Laboratory

Tuesday AM — Room: 389
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Todd Allen, University of Wisconsin-Madison; Gary Was, University of Michigan

8:30 AM Invited
Tomographic Atom Probe Analyses of Grain Boundaries in Ion Irradiated Nanostrucutured Austenitic Stainless Steels: Auriane Etienne[1]; Bertrand Radiguet[1]; *Philippe Pareige*[1]; [1]Rouen University

The irradiation-assisted stress corrosion cracking of austenitic stainless steel is linked to radiation induced segregation at grain boundaries. Thus an atomic scale observation of these segregation is of primary importance to the understanding of the mechanism of segregation under irradiation and to give experimental data to be compared to computer simulation. In this work, austenitic steels have been nanostructured (grain size of few nanometres) and ion irradiated at 350°C. The segregation and depletion of solute at grain boundaries were characterized at the atomic scale with the Tomographic Atom Probe and are compared to simulation and experimental (with other technique) results. This work focused on grain boundary studies and the joint paper (same session) dedicated to dislocation loops and associated solute segregation give an atomic scale description of irradiation induced segregation in austenitic steels.

9:10 AM
Radiation Induced Segregation in 304 and 316 Austenitic Stainless Steels: Study by Tomographic Atom Probe, Transmission Electron Microscopy and Rate Theory Modelling: Auraine Etienne[1]; Philippe Pareige[1]; *Bertrand Radiguet*[1]; [1]Rouen University

The internal structures of pressure water reactor made of Austenitic Stainless steels and are submitted to a high neutron flux. This irradiation induces a degradation of mechanical properties of these materials due to a change of the microstructure under irradiation. A study the evolution of the point defect population and the intragranular induced segregation of solute species under irradiation has been performed. Tomographic Atom Probe (TAP) and Transmission Electron Microscope (TEM) were performed on 304 and 316 steels samples that have been irradiated with 160 keV Fe+ ions at different doses at 350°C. At the same time a rate theory model has been used to predict the evolution of the point defect population under irradiation. A correlation between point defects clusters and solute clusters will be discussed.

9:30 AM
***Ab Initio*-Based Radiation-Induced Segregation Modeling in Fe-Ni-Cr Alloys**: *Julie Tucker*[1]; Todd Allen[1]; Dane Morgan[1]; [1]University of Wisconsin

High concentrations of point defects, such as those created in radiation environments, can cause severe material degradation as they migrate and cluster. Radiation induced segregation (RIS), the process by which the local composition of an alloy is altered near point defect sinks, is a phenomenon that has concerned the nuclear industry for decades. While substantial progress has been made in the area of RIS prediction by empirical fitting, many questions remain about the diffusion mechanisms of point defects and how they are affected by local environment changes in multi-component alloys. This research uses *ab initio* methods to determine diffusion coefficients associated with both vacancy and interstitial migration in Ni rich fcc Fe-Ni-Cr alloys. We find that the alloy kinetics differs significantly from that predicted by simple extrapolation of empirical fits to high-temperature data. The calculated diffusion coefficients are used to parameterize a rate theory type RIS model.

9:50 AM
Understanding Oversized Solute Effects on Radiation-Induced Segregation in Austenitic Alloys: *Micah Hackett*[1]; Jeremy Busby[2]; Gary Was[1]; [1]University of Michigan; [2]Oak Ridge National Laboratory

The addition of oversized solutes to austenitic stainless steels reduces radiation-induced segregation (RIS) of chromium. Zr or Hf was added to 316-type stainless steels and then irradiated with 3.2 MeV protons at temperatures of 400 and 500°C to several different doses. Measurements of the grain boundary RIS show Zr to be more effective than Hf at reducing RIS. The relative effectiveness of Zr vs. Hf is associated with a difference in vacancy binding energy as determined by ab initio calculations. Rate theory modeling demonstrates that the difference in RIS results is accounted for by the change in vacancy binding energy. The oversized solute effect disappears at higher doses due to a loss of solute for the trapping and recombination of point defects, which are responsible for the reduction in RIS.

10:10 AM Break

10:30 AM
Incoherent Interface Structure and the Fast Recombination of Irradiation-Induced Frenkel Pairs in CuNb Multilayer Composites: *Michael Demkowicz*[1]; R. Hoagland[1]; A. Misra[1]; [1]Los Alamos National Laboratory

In contrast to vacancies or interstitials in single crystals of Cu or Nb, point defects that have been absorbed at a CuNb interface do not remain compact and well-localized. Instead, they spread out within the interface by transforming the interface structure in the neighborhood of the site at which they were absorbed. Since the effective size of such interfacial defects is notably larger than that of the corresponding defects in single crystal Cu or Nb, the critical distance for Frenkel pair recombination is significantly increased at CuNb interfaces. Random walk simulations are used to demonstrate that this phenomenon predicts a reduction of radiation damage accumulation in CuNb multilayer composites, in agreement with experimental findings. This work was supported by the Los Alamos National Laboratory Directed Research and Development Program (LDRD) and the Los Alamos National Laboratory Director's Fellowship Program.

10:50 AM
Evolution of Microstructure and Mechanical Properties of Cu/V Nanolayers Subjected to Helium Ion Irradiation: *Engang Fu*[1]; Jesse Carter[1]; Michael Martin[1]; Lin Shao[1]; Haiyan Wang[1]; Stuart Maloy[2]; Ning Li[2]; Xinghang Zhang[1]; [1]Texas A&M University; [2]Los Alamos National Laboratory

Sputter-deposited Cu/V nanolayer films with individual layer thickness varying from 1 to 100 nm have been subjected to 50 keV helium ion irradiation at room temperature with a dose of $6x10^{16}$ He ions/cm^2. The evolution of microstructure and mechanical properties after ion irradiation has been investigated. Significant hardness enhancement has been observed in Cu/V with individual layer thickness of 10 nm or greater, whereas hardnesses barely change in radiated Cu/V with smaller layer thickness. Helium bubbles, 1-2 nm in diameter, induced by ion irradiation, were observed both within the layers and along layer interfaces. Dependence of radiation hardening on layer thickness will be discussed based on the evolution of microstructures (He bubbles), and interaction of radiation induced defects with layer interfaces.

11:10 AM
Nanostructure Fe/W Multilayers Subjected to Helium Ion Irradiation: *Nan Li*[1]; Engang Fu[1]; Haiyan Wang[1]; Amit Misra[2]; Richard G. Hoagland[2]; Jesse J. Carter[1]; Michael Martin[1]; Lin Shao[1]; Xinghang Zhang[1]; [1]Texas A&M University; [2]Los Alamos National Laboratory

We report on the evolution of microstructure and mechanical properties of Fe/W multilayers, with incoherent bcc/bcc interface, subjected to helium ion irradiation. Fe/W multilayer films with individual layer thickness, ranging from 1 nm to 200 nm, were deposited by magnetron sputtering technique, followed by helium ion-irradiations. Peak helium concentration approaches a few atomic percent with 5–7 displacement-per-atom in Fe or W. He bubbles, 1-2 nm in diameters, were observed with helium bubbles aligned along layer interfaces. Film hardness varies after ion implantation and the magnitude of hardness variation depends on the layer thickness of multilayer films. He bubbles and the interfaces may play an important role in hardness enhancement.

11:30 AM
Does Radiation Induced Ordering/Segregation Influence Properties of FeCr Alloys?: *Alfredo Caro*[1]; P. Erhart[1]; G. Bonny[2]; M. Gilbert[3]; J. Marian[1]; M. Caro[1]; [1]Lawrence Livermore National Laboratory; [2]Lawrence Livermore National Laboratory and Reactor Materials Research Unit; [3]EURATOM/UKAEA Fusion Association, Culham Science Centre

A breakthrough in the understanding of the evolution of the microstructure of FeCr alloys under irradiation was made some years ago through neutron diffraction measurements of irradiated/annealed samples which showed negative Cowley short-range order, SRO, parameters at small Cr concentrations, indicating strong ordering of Cr atoms, and an inversion of the sign with increasing Cr concentration, suggesting the formation of α'-precipitates. The implications of these discoveries, and the numerous works that they motivated, on the interpretation of enhanced creep resistance and embrittlement are apparent since SRO is known to affect the mobility of dislocations, and precipitation plays a role in intergranular cracking. In this work we explore the implications of SRO in several properties of the alloy, namely: i- how does SRO affect the phase diagram of the alloy?, ii- how does SRO affect dislocation mobility?, and iii- how does SRO affect the precipitation of α'? Using Molecular dynamics and Monte Carlo simulation we address these issues using a classical representation for the interatomic interactions that properly describes experimental and ab initio findings regarding SRO. We arrive then at a quantitative interpretation of radiation induced ordering/segregation in this alloy.

11:50 AM
Effects of Helium on the Irradiation Induced Microstructure in Iron: *Maria Okuniewski*[1]; Chaitanya Deo[2]; Marc Weber[3]; Farida Selim[3]; Kelvin Lynn[3]; Peter Hosemann[4]; Soenke Seifert[5]; Srinivasan Srivilliputhur[6]; Stuart Maloy[6]; Michael Baskes[6]; Michael James[6]; James Stubbins[1]; [1]University of Illinois at Urbana-Champaign; [2]Georgia Institute of Technology; [3]Washington State University; [4]Los Alamos National Laboratory and Montanuniversität; [5]Argonne National Laboratory; [6]Los Alamos National Laboratory

A systematic group of coordinated experiments and computational modeling was carried out to investigate the microstructural effects of He in irradiated Fe. Single crystal bcc Fe was implanted with varying concentrations of He, up to 450 appm. These specimens were subsequently irradiated with 1 MeV protons at 300 and 450°C. The He-to-damage ratios (appm/dpa) were selected to be comparable with fusion systems (10 appm/dpa) as well as accelerator driven systems (150 appm/dpa). The effects of temperature, dose, and He-to-damage ratios on microstructure and mechanical properties were examined. The experimental analyses included in-situ and ex-situ TEM, positron annihilation spectroscopy, small angle x-ray scattering, and nanoindentation. These experimental results are compared to modeling results obtained via kinetic Monte Carlo techniques.

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials VII: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Sinn-wen Chen, National Tsing Hua University; Srinivas Chada, Medtronic; Chih Ming Chen, National Chung-Hsing University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-Ho Lee, Hong Ik University; Kejun Zeng, Texas Instruments Inc; Katsuaki Suganuma, Osaka University

Tuesday AM — Room: 278
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Katsuaki Suganuma, Osaka University; Sinn-wen Chen, National Tsing Hua University

8:30 AM Invited
The Growth of Semiconductor Nanowires for Research or Industrial Purposes: Djamila Hourlier[1]; Patricia Lefebvre-Legry[1]; *Pierre Perrot*[1]; [1]National Center of Scientific Research

Silicon or germanium-based nanowires are the leading candidates for the building-blocks in next generation nanoelectronic devices. The growth of these nanowires is commonly described either by the Vapor-Liquid-Solid (VLS) or Solid-Liquid-Solid (SLS) processes. In this study a new approach to the problem of nanowire formation which does not violate thermodynamic principles is presented. We will show that many aspects of the growth mechanisms, such as the surface curvature of the solid phase (nano or microscopic range), in equilibrium with the liquid phase are of extreme interest. With the variety of cases encountered in this study, we are now in a position to treat any size liquid alloy (Au-Si or Au-Ge) in equilibrium with any solid (nano or macroscopic size). By using a method to date neglected, and by choosing the SLS process rather than the VLS process, we succeeded in answering the thorny question of the size limit of nanowires.

8:50 AM
Effect of Interface Structures on Melting and Solidification Behavior of Bismuth Nano-Particles Embedded in an Icosahedral Phase Matrix: *Alok Singh*[1]; AnPang Tsai[2]; [1]National Institute for Materials Science; [2]Tohoku University

Effect of interfaces on stability of nano-particles has been brought out by creating novel interfaces of bismuth nano-particles in an Al-Cu-Fe matrix. By heat treatment, three states of matrix phase were obtained, altering the nature of interfaces. Interface structures were studied by TEM and melting and solidification behavior was studied by differential scanning calorimetry (DSC). Earlier studies on bismuth nano-particles embedded in crystalline matrices such aluminum and zinc have reported a lowering of melting temperature of bismuth. We show that, when embedded in the icosahedral phase, a superheating is possible. This is because, as we show, bismuth has a better lattice match with icosahedral phase than with any crystalline phase, and therefore makes matching interfaces.

9:05 AM
Controlled Growth of Atomic-Scale Si Layer with Huge Strain in the Nano-Heterostructure NiSi/Si/NiSi through Point Contact Reaction between Nano-Wires of Si and Ni: *Kuo-Chang Lu*[1]; Wen-Wei Wu[2]; Han-Wei Wu[2]; Carey Tanner[1]; Jane Chang[1]; Lih Chen[2]; K. N. Tu[1]; [1]University of California, Los Angeles; [2]National Tsing Hua University

The point contact reaction between a Si nanowire and a Ni nanowire is reported where the Si nanowire is transformed into a bamboo-type grain of single crystal NiSi from both ends. The reaction was investigated in-situ in an ultra-high vacuum transmission electron microscope. It is proposed that the epitaxial growth of the NiSi is limited by the dissolution rate of Ni into Si at the point contact interface and assisted by interstitial diffusion of Ni atoms within the Si nanowire. Based on the point contact reaction, single crystal NiSi/Si/NiSi heterostructures have been fabricated, in which more than 12% of strain was found in the middle Si layer, the dimension of which can be controlled down to 2 nm, overcoming the limit of conventional patterning process. This study is expected to support the realization of nanoscale electronic devices.

9:20 AM
Formation of the <-1010> /{-12-10} Side Bands-Modulated Structures Due to a Periodic Modulation in the Composition of Mn in Illmenite-Mn_2O_3 Alloys: *Mohammad Shamsuzzoha*[1]; Chandan Srivastava[1]; Pranoti Kale[1]; Padmini Periaswamy[1]; Raghavendra Pandey[1]; [1]University of Alabama

Conventional and analytical transmission electron microscopy techniques have been applied to study the microstructure, phase transition and chemical composition of the illmenite-Mn alloys containing 0-40wt % of Mn and prepared by standard ceramic processing. Bright field electron microscopy revealed that the alloy nominally containing 35-wt% of Mn (an electronic material)exhibits a side bands-modulated structure. Electron diffraction patterns of the modulated structure revealed that the side bands in the sample have the <-1010> /{-12-10} growth habit, and are formed due to the variation in contrast resulted by a periodic but unidimensional variation of interplanar spacings. TEM/EDS line profiles across and normal to these side bands showed a sinusoidal variation in the composition of Mn. The modulated structure is suggested to be caused by a decomposition in the supersaturation of the solid solution of Mn in the lattices of hexagonal illmenite structure.

9:35 AM
Cu with Insoluble MoN for Advanced Barrier-Free Metallization: Jinn P Chu[1]; *Chon-Hsin Lin*[2]; V.S. John[1]; [1]National Taiwan University of Science and Technology; [2]Chin-Min Institute of Technology

As a diffusion barrier is required for copper metallization in microelectronics, searching a new and reliable barrier material with thickness less than 5 nm becomes very challenging when the device feature size continues to shrink beyond 45 nm. Thus, advanced barrier-free Cu metallization is proposed. In this study, a copper seed layer was generated by doping an insoluble MoN, which can improve the thermostability of copper and further inhibit the crystal growth. In addition, this seed has a barrier property that restrains the inter-diffusion between Cu and Si. The seed layer has been characterized using X-ray diffraction, focused ion beam, secondary ion mass spectroscopy, transmission electron microscope, film resistivity and current-voltage (I-V) measurements. The results indicate notable thermal stability enhancements up to 730°C without any apparent interactions with Si. The detail of the results will be further discussed.

9:50 AM Break

10:00 AM Invited
Phase Transitions in the BiVO4-nPbO System: Structural–Electrical Properties Relationships: *Jean Pierre Wignacourt*[1]; Michel Drache[1]; Pascal Roussel[1]; Olfa Labidi[1]; [1]University of Sciences and Technologies of Lille

In a general survey of nPbO-BiVO4 compounds, interesting phases corresponding to n=2 (Pb2BiVO6) are showing several successive structural transitions α->β->δ. Structures of α and δ forms have been previously described from powder diffraction data (X-rays, neutrons). In this work, we have refined these structures from single crystal data, and the resolution of the intermediate βform, so far unsolved, was possible through a stabilisation thermal cycle; its complete structural understanding required a 4D formalism.Two new polymorphic phases, α' and δ'were obtained by substituting Mn or P for V; their structures are closely related to respectively the α phase at room temperature, and the δ phase at 680°C. Electrical conductivities of all structurally characterized compositions were investigated, and correlations were drawn between their conduction properties and structural characteristics. Conductivity properties measured under variable O2 partial pressures for Pb2Bi(V0.75P0.25)O6 were interpreted as mixed ionic-electronic (p type) conduction mechanism.

10:20 AM Invited
Degradation of Ag-Epoxy Conductive Adhesive/Sn Interface in Humid Atmosphere: *Sun Sik Kim*[1]; Keun Soo Kim[1]; Katsuaki Suganuma[1]; [1]Osaka University

Isotropic conductive adhesives, such as Ag-epoxy pastes, have been continuously studied as ecological alternatives to lead-bearing solders. Ag-epoxy conductive adhesive is known to possess excellent heat-resistance against severe thermal exposure during the reflow process. However, regarding joint reliability, Ag-epoxy conductive adhesive has significant drawbacks under the high temperature and humidity exposure. This exposure causes interfacial degradation of the joints, resulting in higher electrical resistance and reduction of joint strength. There have been several arguments on the degradation mechanism, but no definitive explanation has been achieved yet. The present study focuses on microstructural characterization of the interface between the Sn plating and Ag-epoxy conductive adhesive under 85/85%RH condition. The degradation mechanism is considered through microstructural variation of the interface with exposure time. As a result of HREM and AEM, a nano-scaled tin oxide layer, one of the phases detrimental to joint reliability, was found to be partially formed at the interface.

10:40 AM
Active Evaluation of Solder Joint Microstructures by Noise Spectrum Analysis: *Iver Anderson*[1]; David Rehbein[1]; Joel Harringa[1]; [1]Iowa State University

High power audio equipment with optimum sound quality depends on uncompromised performance from all components and from the solder joints used in their assembly. Analysis of the noise spectrum generated by a single frequency continuous wave signal is being developed for detecting alloy differences in as-solidified solder joints and for “real time” tracking of microstructural evolution, e.g., during thermal aging. Each experiment utilized a standard (AFPB) Cu shear test specimen that was joined with a SAC or SAC + X solder, with testing of as-soldered or thermally aged (150C, up to 1000h) joints. Shifting of characteristic noise peaks was detected from different solder alloys and after each thermal aging treatment, along with stable peaks due to the sample apparatus and specimen geometry. Initial results will be verified and identification of microstructural differences causing peak shifts will be attempted. Supported by Nihon-Superior, Inc., through Ames Lab contract no. DE-AC02-07CH11358.

10:55 AM
A Reliability Test Study of the VFBGA Sn-Ag-Cu Solder Joints: *Huann-Wu Chiang*[1]; Ting-Yu Liu[1]; Yi-Shao Lai[2]; [1]I-Shou University; [2]Advanced Semiconductor Engineering, Inc.

The interfacial reactions of the lead-free Sn-3.0Ag-0.5Cu and Sn-37Pb solder joints bonded on electroplated Ni/Au/Cu pads under multi-reflowed and high temperature storage tests were investigated in this study. Metallurgical evolutions of the intermetallic compounds (IMCs) were scrutinized by scanning electron microscopy/energy dispersive x-ray spectroscopy (SEM/EDS) and electron probe microanalysis (EPMA) mapping. Ball shear and ball impact tests (BIT) were performed to examine strengths of these solder joints as functions of multi-reflow and thermal aging times. The relationship between the interfacial microstructure and the joint strength as well as the failure mode of the fractured joints were then analyzed and discussed.

11:10 AM
Reliability of Rigid PCB/Flexible PCB Joint Bonded Using Ultrasonic Vibration: *Jong Bum Lee*[1]; Ja Koo[1]; Seung Jung[1]; [1]Sungkyunkwan University

Rigid printed circuit board (RPCB) has been broadly used because of its advantages such as low-cost, high reliability and excellent processability. The demand for flexible printed circuit board (FPCB), which has light, high glass transition temperature (Tg) and flexibility, continues to expand with market growth of portable electronics. Electrodes between the RPCB and FPCB have been electrically and mechanically connected by anisotropic conductive adhesive (ACA) and non-conductive adhesive (NCA). However, these bonding methods have disadvantages such as high contact resistance and low reliability. The interest in ultrasonic bonding method is growing to connect the electrodes, because of its higher electrical property, higher reliability, shorter processing time and lower processing temperature than adhesive bonding methods. Therefore, the long-term reliability of RPCB-to-FPCB joint bonded using ultrasonic vibration was investigated with different surface finish. The microstructural observation, contact resistance evaluation and peel test were conducted to investigate the failure behavior of the joints.

11:25 AM
Reliability of 3-D Chip Stacked Package with Through-Chip Interconnects: *Jong-Woong Kim*[1]; Young-Chul Lee[1]; Seung-Boo Jung[1]; [1]Sungkyunkwan University

The 3-dimensional (3-D) chip stacking technology is a leading technology to realize a high density and high performance system in package (SiP). There are several kinds of methods for chip stacking such as die attachment with wire bonding, solder through wafer interconnect technology and via formation and filling technology employing flip chip bonding, etc. Among them the interconnection through Cu filled through-via and die stacking with flip chip technology are considered to be one of the most advanced 3-D packaging technology. Therefore, we have tried to optimize the process of through-via formation and Cu electroplating process for Si die stacking. A Pb-free solder was additionally deposited on the electroless-Ni immersion-Au (ENIG) under bump metallization (UBM) to be used for bonding with another carrier. Finally, the Si die was flip chip bonded with another die, and thermal shock testing was conducted to evaluate the reliability of the final stacked package.

Recycling: Electronics Recycling

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Christina Meskers, Delft University of Technology; Greg Krumdick, Argonne National Laboratory

Tuesday AM — Room: 280
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Greg Krumdick, Argonne National Laboratory

8:30 AM Introductory Comments

8:35 AM
Recycling of Electronic Wastes: Degradation and Combustion: Xiangjun Zuo[1]; *Lifeng Zhang*[2]; [1]Norwegian University of Science and Technology; [2]Missouri University of Science and Technology

Firstly, recycling methods of Printed Circuit Boards (PCBs) were summarized. PCBs mainly contains 72% organic substance and 28% metals in mass. Then PCB materials from a waste printer named were combusted and pyrolysed in TG/DTA and MS machines with the aim of separating and recovering the organic and metallic materials. The current PCB waste materials were combusted at 700°C, 800°C, 900°C under 50ml/min, 100ml/min, 150ml/min with synthetic air atmosphere respectively. The sample was also heated at 800°C under 50ml/min, 100ml/min, 150ml/min under argon gas atmosphere. The mass loss, conversion fraction with the temperature, activation energy etc. are investigated. The main compositions of organic substance are found to be the ethoxyline resin bromide or ethoxyline resin chloridate, and the metals in PCB are mainly copper, solder, iron(ferrite), nickel, silver, gold, and palladium, etc.

8:55 AM
Degradation of Plastics to Liberate Metals from Electronic Waste: *Anne Kvithyld*[1]; Scott Shuey[2]; Sean Gaal[3]; Patrick Taylor[4]; [1]Norwegian University of Science and Technology; [2]Phelps Dodge Corporation; [3]SINTEF Materials and Chemistry; [4]Colorado School of Mines

Printed circuited boards contain considerable metal values. In this study the decomposition of common plastics used in printed circuit boards were studied. Teflon (PTFE), polyvinyl chloride (PVC) and polyethylene (PE) were heated in both CO_2 and argon atmospheres using a thermo-gravimetric furnace (TGA) coupled to a mass spectrometer. Soda ash Na_2CO_3 and baking soda $NaHCO_3$ were used as reactants. The degradation temperatures and evolved gases are presented from single plastics and mixtures. Mixed plastics are shown to be an overlap of the individual plastics. The mass loss temperature is the same in CO_2 and argon. However, less residue is formed when heated in CO_2. The possibilities and difficulties of detecting dioxin and furan formation are also presented.

9:15 AM
Decomposition Process for Polychlorobiphenyl Containing 1 to 5 Chlorine by Using Basic Molten Salts as Reaction Field: *Osamu Takeda*[1]; Takeshi Handa[1]; Masayoshi Kimura[1]; Yasufumi Yokka[1]; Yuzuru Sato[1]; [1]Tohoku University

In order to establish a safe, highly efficient, and simple decomposition process for poly chlorinated biphenyls (PCBs), a decomposition process for PCBs by using basic molten salts as a reaction field was investigated. PCBs containing 1 to 5 chlorine (e.g. 2,2',4,4'-tetrachlorobiphenyl), dissolved in n-nonane solution, was supplied into basic molten salts ($NaOH-Na_2CO_3$ or $KOH-K_2CO_3$) maintained at 500-700°C together with the carrier gas (N_2-21 vol% O_2) through an injection tube. The injected PCBs in a bubble were decomposed by the reaction with the molten salts and oxygen during rise in the molten salts. The decomposition efficiency of PCBs increased with increasing temperature and was achieved to be higher than 99.999% in a certain condition. From a series of systematic study, the effectiveness of the decomposition process for PCBs by using molten salts was demonstrated.

9:35 AM
Establishment of Complete Treatment System of Recyclable Materials Generating from Home Appliance Recycling Plants at Mitsubishi Materials Corporation: *Tetsuro Sakai*[1]; Syogo Yamaguchi[1]; [1]Mitsubishi Materials Corporation

Home appliance recycling law was put into force in April 2004 in Japan. Specified kinds of home appliances, such as air conditioners, television sets, refrigerators and washing machines, should be recycled according to the law. Mitsubishi Materials Corporation (MMC) in cooperation with home appliance manufacturers is operating five home appliance recycling plants in Japan. MMC established complete treatment system of recyclable materials generating from home appliance recycling plants, which is formed by unique collaboration among home appliance recycling plants, copper smelter and refinery, and cement plants. Recyclable materials containing copper and precious metals are only handled and treated within the MMC group. By using this lock-in system, safe treatment and traceability of materials are guaranteed as 100% materials recycle.

9:55 AM
Recovery of Copper from Printed Circuit Boards (PCBs) Scraps by Mechanical Pre-Treatment and Hydrometallurgical Routes: Manis Jha[1]; Jinki Jeong[1]; *Jae-chun Lee*[1]; Min-Suck Kim[1]; Byung-Su Kim[1]; [1]Korea Institute of Geoscience and Mineral Resources

In respect to conventional pyro-metallurgical process to recycle waste PCBs in copper smelter, a feasible hydrometallurgical copper recycling process following pre-treatment, acid-leaching and solvent-extraction has been proposed. The 99.2% of copper was liberated from the PCBs by pre-treatment consisted of shredding, iron separation by hand magnet, stamp milling, air-classification of metallic / resin particles and the separation of fine metallic particles by corona electrostatic separator. The copper was leached from the beneficiated particles of PCBs with 6M H2SO4 at 90°C in 1 hr. The leach liquor was then processed to recover copper using 20% LIX 84I diluted in kerosene. Almost total copper was extracted to organic phase at O/A ratio 1, pH 1.75 and mixing time 5 minutes leaving nickel in the raffinate. The loaded copper from the organic phase could be completely stripped with strong sulphuric acid to recover copper as metal or salt by electrolysis or crystallization, respectively.

10:15 AM Break

10:25 AM
Dissolution Rates of Precious Metal Compounds in Acid: *Hideaki Sasaki*[1]; Masao Miyake[2]; Hisao Kimura[1]; Masafumi Maeda[1]; [1]University of Tokyo, Institute of Industrial Science; [2]University of Illinois at Urbana-Champaign

A precious metal recovery process utilizing active metal vapor was proposed previously. In the process, scraps are treated with the vapor prior to acid leaching in order to make contained precious metals to form compounds that dissolve easily. In this study, dissolution rates of precious metal compounds were measured electrochemically using channel flow double electrode method aiming to inquire what compounds should be formed by the vapor pretreatment. It was revealed that the apparent dissolution rate of Pt was about 100 times higher from Pt-75%Zn than from pure Pt. The dissolution rate of Au-Zn was studied and compared to that of Pt-Zn. Dissolved surfaces of Pt-Zn and Au-Zn compounds were examined by SEM and XRD to consider their dissolution behaviors.

10:45 AM
Treatment of Gold-Containing Technogenous Wastes Tailings by Electrochemical Method, in Condition of "Slight" Oxidation Regime: Tsisana Gagnidze[1]; Teimuraz Royva[1]; Tamaz Lejava[1]; *Manana Mamforia*[1]; [1]R.agladze Institute of Inorganic Chemistry and Electro Chemistry

A processing of technogenous wastes, which involve a gold-containing sulphide minerals, is a potential source to supplement the gold reserves. Hence, searching of ecologically safe and economically reasonable methods for processing of gold-containing technogenous wastes is a topical problem. To increase a parameters of gold extraction and to simplify a technology of the processing of above-mentioned wastes of sulphide minerals, an investigations have been carried out in the direction of the use of the method of electrochemical leaching without any preliminary processing of the mineral. It was established experimentally that an introduction of nitrogen-containing organic additive into chloride solution favours to reduce a Red/Ox potential of the solution from 0,8/1,2 V to 0,350,5V, which permits to conduct the process of direct electrochemical leaching at "soft" oxidation regimes at temperatures 20-30°C without an environmental contamination by molecular chlorine and with a high degree of a gold extraction up to 80-90%.

11:05 AM
Adsorption of Copper from the Effluents of Electronic Industry Using the Cation Exchange Resin Amberlite IR-120 (Na): Nguyen Van Nghiem[1]; *Jae-chun Lee*[1]; Manis Jha[1]; Jae-Min Yoo[1]; Kyoungkeun Yoo[1]; Taek Sung Hwang[2]; [1]Korea Institute of Geoscience and Mineral Resources; [2]Chungnam National University

Copper is widely used as interconnecting material in the electronic industry, inevitably resulting in the generation of large amount of copper bearing waste streams during different processing steps viz. electroplating, etching, rinsing, and chemical and mechanical polishing (CMP) etc. Ion exchange (IX) studies with the solutions containing 300 to 700 ppm copper have been carried out using the cationic exchanger Amberlite IR-120 (Na). Various process parameters viz. contact time, solution pH, resin dose, and acid concentration of eluant were investigated for recovering copper from the effluents. Complete adsorption of copper was achieved at equilibrium pH 2.5 and aqueous/resin (A/R) ratio 100 mL/g in 14 minutes contact time. The mechanism of adsorption of copper was found to follow the Langmuir isotherm and second order reaction rate. The copper was eluted effectively from loaded resin with dilute sulphuric acid to produce copper-enriched solution.

Sloan Industry Centers Forum: Techno-Management Issues Related to Materials-Centric Industries: Session II

Sponsored by: The Minerals, Metals and Materials Society
Program Organizers: Subodh Das, Secat Inc; Diran Apelian, Worcester Polytechnic Institute

Tuesday AM March 11, 2008 — Room: 270 — Location: Ernest Morial Convention Center

Session Chair: Subodh Das, Secat Inc

8:30 AM Introductory Comments by Diran Apelian and Subodh Das

8:40 AM Invited
Technology Management in the Global Steel Industry: Strategy and Organization Models: *Ravi Madhavan*[1]; [1]University of Pittsburgh
As the globalization of the steel industry gathers pace, steel producers are faced with unprecedented strategic and organizational challenges and opportunities, with an emphasis on the creation of multinational structures and processes reflective of the global basis of competition. In particular, developing a technology strategy that is consistent with the global distribution of resources and markets is an important concern for the newly multinational steel company. This presentation will provide an overview of some of the current challenges and opportunities in technology management, as well as suggest appropriate strategic and organizational models for thinking about them.

9:00 AM Invited
Winds of Change and Management Challenges in the Global Steel Industry: *Sanak Mishra*[1]; [1]Mittal Steel India Ltd
The Apparent Steel Use (ASU) in the World is projected at 1.251 billion metric tons in the year 2008, at a healthy growth rate of 6.1%. This is back to back with an estimated growth of 5.9% in 2007 and 8.5% in 2006. In fact the steel industry has had a boom time for about five years now. What is more significant, there have been dramatic changes in the characteristics of the industry, following the emergence of China as largest steel producer as well as consumer, the impact of consolidation processes piloted by large mergers and acquisitions and the attention received by the so-called BRIC countries (Brazil, Russia, India, China), as happening places. Consequent upon these changes, management challenges in the industry are more demanding than ever before and these will be explored in this presentation.

9:20 AM Break

9:30 AM Invited
Powder Metallurgy: The Challenges of a Rapidly Changing Global Industry: *Chickery Kasouf*[1]; [1]Worcester Polytechnic Institute
Powder metallurgy (P/M) produces components for many OEM and higher tier suppliers. As manufacturing growth shifts away from the United States into other economies, especially Asia, North American companies are faced with significant challenges. For example, GKN, the world's largest producer of P/M parts, recently closed four US plants as part of a consolidation in response to the contraction of the US automotive industry. On the other hand, there is tremendous growth in China resulting in many start-ups. Moreover, despite the inhospitable U.S. market, and the requirement for global capabilities, there is still start-up activity in the U.S. due to the low entry barriers in the industry. The challenges for P/M will be discussed in light of the implications for other metal processing industries.

9:50 AM Invited
Techno-Management Issues Facing Metal Processing Industries: *Anne Stevens*[1]; [1]Carpenter Technology
Technology Differentiation has been key to Carpenter's success. Product and Process differentiation are both important to meet our customers requirements. An increased effort is required to improve the availability of a world class engineering talent pool. University-Industry co-operation to increase the funding of metals related research efforts needs to be emphasized. Sourcing and developing the best process technology available worldwide is critical to improving quality and reducing costs. The presentation will discuss several key strategic challenges facing the specialty metals industry.

10:10 AM Invited
North American Paper Industry - Impacts of Evolutionary and Revolutionary TechnoBusiness Developments: *Jacquelyn McNutt*[1]; [1]Georgia Institute of Technology
Looking back ~ The North American Paper Industry experienced a major technical overhaul during the 20th Century ~ largely driven by a focus on technical research. These efforts transformed the industry into a highly efficient, environmentally compliant, and high product quality oriented sector. But sustained business success has still been very elusive in spite of this major technical transformation. Looking Out ~ The North American Paper Industry ~ must find a more successful business platform to avoid major disruptions and dislocations in the years ahead. Yet while the industry is trying to look within to find that internal transformation solution, there are major factors going on in parallel outside the industry that will cause the industry to face a "TechnoBusiness" Evolution if not Revolution in the first part of the 21st Century ~ with our without their own efforts. The noted major new factors both inside and outside the Industry that will drive this agenda, include ~ Global Warming/Dimming, Bio-energy/Bio-fuels, Sustainability, Nanotechnology, Cross Industries/Global/Societal Issues. The purpose of this presentation is to provide a broad overview of both the historical and the looking out factors that have and will shape this vital industry sector.

10:30 AM Invited
North American Paper Industry: Transforming to the Forest Biorefinery: *Paul Stuart*[1]; [1]Abitibi-Consolidated Inc. and Ecole Polytechnique, Department of Chemical Engineering Chair
Commodity industries such as pulp and paper are characterized by a low R&D intensity, which have migrated from process innovation research to enterprise efficiency research (achieved for example through analysis of the supply chain following the implementation of ERP and other systems). Investments in this kind of enterprise efficiency naturally "align" to existing products. Thus commodity industries and especially those that are capital intensive create inertia against a model of "punctuated equilibrium" (where punctual innovations result in new products which add significant value to company performance). The forest biorefinery is a concept being increasingly considered by forestry companies as a strategy to diversify their basic business model; however the implications of this transformation go beyond a requirement for significant capital spending in a cash-limited industry. It is far from obvious how the forestry industry might quickly jump from a manufacturing-centric culture to a product-centric culture. The objective of this presentation will be to summarize some of the key driving forces and barriers to successful commercial implementation of the forest biorefinery.

Structural Aluminides for Elevated Temperature Applications: FE and Other Alumindes

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Young-Won Kim, UES Inc; David Morris, Centro Nacional de Investigaciones Metalurgicas, CSIC; Rui Yang, Chinese Academy of Sciences; Christoph Leyens, Technical University of Brandenburg at Cottbus

Tuesday AM March 11, 2008 — Room: 394 — Location: Ernest Morial Convention Center

Session Chairs: Haruyuki Inui, Kyoto University; David Morris, Centro Nacional de Investigaciones Metalurgicas, CSIC

8:30 AM Invited
The Role of Stable Nanoparticles on Improving the Creep Resistance of Iron Aluminides: *David Morris*[1]; Maria Muñoz-Morris[1]; Ivan Gutierrez-Urrutia[1]; [1]Centro Nacional de Investigaciones Metalúrgicas Consejo Superior de Investigaciones Científicas
The high-temperature creep strength of iron aluminides is relatively low and can be improved by alloying additions and processing improvements,

especially those that lead to large numbers of stable and fine particles. The high temperature creep behaviour has been examined at temperatures of 700°C and above in a recipitation-strengthened Fe-Al-Zr cast alloy and in an oxide dispersion-strengthened Fe-Al-yttria mechanically-alloyed material. In the first, precipitates of an Fe-Zr intermetallic form during preliminary heat treatments or during creep itself. The distribution of the precipitates is modified by the dislocations present during creep, which affects the creep behaviour. The oxide dispersion provides good strengthening up to above 800°C. At 700°C these particles create threshold stresses that lead to very low creep rates. At higher temperatures, general climb allows dislocations to overcome the small obstacles and no threshold stresses are noted.

9:00 AM
In-Situ TEM Analysis of Deformed FeAl-Ni-B (B2) Alloy: *Anna Fraczkiewicz*[1]; Brigitte Decamps[2]; David Colas[1]; Olivier Calonne[1]; Francois Louchet[3]; [1]Ecole Nationale Superieure des Mines; [2]Laboratoire de Chimie Métallurgique des Terres Rares, UPR 209 Centre National de la Recherche Scientifique; [3]Laboratoire de Glaciologie et Geophysique de l'Environnment, Centre National de la Recherche Scientifique

Room temperature brittleness of B2-ordered FeAl remains their main flaw. Different origins of this lack of ductility are considered: hydrogen embrittlement, a high value of the DBTT, as well as superdislocation reactions, leading to the presence of sessile <100>. This work deals with dislocation reactions in a Fe40Al-5Ni-B (at. %) single crystal, in situ TEM strained at 600°C. Specific dislocation configurations were observed: two short, "fork-shaped" segments associated with a straight-lined and highly contrasted segment. These configurations are only partially glissile: only the short segments move under the stress. Still, their evolution is observed, through expansion of individual loops from "fork-shaped" segments. These configurations are formed by reactions between two different <111> superdislocations, gliding on different {112} planes. This mechanism is somewhat similar to that observed previously in FeAl by Munroe and Baker. The possible effects of such dislocation reactions on brittle cleavage fracture of FeAl will be discussed.

9:20 AM
Exothermic Reactions in Cold-Rolled and Physical-Vapor-Deposited Ni/Al Multilayer Foils: *Laszlo Kecskes*[1]; Xiaotun Qiu[2]; Jesse Grater[2]; Jiaping Wang[2]; [1]US Army Research Laboratory; [2]Louisiana State University

The exothermic reaction pathways in cold-rolled and physical vapor deposited Ni/Al reactive multilayer foils were investigated. To understand the conditions approaching self-propagating high-temperature reactions, Ni/Al foils were heated to 1000°C in a differential scanning calorimeter. During thermal explosion, in both types of foils multiple (three to four) exothermic peaks were noted, corresponding to a multi-step reaction process. Determined by a reduced interlayer spacing in the PVD foils, the peak positions were found to sharpen, separate, and shift to lower temperatures. Subsequent to obtaining the total calorimetric heat output for a given heating rate, samples were also heated to each peak temperature to allow identification of the intermediate reaction product and evolution of the microstructure from reactants to steady state products. XRD and SEM showed that lower temperature peaks corresponded to formation of Al3Ni, while the higher temperature peaks corresponded to conversion of Al3Ni to the nominal stoichiometric AlNi.

9:40 AM
Fatigue Crack Growth Resistance of a Fe-40Al Alloy Prepared by Mechanical Alloying and Forging: *Gilbert Henaff*[1]; Naziha Berramdane[1]; Guillaume Benoit[2]; Yannick Girard[3]; Sebastien Launois[4]; [1]Ecole Nationale Supérique De Mécanique Et D´Aérotechnique ; [2]Ecole Nationale Supérique De Mécanique Et D´Aérotechnique; [3]European Aeronautic Defense and Space Company IW; [4]Atomic Energy Commission

The FeAl 40 grade 3 alloy strengthened by oxide dispersion introduced by mechanical alloying presents attractive performances for application in aircraft components at elevated temperature. However several issues relative to its damage tolerance still need to be addressed. The present study tackles this issue, with a special attention paid to the influence of load ratio, temperature, environment and load history. It comes out that the FCG behaviour is only slightly affected when the load ratio is changed in relation with a limited crack closure effect. The relevance of water vapour adsorption, identified on the same alloy tested in the as-hipped condition as the environmentally-assisted mechanism controlling FCG in air, is also analysed. Finally, it is also shown that a periodic overload in a baseline loading at R=0.1 induces a significant retardation effect on crack growth. However this beneficial effect cannot be accounted for by closure effects enhanced by overloads.

10:00 AM
The Yield Strength Anomaly in Fe_2MnAl Single Crystals: *Yifeng Liao*[1]; Ian Baker[1]; [1]Dartmouth

A [411]-oriented single crystal $Fe_{55}Mn_{19}Al_{26}$ was grown using a modified Bridgeman technique and its mechanical behavior investigated as a function of temperature. Specimens were strained under compression over the temperatures range 300-900K at a range of stained rates. A yield strength anomaly was observed in which the yield strength peak was ~180 MPa at 600 K, with the yield strengths ranging from 170 MPa at 300K to 70 MPa at 900K. The yield peak appeared to be little affected by the strain rate. Microstructural analysis in the TEM showed that the compound adopted the $L2_1$ structure at room temperature, but that it transformed to the B2 structure at ~900K. In the $L2_1$ state the superdislocations were four-fold dissociated with the outer partials separation being 4 nm while that of the inner partials was 15nm. The interaction of the gliding dislocations with the thermal anti-phase boundaries was studied by *in-situ* straining.

10:20 AM Break

10:30 AM
First-Principles Prediction of Site Preference of Transition-Metal Elements in B2 NiAl: *Chao Jiang*[1]; [1]Los Alamos National Laboratory

B2 NiAl intermetallic compound is a promising material for high temperature structural applications due to its high melting temperature and excellent oxidation resistance. However, its application is limited by the poor room temperature ductility. One approach to improve the ductility of B2 NiAl is through alloying. A precise knowledge of the site occupancy behavior of ternary elements is thus required to understand their roles in modifying the mechanical properties of NiAl. In this study, we combine first-principles calculations with the Wagner–Schottky model to predict the site preference of 3d, 4d, and 5d transition-metal elements in B2 NiAl. Model calculations indicate that such a preference can be a strong function of both alloy composition and temperature. It is our hope that our study can stimulate further experimental investigations and provide a guide for future development of NiAl-based alloys.

10:50 AM
Oxidation Behavior and Thermal Stability of Ni-20Al-20Pt Refractory Alloy at High Temperature Exposure: *Rabindra Mahapatra*[1]; [1]Naval Air Systems Command

The isothermal oxidation behavior of Ni-20Al-20Pt (in at.%) refractory alloy was investigated up to a period of 312 hr in air from 1200 to 1400°C. A comparison of the oxidation behavior of this alloy with a conventional Ni-base superalloy (Inconel 713)shows an order of magnitude oxidation resistance. This experimental alloy oxidizes by forming Al2O3 and a trace of PtO with Al2O3 oxide layer in contact with air. Optical and Scanning Electron Microscopy (SEM) were used to study the microstructure, morphology, and composition of scale formed after oxidation. The thermal stability of the alloy after extended periods of exposure in air at 1200. 1300, and 1400°C was studied using Transmission Electron Microscopy (TEM). The mocrostructure of the alloy observed to be stable at 1300°C for 312 hr exposure in air.

11:10 AM
Physical Properties of Single Crystals of Co3(Al,W) with the L12 Structure: *Haruyuki Inui*[1]; Katsushi Tanaka[1]; Kyosuke Kishida[1]; Takashi Ohashi[1]; [1]Kyoto University

The recent discovery of the stable L12-ordered intermetallic compound, Co3(Al,W) coexisting with the solid-solution based on Co with a fcc structure has opened up a pathway to the development of a new class of high-temperature structural material based on cobalt, 'Co-base superalloys'. However, almost nothing is known about mechanical properties of these Co-based two-phase alloys, especially of the constituent L12 phase, Co3(Al,W). We have investigated some physical properties (elastic constants, thermal conduction, thermal conductivity and plastic deformation behavior) of single crystals of Co3(Al,W) with the L12 structure. The values of all the three independent single-crystal elastic constants and polycrystalline elastic constants of Co3(Al,W) experimentally determined are considerably smaller than those previously calculated by others. When judged from the values of Poisson ratio, Cauchy pressure and Gh/Bh, the ductility of Co3(Al,W) is expected to be sufficiently high so that Co3(Al,W) can be practically used as the constituent phase of 'Co-base superalloys'.

11:30 AM
The Influence of Cr and B Additions on the Mechanical Properties and Oxidation Behaviour of L21-Ordered Fe-Al-Ti-Based Aluminides at High Temperature: *Ronny Krein*[1]; Martin Palm[1]; [1]Max-Planck-Institut für Eisenforschung GmbH

Several Fe-25Al-Ti-X (X = Cr, B) alloys with 15 or 20 at.% Ti and varying Cr and B contents have been investigated with respect to their mechanical properties and their oxidation behaviour at elevated temperatures. The mechanical properties have been characterized by means of high-temperature compression tests, 4-point-bending tests and creep tests at 750, 800 and 850°C. The oxidation behaviour has been examined using thermogravimetric analyses at 800°C in synthetic air. The addition of Cr and B are both beneficial for the creep resistance and has no influence to the still excellent oxidation behaviour. The alloys exhibit two flow stress anomalies at around 550 and 800°C, a phenomenon which has been observed for the first time. The appearance of the additional flow stress maximum at around 550°C can be suppressed by aging and by increasing the deformation rate.

11:50 AM
From Nanometric Ni/Al Multilayers to Intermetallic Thin Films: T. Vieira[1]; *J. Noro*[2]; A. S. Ramos[2]; [1]University of Coimbra; [2]Instituto de Ciência e Engenharia de Materiais e Superfícies

Ni aluminides are potential candidates for practical applications as thin films, which can be formed by thermal annealing of alternate layers of Ni and Al that react exothermically at rather low temperatures. The aim of this work is to produce nanometric Ni/Al multilayer thin films and to evaluate their structural and mechanical properties (H, E and sigma0.2) having in mind its application for joining microparts. Thin films of the B2-NiAl ordered intermetallic phase were produced after thermal annealing of Ni/Al multilayers deposited by d.c. magnetron sputtering from pure nickel and aluminium targets. The substrates' holder was a copper block to avoid intermixing and reaction during deposition. The multilayers were designed in order to have an overall atomic composition of 50Al:50Ni and modulation periods,lambda,ranging from 5 to 120 nm. Depending on the multilayer period, the formation of the NiAl equilibrium phase could be preceded by NiAl3 and/or Ni2Al3 phase(s).

Ultrafine-Grained Materials: Fifth International Symposium: Stability, Technology, and Property

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Yuri Estrin, Monash University and CSIRO Melbourne; Terence Langdon, University of Southern California; Terry Lowe, Los Alamos National Laboratory; Xiaozhou Liao, University of Sydney; Zhiwei Shan, Hysitron Inc; Ruslan Valiev, UFA State Aviation Technical University; Yuntian Zhu, North Carolina State University

Tuesday AM — Room: 395/396
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Yuntian Zhu, North Carolina State University; Günter Gottstein, RWTH Aachen; Qiuming Wei, University of North Carolina; Yinmin Wang, Lawrence Livermore National Laboratory

8:30 AM Invited
Stabilization of Nanocrystalline and Ultrafine Grain Sizes by Solute Additions: *Carl Koch*[1]; Ronald Scattergood[1]; Kristopher Darling[1]; Jonathan Semones[1]; [1]North Carolina State University

Nanocrystalline and ultrafine grained microstructures offer improved mechanical behavior. However, they are subject to coarsening due to the driving force from the large grain boundary area, and therefore energy, that the small grains provide. While some nanocrystalline metals exhibit grain growth, usually abnormal grain growth, even at room temperature, there are many examples of the stabilization of grain sizes to significant fractions of the material's melting point. Both kinetic (reduction of grain boundary mobility), and thermodynamic (reduction of the specific grain boundary energy), approaches have been used. This talk will present evidence for the stabilization of grain sizes in nanocrystalline materials from the literature and from recent research from the authors' laboratory. Reduction of the specific grain boundary energy by solute segregation will be emphasized.

8:50 AM
Mechanical and Biological Properties of Nanostructured Titanium: *X. Zhang*[1]; Yinmin (Morris) Wang[2]; [1]Shanghai Jiao Tong University; [2]Lawrence Livermore National Laboratory

Nanostructured titanium is known to have high strength (~1GPa) and decent useful ductility. Because of its excellent biocompatibility, nanostructured titanium is also considered as one of the top candidates for bio-implant type of applications. In this work, we will present some of our recent progresses on improving the biomechanical properties of the nanostructured titanium. Our focus will be on the reduction of Young's modulus and enhanced adhesion properties between nanostructured titanium and animal tissues. This work was supported by the Nano Foundation of Shanghai, China, and was also partially performed under the auspices of the US Department of Energy by University of California, Lawrence Livermore National Laboratory under contract of No. W-7405-Eng-48.

9:05 AM Invited
Effect of Isothermal Annealing on the Microstructure of Nanocrystalline: *Indranil Roy*[1]; Hsiao-Wei Yang[1]; Linh Dinh[1]; Farghalli Mohamed[1]; [1]University of California

Bulk nanocrystalline (nc) Ni specimens having an average grain size of 100 nm were subjected to isothermal annealing at 573 K for different holding times. The microstructure developed in nc-Ni following annealing was examined by means of transmission microscopy (TEM). Also, electron backscatter diffraction (EBSD) was used to generate orientation maps. The results show the presence of a large volume of annealing twins. Post deformation TEM micrographs reveal that dislocations are blocked at the nano-twinned boundaries resulting in dislocation accumulation and entanglement.

9:25 AM
Influence of Iron Oxide Particles on the Strength of Ball-Milled Iron: *Donald Lesuer*[1]; Chol Syn[1]; Oleg Sherby[2]; [1]Lawrence Livermore National Laboratory; [2]Stanford University

Detailed microstructural and mechanical property studies of ball-milled iron, in the powder and consolidated states, are reviewed and assessed. The analyses cover three and one-half orders of magnitude in grain size (from 6 nm to 20 µm) and focus on the influence of oxide particles on the strength. The study includes the early work of Jang and Koch, Kimura and Takaki and continues with the more recent work of Umemoto et al. and Belyakov, Sakai et al. It is shown that the major contributors to strength are the nano-oxide particles. These particles are created by adiabatic shear banding during ball-milling leading to a bimodal distribution of particles. The predicted strength from particles, σ_p, is given by $\sigma_p = B\cdot(D)^{-1/2}$ where D is the surface-to-surface interparticle spacing, and B = 395 $MPa\cdot\mu m^{1/2}$. A model is proposed that accounts for the influence of the bimodal particle size distribution on strength.

9:40 AM
Production, Properties and Application Prospects of Bulk Nanostructured Materials: *Radik Mulyukov*[1]; Renat Imayev[1]; Ayrat Nazarov[1]; [1]Russian Academy of Sciences, Institute for Metals Superplasticity Problems

Principles of the method of multiple isothermal forging for the production of bulk nanostructured materials will be formulated. The method was pioneered and has been widely used at the Institute for Metals Superplasticity Problems. The common features and distinctions of this method with severe plastic deformation methods such as high-pressure torsion and equal-channel angular pressing will be analyzed. It will be demonstrated that multiple isothermal forging is a versatile method allowing for the production of bulk and sheet nanostructured semi-products with the grain size down to about 50 nm and is applicable to various metals and alloys including heat-resistant, hard-to-deform and intermetallic ones. Novel mechanical properties of bulk nanostructured materials produced by this method will be presented. The prospects of their structural and functional applications will be outlined.

9:55 AM Invited
Grain Refinement and Mechanical Properties of Two-Phase Cu-Zn Alloys Subject to Constrained Groove Pressing: *Leon Shaw*[1]; Kaiping Peng[2]; Ying Zhang[2]; K.-W. Qian[2]; [1]University of Connecticut; [2]Fuzhou University

The applicability of constrained groove pressing (CGP) for grain refinement and improvements in mechanical properties of two-phase Cu-Zn alloys is demonstrated in this study. It is shown that the grain size and volume fraction of the beta-prime phase in the Cu-Zn alloy should be kept small through proper heat treatment, and the die design with wider groove can reduce stresses at the most severely loaded region of the workpiece, all of which allow more numbers of pressing to attain grain refinement. The hardness and tensile strength increase with the number of the CGP cycle. The improved tensile properties are related to microstructural evolution induced by CGP.

10:15 AM Break

10:30 AM Invited
Thermal Stabilty of Nanostructured Metals: *Niels Hansen*[1]; Xiaoxu Huang[1]; Roy Vandermeer[1]; [1]Risoe National Laboratory

Common to metals deformed to high strains is a very fine microstructure, high strength and limited ductility. Structure and property optimization by annealing after deformation must therefore be explored. In the present study commercial purity aluminium has been annealed after cold rolling to ultrahigh strains up to εvM=6 and the annealing process has been analyzed in terms of recovery and conventional recrystallisation. The recovery kinetics has been analyzed for specimens annealed in the temperature range 140-220°C and it is found that increasing strain has no effect on the activation energy of recovery but the more highly deformed metal does have recovery rate much larger than the less deformed material. Deformation and annealing conditions must therefore be balanced when property optimization is sought through a recovery anneal of metals deformed to ultrahigh strains.

10:50 AM
Quasi-Static and Dynamic Mechanical Properties of Commercial-Purity Tungsten (W) Processed by ECAE below the W Recrystallization Temperature: *Z. Pan*[1]; Suveen Mathaudhu[2]; L. Kecskes[2]; K. Hartwig[3]; Q. Wei[1]; [1]University of North Carolina at Charlotte; [2]US Army Research Laboratory; [3]Texas A&M University

Thermo-mechanical processing tungsten (W) has proved to be difficult due to its poor workability. Particularly, commercial purity polycrystalline W exhibits a very high ductile to brittle transition temperature (DBTT). Recently, severe plastic deformation (SPD) has been used to refine the W grain size of. Previous experiments have reported the quasi-static (QS) and dynamic mechanical behavior of the SPD W showing unique results. However, the processing, particularly equal-channel angular extrusion (ECAE) was performed at relatively high temperatures to avoid cracking of the work piece. Recent developments in ECAE processing allowed us to process W at lower temperatures. In this work, we have processed commercial purity W via different ECAE routes at temperatures well below the nominal recrystallization temperature of 1400°C. We have systematically evaluated the QS and dynamic compressive behavior of the processed W. Quasi static compression tests were performed using an MTS machine at room temperature. It was observed that, with decreasing temperature, the ECAE samples showed higher yield and flow stresses than those processed near the recrystallization temperature; no obvious strain hardening was observed in the QS stress strain curves below a certain temperature. Quasi static strain rate jump tests show that the strain rate sensitivity of the ECAE W is in the range of 0.02 0.04, about half the value for coarse grained W. Uni axial dynamic compressive tests were also performed on a Kolsky bar (or split Hopkinson pressure bar) system. Scanning electron microscopy was used to observe the post loading surfaces of the deformed samples to understand the deformation and failure mechanisms under quasi static and dynamic compression.

11:05 AM Invited
Recent Development in Nanometals by Hydrostatic Extrusion: *Krzysztof Kurzydlowski*[1]; [1]Warsaw University of Technology

Hydrostatic extrusion is an efficient method of grain refinement down to nanometer scale in metallic materials. The paper shows, that tt can be directly used to obtain a mean grain size smaller than 100 nm and a significant fraction of high angle grain boundaries in aluminium alloys, titanium or iron. It is also demonstrated that grain size reduction to this level in some other materials, e.g. nickel, requires combination HE, as a final operation, with of some other SPD methods.

11:25 AM
Effect of ECAP Deformation on the Thermal Stability of an Aluminum Alloy 3103: Xenia Molodova[1]; *Günter Gottstein*[1]; [1]RWTH Aachen University

Studies of the annealing behavior of severely plastically deformed materials are rare compared to other features of such processed materials. As shown recently, discontinuous recrystallization is the governing phenomenon during heat treatment of severely deformed materials. In the present study the microstructure evolution of an aluminium alloy 3103 subjected to ECAP applying route Bc was investigated after deformation and subsequent heat treatment. The isothermal annealing was carried out at 330°C. The deformed and annealed states were analyzed by EBSD, TEM and microhardness tests. It will be demonstrated that the ECAP deformed material exhibited an increased stability against discontinuous recrystallization with growing number of passes. This superior thermal stability is desirable for many applications of UFG alloys. In addition, the ECAP deformed material was subjected to a low temperature annealing prior annealing at 330°C. The effect of this precedent recovery treatment on the thermal stability will be discussed.

11:40 AM
Formation of Ultrafine-Grained Microstructure in HSLA Steel Profiles by Linear Flow Splitting: *Tilman Bohn*[1]; Enrico Bruder[1]; Clemens Müller[1]; [1]Technische Universitat Darmstadt, Department of Materials Science, Division Physical Metallurgy

Linear flow splitting is a new cold forming process for the production of branched sheet metal structures in integral style. The process induces extremely high deformation degrees without formation of cracks in the split sheets due to hydrostatic compressive stresses. Investigations on a HSLA steel (ZStE 500) show the formation and fragmentation of a dislocation cell structure in the severely deformed regions of the steel sheet. This results in ultrafine-grained microstructures and improved mechanical properties, similar to SPD processes as ECAP or HPT. EBSD measurements reveal a gradient in grain size with an increase in direction perpendicular to the surface whereas micro hardness decreases in the same direction. Based on these results, basic principles of the linear flow splitting process and its expected potential are discussed.

11:55 AM
Effect of Stacking Fault Energy on the Mechanical Behavior of Nanostructured Co-Ni Alloys: *Pei-Ling Sun*[1]; Yonghao Zhao[2]; Jason Cooley[3]; Michael Kassner[4]; Yuntian Zhu[5]; [1]Feng Chia University; [2]University of California at Davis; [3]Los Alamos National Laboratory; [4]University of Southern California; [5]North Carolina State University

Co-Ni alloy has very small solution-hardening effect and is, therefore, perfect for studying the effect of stacking fault energy on the mechanical properties. Nanostructured Co-Ni alloy samples were processed by high-pressure torsion. The addition of cobalt to nickel reduces the stacking fault energy (SFE) in nickel. These nanostructured Co-Ni alloys were then tested under tension. It was found that increasing SFE reduces the tensile stress in Co-Ni alloys. The related microstructures will be examined in TEM and compared with experiments in the literature.

2008 Nanomaterials: Fabrication, Properties, and Applications: Application

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee
Program Organizers: Wonbong Choi, Florida International University; Seong Jin Koh, University of Texas at Arlington; Donna Senft, US Air Force; Ganapathiraman Ramanath, Rensselaer Polytechnic Institute; Seung Kang, Qualcomm Inc

Tuesday PM — Room: 273
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Zhong Lin Wang, Georgia Institute of Technology; Seung Kang, Qualcomm Inc

2:00 PM Invited
Sculpture and Properties of Functional Nanostructures and Nanolayers for Applications: *Ganapathiraman Ramanath*[1]; [1]Rensselaer Polytechnic Institute

Harnessing nanostructures for many applications requires the sculpture of nanoscale building blocks with control over atomic-level structure, surface chemistry, and their assembly into controllable configurations. This talk will describe three different examples from our recent works in on directing the growth and assembly of assemblies of nanostructures of magnetic and thermoelectric materials, and novel properties of molecular nanolayers. Our approaches open up new possibilities for a variety of applications, e.g., data storage, power generation and refrigeration, and nanodevices. I will first illustrate the synthesis and assembly of nanoparticles of magnetic and thermoelectric materials with control over crystal structure, shape and surface chemistry. We have devised entirely new strategies to obtain high-coercivity FePt-silica core-shell nanomagnets with simultaneous control over size, dispersity, composition and phase stability, by using surfactant-stabilized water nanodroplets, and to realize low temperature chemical ordering by impurity incorporation. The nanoparticles can be assembled into chains of desired length and diameter, or ordered films by exploiting molecular coupling agents as braiders and spacers Directed synthesis of molecularly braided magnetic nanoparticle chains using molecular couplers. A new dynamic templating method to obtain rod-shaped assemblies by synergistic growth of two phases will be presented Rod-shaped assemblies of FePt-PtTe2 through dynamic templating. I will then describe a new surfactant-mediated technique to obtain single-crystal nanorods of chalcogenides with and without branching Low-temperature templateless synthesis of single-crystal bismuth telluride nanorods, by manipulating surfactant type, concentration and temperature. I will finally demonstrate the use of organosilane and polyelectrolyte nanolayers to enhance the chemical integrity, electrical device reliability and mechanical toughness of metal-dielectric thin film interfaces and porous materials Self assembled nanolayers as adhesion enhancers and diffusion barriers.

2:30 PM
Damping of Hybrid Surface Plasmon Modes in Ag Nanoparticles; Application to Vapor Sensing: Sriharsha Karumuri[1]; *Kaan Kalkan*[1]; [1]Oklahoma State University

The present work demonstrates ultra-fast sensing of Hg vapor by a novel sensing mechanism: nonradiative damping of hybrid dipolar surface plasmon modes in metal nanoparticles. Although, the Mie theory predicts Hg adsorption can lower the plasmon extinction in Ag nanoparticles, previous work has only shown its increase due to a different competing mechanism: electron transfer from Hg. The investigators show that once the intrinsic damping on the surface of a silver nanoparticle is reduced significantly, additional damping due to adsorption becomes the dominant mechanism for sensing. Further, a dramatic gain in response speed is observed. An 8% change in plasmon extinction is recorded in the first 30 s of exposure to few ppm of Hg. The investigators will report on: their plasmon damping model where Hg atoms serve as electron scattering centers; Ag nanoparticle reduction on Si substrates and subsequent plasmon damping-reduction process; as well as various AFM, SEM, and EDX characterizations.

2:45 PM
Fabrication and Characterization of Laser Drilled Vias in SiO_2 Nanofluidic Devices: *Xiaoxuan Li*[1]; William Hofmeister[1]; [1]University of Tennessee

Nanofluidics is used to study fluid flow in nanometer sized structures or objects. Biomolecules can be confined and trapped in nanospace in nanofluidics in separation science and single molecule detection in life science, biophysics, and analytical chemistry. In this paper, electron beam photolithography was used to fabricate nanochannels in fused silica (SiO_2) and nanochannel patterned glass chip was bonded with blank SiO_2 chips to fabricate nanofluidics. The nanofluidics device was used in laser fluorescence spectroscopy to study single molecules in nanochannels. Fabrication and characterization of nanochannels in SiO_2 was reported. The vias in SiO_2 nanofluidics devices were manufactured by a CO_2 laser in "superpulse" mode and their microstructure was studied. The laser drilling parameters including number of laser pulses, laser pulse-on time, laser beam size, thickness of SiO_2 glass were evaluated for the optimization of access hole morphology.

3:00 PM
Fabrication and Microstructure Characterization of Graded Anisotropy Media: *Robb Morris*[1]; Gregory Thompson[1]; Yuki Inaba[1]; J. Harrell[1]; [1]University of Alabama

The superparamagnetic limit poses a fundamental barrier that threatens to limit further increases in magnetic storage density. Attempts to increase the thermal stability by making the grains "magnetically harder" have failed because they have also made them impossible to write. One solution to this conundrum is to use exchange coupled composites, in which one end of the grain is magnetically soft (low Ku) and the other end magnetically hard (high Ku). To date, most of the work has focused on composites with discrete layered interfaces. This talk will address the use of a graded compositional profile to achieve a gradient in Ku. Our proof-of-principle study has used a graded CoPt film. The technical fabrication challenges, performance and stability of different gradient profiles will be addressed in terms of the films' microstructure, which has been characterized by TEM, XRD, LEAP and AGM analysis.

3:15 PM
Faceting and Surface Energy Effects on the Self-Assembly of Epitaxial Quantum Dots: *Lawrence Friedman*[1]; [1]Pennsylvania State University

Epitaxial self-assembled quantum dots (SAQDs) will allow breakthroughs in electronics and optoelectronics. SAQDs result from Stranski-Krastanow growth whereby epitaxial 3D islands form spontaneously on a planar thin film. Common systems are Ge_xSi_{1-x}/Si and In_xGa_{1-x}As/GaAs. SAQDs are typically grown on a (001) surface. The formation and evolution of SAQDs is determined by the interaction of surface energy and elastic strain; however, there are competing theories of the nature of the planar (001) surface: (1) It is a stable non-facet. (2) It is a stable crystal facet. (3) It is an unstable anti-facet. The first theory appears most often in modeling literature, but the second two theories take explicit account of the discrete nature of a crystal surface. Here, the impact of the three theories on SAQD formation is discussed along with the possibility of a phase transition. Can they all three be correct?

3:30 PM
Formation of Metallic Nanoparticles through Colloid-Induced Spontaneous Metallization: *Tzy-Jiun Luo*[1]; [1]North Carolina State University

We report a room temperature synthesis of metallic nanoparticles that is based on spontaneous metallization induced by colloidal solution of silane that contains methanol. Metallic ions (e.g. Sn4+, Ag+) have been successfully reduced in the presence of organically modified silane colloids while the same reaction was not observed in a polymer solution that contains similar functional groups. Metallic nanoparticles with diameters around 5 ~ 7 nm were identified using TEM. Kinetic studies were conducted using UV-Vis spectroscopy. Nanoparticle containing silica colloids can be easily transformed into monolith sol-gel materials, and have been examined using XRD and electrochemical methods. The results have shown that nanoporous silica materials doped with metallic nanoparticles exhibit enhanced electrochemical properties and stability. Currently, study of this nanocomposite for electrochemical sensing is underway.

3:45 PM
Electrophoretic Phenomena of Fine Particles in Aqueous and Non-Aqueous Solvents: *Junji Shibata*[1]; Daisuke Yamashita[1]; Masato Tateyama[1]; Norihiro Murayama[1]; [1]Kansai University

The electrophoretic phenomena of fine polystyrene particles in aqueous and non-aqueous solvents were investigated in the electric field. The spherical polystyrene particles of 0.03, 0.1, 1.0, 4.3 and 9.6μm were used as a sample particle, and water-alcohol mixtures such as methanol, ethanol, etc. were also used as a solvent of suspension. The electrophoretic mobility of fine particles is decreased with a decrease in volume percent of water in water-alcohol mixtures. These tendencies of the decrease in electrophoretic mobility can be expressed by the Smoluchowski' equation. The forces acting the particles in electric field are considered to be electrical force, friction force and the other forces like asymmetric effect and electrophoretic retardation effect which appear in ionic behavior. The behavior of fine polystyrene particles in non-aqueous solvents can be explained by the forces acting the particles in the electric field.

4:00 PM Break

4:15 PM Invited
Direct Writing: How Can We Leverage Nature's Directed Self-Assembly to Create Novel Constructs?: *Douglas Chrisey*[1]; David Corr[1]; Cerasela Dinu[1]; [1]Rensselaer Polytechnic Institute

The rapidly growing fields of nanoscience and nanotechnology have stimulated considerable interest in methods for building structures that have nanometer dimensions. As a result, there is interest in patterning techniques that offer nanometer resolution using experimentally simple set-ups as well as in systems which can mimic cellular self-assembly to direct patterning on engineered environments. We describe two different strategies for building structures on a substrate. The first approach involves CAD deposition of controlled patterns of cells to direct the formation of tissue constructs while the second approach relates to the possibility of using the evolutionary machineries of the cell, microtubules, and the kinesin molecular motor to direct the bottom-up patterning at the nanoscale level.

4:45 PM
Large-Scale Placement of Single Nanoparticles with Nanoscale Precision: *Hong-Wen Huang*[1]; Vishva Ray[1]; Seong Jin Koh[1]; [1]University of Texas at Arlington

Nanoparticles have drawn a lot of attention due to their unique and size-dependent electrical, optical, and magnetic properties. Practical devices utilizing nanoparticles, however, require techniques that enable precise placement of single nanoparticles on targeted substrate locations. Here, we present a large-scale and precise placement of nanoparticles, where single nanoparticles are electrostatically guided and placed on the centers of circular patterns, one nanoparticle per one circular pattern. The electrostatic guiding structure was formed using self-assembled monolayers (SAMs) with pattern diameter of ~130 nm. We demonstrate placement of single 20nm Au nanoparticles onto centers of the circular patterns with precision of ~7 nm. Out of 400 patterns, more than 70% of the circular patterns were occupied by exactly one Au nanoparticle. We envision that this method could be applied for fabrication of single electron devices, photonic devices, biological/chemical sensors, and other devices that require precise nanoparticle placement.

5:00 PM
Position Controlled Fabrication of Metal Nanostructures with Focused Electron Beam: *Kazuo Furuya*[1]; Masaki Takeguchi[1]; Minghui Song[1]; Kazutaka Mitsuishi[1]; Miyoko Tanaka[1]; [1]National Institute for Materials Science

Electron beam induced deposition (EBID) is one of the promising techniques, because of short wavelength which results in the resolution limit less than several 10 nm. We have been using FE-SEMs and FE-TEMs for the fabrication of position and size controlled metal nanostructures. A gas introduction system with a nozzle of 0.1 mm was developed. The flux of the gas for EBID was estimated to be approximately 2x10-4 Pa l s-1. The position and size control was carried out careful manipulation of electron beam. The diameters of the nanostrucrures were ranged from 4 to 20 nm. Magnetic nanostructures were also obtained with iron containing precursor gas, Fe(CO)5. Free-standing iron nano-antennas were examined by FE-TEM with electron holography. The magnetic field produced an electron hologram for the nanostructures. It is found that magnetic fields were leaked from the nanostructure body and the whole was likely to be one "nano-magnet".

5:15 PM
Phase Field Modeling of Nansocale Island Dynamics: *Zhengzheng Hu*[1]; Shuwang Li[1]; John Lowengrub[1]; Steven Wise[2]; [1]University of California, Irvine; [2]University of Tennessee

In this talk, a Burton-Cabrera-Frank (BCF) island dynamics model is used to analyze the epitaxial growth of a circular island. We show that there exists a critical deposition flux for which a single-mode perturbation remains unchanged and this flux together with other parameters may be exploited to control the shape of the island. To demonstrate the idea, a phase-field model that accounts for Ehrlich-Schwoebel effect and surface diffusion, is used to simulate the dynamics of the island. A block-structured adaptive mesh refinement method and an efficient numerical method is developed using a second order accurate fully implicitly time discretization together with a second order accurate finite difference spatial discretization. We close by demonstrating the nonlinear evolutions of islands to show the extent of applicability of our ideas on shape control at the nanoscale.

5:30 PM
Temperature, Thickness and MeV Si Ions Bombardment Effects on the Thermoelectric Generator from $ErFe_4Sb_{(6-y)}Ge_y$ Thin Films: *Satilmis Budak*[1]; Sadik Guner[1]; Claudiu Muntele[1]; Daryush Ila[1]; [1]Alabama A&M University

We have grown monolayers of $ErFe_4Sb_{(6-y)}Ge_y$ (y = 2,4) thin films on silica substrates with varying thickness between 200-1000 nm using ion beam assisted deposition (IBAD). MeV Si ions bombardments have been performed on samples with varying fluences.The Seebeck coefficient, electrical and thermal conductivity measurements were carried out before and after bombardment to calculate the figure of merit, ZT. MeV Si ions bombardment caused changes on the thermoelectric properties of films. Rutherford Backscattering Spectrometry (RBS) was used to analyze the elemental composition of the deposited materials and to determine the layer thickness of each film. Research sponsored by the Center for Irradiation of Materials, Alabama A&M University and by the AAMURI Center for Advanced Propulsion Materials under the contract number NNM06AA12A from NASA, and by National Science Foundation under Grant No. EPS-0447675.

5:45 PM
Photonic Curing for Low Temperature Sintering of Printed Nano-Particulate Materials: *James Sears*[1]; Michael Carter[1]; Jeffery West[1]; [1]South Dakota School of Mines and Technology

Photonic Curing is being developed to cure or sinter metal nano-particle based films by exposing them to a brief, intense pulse of light from a xenon flash lamp. This photonic curing technology allows for rapid and selective heating that fuses nano-scale metallic ink particles into functional components. This technology allows the curing or sintering of nanoscale metallic ink patterns on low-temperature substrates including flexible circuit boards, flat panel displays, interconnects, RFID tags, and other disposable electronics without the use of heat. This paper reports on the results obtained after sintering conductive, magnetic, and dielectric nano-particle inks. Sintering was performed with the photonic curing technique developed by NovaCentrix, 2W frequency doubled Nd:YAG CW laser, and a conventional muffle furnace. Sample thickness, micro-structural details, resistivity, and sintering characteristics are also examined and compared for the sintering techniques.

3-Dimensional Materials Science: Modeling and Characterization across Length Scales I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee
Program Organizers: Michael Uchic, US Air Force; Eric Taleff, University of Texas; Alexis Lewis, Naval Research Laboratory; Jeff Simmons, US Air Force; Marc DeGraef, Carnegie Mellon University

Tuesday PM Room: 286
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Andrew Geltmacher, Naval Research Laboratory; Dennis Dimiduk, US Air Force

2:00 PM
Construction of Simplified Three-Dimensional Grain Boundary Surfaces from Serial Section Micrographs: *Scott Dillard*[1]; John Bingert[2]; Dan Thoma[2]; Bernd Hamann[1]; [1]University of California, Davis; [2]Los Alamos National Laboratory

A method is presented for extracting 3D grain boundary surfaces from serial sections of polycrystalline materials comprising a mixture of electron back-scatter diffraction (EBSD) data and light-optical micrographs. This method relies on a constrained Potts model to interpolate grain boundaries between segmented section images, allowing for greater sectioning thickness and more spacing between EBSD micrographs. The result is a segmented volume from which a triangulated boundary surface is extracted, complete with topological information about the connectivity of grain faces, edges and vertices. A method to reduce the number of triangles in this surface without introducing error is also described. The application of these methods to an actual 3D data set will be demonstrated through the analysis of a shocked tantalum polycrystal.

2:20 PM
Integration of Focused Ion Beam Serial Sectioning and Orientation Imaging Microscopy for 3-D Microstructure Reconstruction: Suk-Bin Lee[1]; Emine Gulsoy[1]; Michael Groeber[2]; Gregory Rohrer[1]; Anthony Rollett[1]; Michael Uchic[3]; Jeff Simmons[3]; *Marc DeGraef*[1]; [1]Carnegie Mellon University; [2]Ohio State University; [3]US Air Force

A new method for reconstructing a 3D microstructure using the focused ion beam-orientation imaging microscope (FIB-OIM) is introduced. The method was applied to the IN100 superalloy, and results cross-sectional ion-induced secondary electron (ISE) images and corresponding electron back-scattered diffraction (EBSD) maps. Both data series show small translational misalignments. For the EBSD map alignments, we developed a procedure based on the expectation that good alignment will result in minimal average disorientation between pixels in adjacent layers. After alignment of all ISE images using a convolution-based algorithm, the images were brought in coincidence with the orientation data. The final grain boundaries were obtained by employing an eroded orientation data volume combined with the 3-D Euclidean distance map of the image data volume as the starting input for a 3-D watershed algorithm. We will discuss the experimental and numerical methods developed for this approach.

2:40 PM
3D Visualization of Solid Phase Morphology during Solidification of Al-Cu Alloys in Unconstrained and Constrained Growth Conditions: *Demian Ruvalcaba-Jimenez*[1]; Dmitry Eskin[1]; Laurens Katgerman[2]; [1]Netherlands Institute for Metals Research; [2]Delft University of Technology

Microstructure development during solidification has been studied in post-solidified samples and samples quenched during solidification. Visualization of microstructure has been done over 2D images. 2D images can accurately represent the structure if the morphology of phases is geometrically simple e.g. spheres. However, during solidification, growth and coarsening occur simultaneously developing complex morphologies of phases. Moreover, thermo-solutal gradients can be imposed asymmetrically during unconstrained growth solidification conditions affecting dendritic growth. Therefore, accurate representation of the microstructure under these conditions may only be archived by reconstructing it in 3D. In the present study, 3D visualization of microstructure development during solidification is presented for unconstrained and constrained growth conditions. Quenching, serial sectioning and 3D rendering in the reconstruction of unconstrained growth (in Al-7 wt% Cu alloy) is discussed. Finally, partial 3D visualization is demonstrated by image processing for images taken in-situ during constrained growth of columnar dendrites (in Al-20 wt% Cu alloy).

3:00 PM
On the Direct Three Dimensional Characterization of Microstructural Features in α/β and β Ti-Alloys Using Robo.Met-3D and FIB: *Robert Williams*[1]; Peter Collins[1]; Hamish Fraser[1]; [1]Ohio State University

Two techniques for the direct three dimensional characterization of microstructures, the Robo.Met-3D and the small dual-beam Focused Ion Bean (DB-FIB), have been used to characterize features present in α/β and β Ti-based alloys. The further development and expansion of these types of techniques is important, as many details of the microstructures are either misinterpreted or not observed when only considering classical two-dimensional projections. Examples at both length scales illustrating such cases will be shown, and compared with traditional stereological methods. As these techniques are seeing increased usage, it has become important to develop descriptions of the scale of features best observed using certain techniques, and how best to relate features observed using one technique (e.g., α-lath thicknesses in FIB) to features observed using the other (e.g., colony sizes using Robo.Met-3D). Finally, a direct comparison between reconstructions made of the same types of features using the different techniques will be presented.

3:20 PM Break

3:40 PM Invited
Application of 3D Microstructural Characterization to Dynamic Deformation and Damage: *John Bingert*[1]; Veronica Livescu[1]; Scott Dillard[2]; Lisa Dougherty[1]; Daniel Worthington[2]; Pat Dickerson[1]; Davis Tonks[1]; [1]Los Alamos National Laboratory; [2]University of California, Davis

The evolution of damage in polycrystalline materials from high-rate, dynamic deformation processes is a function of both the applied stress state and the material's microstructure and properties. Three-dimensional interrogation and quantification of damaged microstructures provides insight toward understanding the interaction between microstructure and the damage evolution process. This work investigates damage and deformation in materials under different conditions, including shocked, incipiently spalled tantalum and dynamically sheared 1018 steel. Serial sectioning by mechanical polishing and ion-beam milling were performed in order to reconstruct microstructures over a range of length scales. Electron backscatter diffraction was applied to investigate the relative levels of induced plasticity, revealing surprisingly contiguous and complex plastic linkages. The primary motivation of quantifying damage is to provide realistic input for, and validate, predictive damage models. Tantalum microstructural data were thus incorporated into a polycrystal plasticity finite element calculation, for which preliminary simulation results will be presented.

4:10 PM
Three-Dimensional Mesoscale Mechanical Modeling of a Beta Titanium Alloy: *Andrew Geltmacher*[1]; Muhammad Qidwai[2]; Alexis Lewis[1]; [1]Naval Research Laboratory; [2]SAIC

The three-dimensional (3D) microstructure of a beta-Ti alloy (beta-21s) was characterized using serial sectioning and computerized 3D reconstruction. The reconstructed volumes, comprising hundreds of beta-Ti grains, provide information on grain morphology and crystallography. These 3D volumes contain experimental data derived from optical microscopy and Electron Backscatter Diffraction (EBSD). This data is input into 3D Image-Based Finite Element Models to analyze the spatial evolution of state variables, such as stress, strain and local plasticity in the microstructure under mechanical loading conditions. These models incorporate anisotropic elasticity, von Mises plasticity and/or crystalline plasticity to simulate the interactions between different microstructural features and mechanical behavior, and to determine which microstructural features serve as initiation sites of deformation and failure.

4:30 PM
Identifying Critical Features in a Beta-Ti Microstructure Using Image-Based Finite Element Modeling: *Alexis Lewis*[1]; Muhammad Qidwai[2]; David Rowenhorst[1]; George Spanos[1]; Andrew Geltmacher[1]; [1]Naval Research Laboratory; [2]Science Applications International Corporation

Three-dimensional image-based finite element models were used to analyze stress and strain evolution in a 3D beta-Ti microstructure (beta-21s), obtained using serial sectioning. The simulation outputs and data from the reconstructed microstructures, including grain volume, true 3D grain shape, crystallographic orientation, 3D grain boundary networks, and distributions of grain boundary crystallographic normals are used to interrogate correlations between microstructural features and mechanical response. In this beta-Ti microstructure, high stresses and strains were observed at grain boundaries, and the morphological and crystallographic properties of these boundaries were analyzed to determine which factor(s) contribute to high local strains under a variety of loading condition.

4:50 PM
Full-Field Modeling of the Elastic Behavior of Foams Using Direct Input from Their Measured 3-D Structure: *Ricardo Lebensohn*[1]; Anthony Rollett[2]; [1]Los Alamos National Laboratory; [2]Carnegie Mellon University

A formulation based on Fast Fourier Transforms (FFT) for the calculation of the micromechanical behavior of heterogeneous materials has been adapted to predict the local elastic fields and the effective elastic properties of foams using direct voxelized input from their actual 3-D structure. In this presentation we show an application of the FFT-based model to prediction of the elastic behavior of carbon foams of about 90% porosity under uniaxial tension. The 3-D microstructure used as input was obtained by serial sectioning and light microscopy. The model predicts local stress concentrations along ligaments oriented longitudinally with respect to the applied load and an effective Young modulus consistent with experimental measurements.

5:10 PM
Three-Dimensional Reconstruction of Metallic Microstructures for Uncertainty Representation and Propagation in Finite Element Simulations of Local Mechanical Behavior: *Andrea Keck*[1]; [1]Arizona State University

Prediction of scatter on the mechanical behavior of metallic materials due to microstructural heterogeneity is quite important in a variety of scientific and technological applications. Three-dimensional (3D) representations of microstructures of 2xxx Al alloys are created via a combination of dual-scale serial sectioning techniques, with a smaller scale for particles and a larger scale for grains, and available microstructure reconstruction software. Basic statistics are measured from the 3D reconstructions, which are also used to create finite element meshes. Comparisons are made between statistics obtained from 2D and 3D reconstructions and criteria to find the smallest RVE needed for an example application are addressed. In addition, stochastic homogeneization methodologies to propagate the uncertainty from the micro- to the meso-scale based on these reconstructions are discussed. Work funded by a Structural Health Monitoring MURI, Department of Defense AFOSR Grant FA95550-06-1-0309, Victor Giurgiutiu program manager.

5:30 PM
Three-Dimensional (3D) Microstructure Visualization and Finite Element Modeling of Deformation and Crack Growth in Particle Reinforced Metal Matrix Composites: *Nikhilesh Chawla*[1]; [1]Arizona State University

The mechanical behavior of materials is inherently controlled by microstructure. In particular, composite materials, consisting of two or more components or phases, have highly complex microstructures. This makes modeling of the mechanical behavior a challenge. We have developed a three dimensional (3D) approach to (a) construct a "virtual microstructure" in 3D by serial sectioning technique, and (b) finite element modeling using the 3D microstructure as a basis. In this talk the fundamentals of the 3D virtual microstructure modeling methodology will be explored. This methodology was used to study the deformation behavior of SiC particle reinforced metal matrix composites. The role of second phase fraction, morphology, and aspect ratio on deformation was quantified and will be discussed. The 3D crack growth behavior of the composites was modeled using finite element modeling. Results from the microstructure-based 3D simulations were found to be in good agreement with experimental observations.

Alumina and Bauxite: Bauxite

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Sringeri Chandrashekar, Rio Tinto Aluminium Limited; Peter McIntosh, Hatch Associates

Tuesday PM	Room: 296
March 11, 2008	Location: Ernest Morial Convention Center

Session Chair: Everett Phillips, Nalco Company

2:00 PM Introductory Comments

2:05 PM
Chinese Bauxite and Its Influences on Alumina Production in China: *Songqing Gu*[1]; [1]Zhengzhou Research Institute of Chalco

It is shown in this paper that the main bauxite deposit in China is located in such central and south-west provinces as Shanxi, Henan, Guizhou and Guangxi provinces etc. The chemical and mineralogical composition of various Chinese bauxites is reviewed to present the characteristics of the Chinese bauxites, which have great influences on the alumina production process. Key technology and equipment applied in China are also presented.

2:30 PM
Alunorte Bauxite Dewatering Station - A Unique Experience: *Ayana Oliveira*[1]; Juarez De Moares[1]; Jorge Lima[1]; [1]Alunorte-Alumino do Norte do Brasil

Alunorte concluded the expansion 2 project increasing the installed production capacity from 2.5 for 4.3 mi t/y in 2006, through new 2 lines. Bauxite supplying for the new lines was conceived using pipeline from the mine to the refinery. With a length of 242 km and a diameter of 24 inches, the pipeline carries bauxite slurry with 50% solid concentration and a density of 1.45 Kg/cm^3. This slurry is received in a distribution station and pumped to the dewatering station, where it is dewatered through five hyperbaric filters, in order to reduce the moisture content to about 12 to 14%. This paper presents the process conditions at the hyperbaric filters, which made possible the start-up of the first unit of bauxite dewatering in the world that will be replicated in the next expansion, scheduled to start-up in 2008.

2:55 PM
A New Method Using Hydraulic Excavator and Tractor on Paragominas Bauxite Mine: *Octávio Guimarâes*[1]; Henrique Santos[1]; Hildegundes Silva[1]; [1]Companhia Vale do Rio Doce - CVRD

The Paragominas bauxite mine (CVRD) started its operation in 2007 and has 303.000.000 metric tones of dry washed bauxite. It's the first bauxite mine that uses an ore pipe (pipeline) in the world. The continuous flat tabular bauxite deposit, averaging 1.47 m thick, under an overburden that averages 11.04 m, characterizes the geometry of the operation. The mining sequence consists of deforestation, waste stripping, excavation, loading and transporting the bauxite. The mining operation has two major conditions to consider: a high stripping ratio (7.6 in volume) and a high rainfall, thus it is necessary to have a mining system that is conducive to waste stripping and ore selectivity. This paper describes the system developed to achieve these goals using hydraulic excavators and bulldozers for waste stripping, and hydraulic excavators and trucks for ore production.

3:20 PM
Design and Operation of the World's First Long Distance Bauxite Slurry Pipeline: Ramesh Gandhi[1]; Michael Weston[1]; *Maru Talavera*[1]; Geraldo Brittes[2]; Eder Barbosa[2]; [1]Pipeline Systems Incorporated; [2]Companhia Vale do Rio Doce - CVRD

Mineracão Bauxita Paragominas (MBP) is the first long distance slurry pipeline transporting bauxite slurry. Bauxite had developed a reputation for being difficult to hydraulically transport using long distance pipelines. This myth has now been proven wrong. The 245-km- long, 13.5 MT/y capacity MBP pipeline was designed and commissioned by PSI for CVRD. The pipeline is located in the State of Para, Brazil. The Miltonia bauxite mine is in a remote location with no other efficient means of transport. The bauxite slurry is delivered to Alunorte Alumina refinery located near Barcarena. This first of its kind pipeline required significant development work in order to assure technical and economic feasibility. This paper describes the technical aspects of design of the pipeline. It also summarizes the operating experience gained during the first year of operation.

3:45 PM Break

4:00 PM
Bauxite to Gallium - The Electronic Metal of the Twenty-First Century: *Chitta Mishra*[1]; M. Nair[2]; [1]National Aluminium Company Limited; [2]NALCO Bhawan

Alumina is produced from Bauxite using Bayer Process where alumina present in Bauxite reacts with aqueous caustic soda to form sodium aluminate. The sodium aluminate solution, or Bayer liquor, is continuously recycled; this results in equilibrium concentration of 100ppm to 125ppm Gallium in the liquors. A bleed stream is separated from the liquor for extraction of Gallium. Gallium is a strategic metal and has been gaining worldwide importance by virtue of its application in the electronic industries. NALCO has plans for setting up of a 7TPA Gallium Extraction Plant (99.99%/99.999% purity) at its Alumina Refinery, utilizing the eco-friendly Ion-exchange Resin technology of Nippon Light Metal, Japan. The extraction of Gallium from NALCO's Bayer liquor containing 120ppm of Gallium has been successfully completed both in the laboratory and pilot plant scales. Preparation of Techno- Economic Feasibility Report for commercialization of the technology is underway.

4:25 PM
Behaviour of Berylium in Alumina Production from Bauxites by Bayer Process and Development of Reliable Method of Its Determination: *Alexander Suss*[1]; Irina Paromova[1]; Andrey Panov[1]; Olga Shipova[1]; Natalia Kutkova[1]; [1]Russian National Aluminium-Magnesium Institute

The problem of beryllium in aluminium industry has been raised by OSHA (Occupational Safety and Health Administration) and other labor safety bodies in the USA and Canada with the aim to toughen beryllium standards in the aluminium smelting area. Now the work group of the International Aluminium Institute (IAI) is engaged in the investigation of this problem. The necessity of this study was caused by the fact that bauxite is the only source of beryllium coming into alumina (and then to an electrolytic cell), and there were practically no systematic investigations on beryllium content in raw materials and beryllium behavior in alumina production anywhere in the world. The optimum conditions of beryllium determination in bauxite by mass spectrometry (ICP MS) method have been found. Principle possibility of beryllium determination by spectrophotometry was successful with a detection limit of about 0.1 ppm. Two methods have been developed for the reliable determination of beryllium in bauxite or red mud; one involves mass spectrum analysis and another involves spectrophotometric analysis. It was shown in the course of the development of the decomposition methods; beryllium is completely dissolved in solution prior to analysis. From 61 investigated types of bauxite from various regions of the world the highest contents of beryllium was found in 7 types of bauxite from various deposits of the world. It was also found that there is a correlation dependence of Be content on a number of factors. During investigations of beryllium behavior in Bayer process, effects of temperature, digestion time and presence of impurities (kaolinite and lime) on the solubility of beryllium has been checked. The obtained results indicate it is possible to forecast behavior of Be in the Bayer process.

4:50 PM
Impact of Jamaican Bauxite Mineralogy on Plant Operations: *Desmond Lawson*[1]; Ab Rijkeboer[2]; Lawrence Andermann[3]; Austin Mooney[1]; [1]West Indies Alumina Company; [2]Rinalco B. V.; [3]SNF Holding Company

Over the period 2003 to 2006 the bauxite feed to the Kirkvine refinery has undergone a dramatic change in composition with severe impact on plant performance. Surprisingly, the change in composition is not directly evident from the XRF analysis of the major elements, which showed that the total Al_2O_3, Fe_2O_3 and LOI contents of the bauxite remained largely unchanged. A more detailed examination however, shows a remarkable change in Al_2O_3 distribution over the various minerals, indicative of a substantial change in bauxite mineralogy. Over the same period significant changes in plant behaviour were experienced with respect to, notably alumina recovery, mud settling and liquor chemistry.

Aluminum Alloys: Fabrication, Characterization and Applications: Alloy Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Subodh Das, Secat Inc; Weimin Yin, Williams Advanced Materials

Tuesday PM   Room: 293
March 11, 2008   Location: Ernest Morial Convention Center

Session Chairs: Subodh Das, Secat Inc; Weimin Yin, Williams Advanced Materials

2:00 PM
Crystallographic Texture Development of Aluminum Alloy Sheet under Single and Multipass In-Plane Stretching: *Stephen Banovic*[1]; Mark Iadicola[1]; Tim Foecke[1]; [1]National Institute of Standards and Technology

Increasing gas mileage has been one of the driving forces to reduce overall automotive vehicle weight. By replacing conventional steel sheet metal with lightweight materials, specifically aluminum alloys, this objective can be realized. Towards achieving this goal, research has been initiated to measure the multiaxial flow curves of commercially available aluminum sheet. An important aspect to understanding the mechanical behavior of the sheets is the associated microstructural description of the deformed materials. This talk will focus on the microstructural evolution of various aluminum alloys using x-ray diffraction techniques, to discern crystallographic texture, on single and multipass in-plane stretchings. The observed changes in texture, as a function of plastic strain level and strain state, will be discussed with respect to plastic constraint and compared to measured multiaxial flow surfaces.

2:20 PM
Formation of Special Textures by Precipitation in Continuous Cast AA5083 and AA3004 Aluminum Alloys: *Qiang Zeng*[1]; Xiyu Wen[1]; Tony Zhai[1]; [1]University of Kentucky

The development of microstructure and crystallographic macrotexture of the cold rolled continuous cast Al-Mn-Mg alloy hot band with different pre heat treatment conditions after annealing was investigated. It was found that the intensity of P orientation depends on the size and density of precipitates and the cold rolling reduction. The specimen with the very fine and well distributed precipitates can generate strong P orientation in high cold rolling reduction. TEM observation showed that the very fine precipitates exert a very effective pinning effect on dislocation and subgrain boundaries, which would be the reason for the strong P orientation. The samples with the higher volume fraction of P orientation yielded an inferior mechanical property. EBSD results revealed that P orientated grains were coarser and more elongated. The higher volume fraction of P component, the more coarse and elongated grains were generated, which would be the reason for the inferior mechanical property.

2:40 PM
Effect of Alloying Elements on the Segregation of Complex Phases in A356 Alloy Adding Mg, Cu, Ni and Sr as Modifier, Using Thermal Analysis and Characterization Microstructural: *Aline Hernandez-Garcia*[1]; Alejandro García-Hinojosa[1]; F. T. Estéves Alcázar[1]; Yvan Houbaert[2]; [1]Universidad Nacional Autónoma de México; [2]Universidad de Gante, Bélgica

The properties in cast conditions could be increased if adequate alloying elements are added and also treatment in liquid phase (modification) is applied. This will turn the base alloy in multicomponent with the formation of new and complex phases. The fraction, morphology and distribution of these new phases control of the microstructure will have an important impact in the development of new mechanics properties. This work studied the effect of the Cu, Mg and Ni alloying elements add to A356 alloy and modified with Sr. The complex alloys were analyzed using thermal analysis, metallographic etching qualitative-

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quantitative, with support of image analysis and scattering electron microscopy (SEM.EDS). The metallographic analysis was confronted with the predicted by the commercial software Thermocalc. Finally tensile properties and hardness were evaluates to explain the effect of the alloying elements. The better properties were reached for the Al-7Si-1.5Cu-1Mg-0.5Ni cast alloy modified with Sr.

2:55 PM
Investigation into Phase Relations in the Aluminum-Boron System: *Dominick Bindl*[1]; [1]University of Minnesota

This research project was conducted with the aims of placing thermal constraints on the stabilities of both aluminum diboride and aluminum dodecaboride phases for use as reinforcements in aluminum MMCs. DTA, TGA, XRD and optical microscopy analysis techniques were utilized to track phase changes through thermally varied isothermal castings. From this project it was seen that there is a transition tentatively identified as the formation of gamma-AlB¬12 with an onset ~ 900°C as identified with Muffle Furnace heating and observed at 1010°C when seen in DTA trials. AlB_2 was observed to oxidize preferentially over dodecaboride phases with a hexagonally configured Al_2O_3 oxide. This research serves to anticipate further research into targeted aluminum dodecaboride formation and stabilization for use as aluminum MMC reinforcement.

3:10 PM
Subgrain Coarsening of Aluminum Alloy AA 5005 by EBSD Observations: *Shengyu Wang*[1]; Anthony Rollett[1]; [1]Carnegie Mellon University

The subgrain structure of hot rolled aluminum alloy AA 5005 has been characterized on as-received samples using Electron Backscatter Diffraction (EBSD). However, to observe the actual microstructural evolution during recrystallization at elevated temperatures, hot stage EBSD has been implemented to capture in-situ development of the subgrain structure at the initial recrystallization stage. The in-situ results will be compared with current Monte Carlo simulation results, which are based on experimental data fitted 3D microstructure and textures with anisotropic grain boundary properties discriminating high angle grain boundaries, low angle grain boundaries and some special grain boundaries. The main objective of this study is to understand the circumstances under which we can expect abnormal (sub-)grain growth leading to nucleation of recrystallization. Therefore the results are compared to simulations of subgrain coarsening with initial (unrecrystallized) structures obtained by statistical reconstruction.

3:25 PM
Fatigue of Extruded AA6063: *Nick Nanninga*[1]; Calvin White[1]; [1]Michigan Technological University

The effect of orientation as well as of extrusion seam and charge welds on the fatigue life of specimens from a hollow AA6063 alloy extrusion profile have been characterized in the form of S-N curves. Specimen orientation with respect to the extrusion direction has a large effect on fatigue life. Transverse specimens exhibit only a fraction of the high cycle fatigue life exhibited by longitudinal specimens. At lower stress levels, transverse specimens with seam and/or charge welds oriented transverse to the loading direction exhibited less than half the fatigue life of transverse specimens not containing welds. Specimens, with and without welds present, loaded 45 degrees with respect to the extrusion direction, display only moderate reductions in fatigue life. The variations in fatigue life are explained by surface roughness and variations in microstructure.

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Effect of Die Line Roughness on the Fatigue Life of Extruded AA6082: *Nick Nanninga*[1]; Calvin White[1]; [1]Michigan Technological University

To better understand the relationship between extrusion die line features and fatigue behavior, the fatigue life of polished and as-extruded specimens taken from a hollow extruded AA6082 profile have been characterized in the form of S-N curves. As-extruded specimens, taken transverse to the extrusion direction, i.e. containing die lines transverse to the loading direction, exhibit fatigue lives that are less than ten percent of those for longitudinal specimens at low stress levels. Transverse specimens containing seam welds show even slightly shorter fatigue lives. Transverse specimens that have been polished to remove die lines exhibit fatigue lives comparable to longitudinal specimens. Surface roughness of specimens with and without seam welds was characterized with standard profilometry techniques. Actual profilometer surface scans were used to create FEA models for evaluating stress concentrations associated with die lines, which appear to largely explain the reduced fatigue life of as-extruded transverse specimens.

4:15 PM
Effect of Si Distribution on the Fracture Toughness of A356 Aluminum Alloys: *Yong Nam Kwon*[1]; Kyuhong Lee[2]; Sunghak Lee[2]; J.-H. Lee[1]; [1]Korea Institute of Materials Science; [2]Pohang University of Science and Technology

Correlation of microstructure with mechanical properties of cast A356 alloys was investigated in this study. Three processes used for this study were low-pressure-casting, rheo-casting and casting-forging. Their mechanical properties and fracture toughness were analyzed in relation with microfracture mechanisms. All the cast A356 alloys contained eutectic Si particles segregated along solidification cells. Distribution of Si particles could be modified by casting-forging processes. Microfracture observation results showed that eutectic Si particles were cracked first, but that the aluminum matrix played a role in blocking crack propagation. Tensile properties and fracture toughness of the cast-forged alloy were superior to those of the low-pressure-cast or rheo-cast alloy according to the matrix strengthening and homogeneous distribution of eutectic Si particles due to forging process.

4:35 PM
Fatigue Behavior of Cast A319-T7 Aluminum Alloy: *Younghwan Jang*[1]; Sangshik Kim[1]; Jeonghwan Roh[2]; Gyejeong Lee[2]; Yoo In Jeong[3]; [1]Gyeongsang National University; [2]ACK Company, Ltd; [3]Korea Aerospace Industries, Ltd

The casting process inevitably produces defects, such as porosity and trapped oxide films, which are detrimental to the fatigue life. In this study, the effect of casting defects on fatigue behavior of cast A319-T7 aluminum alloy was investigated. Staircase fatigue test up to 10^7 cycles was conducted at an R ratio of -1 and at room temperature and 150°C, respectively. The characteristics of casting defects on the fracture surface of fatigue tested specimens, particularly at the initiation sites, were quantitatively identified by using SEM and image analyzer. It was found that the fatigue life was greatly influenced by the size of casting defects. The fatigue endurance limit for A319-T7 alloy specimen appears to be controlled by maximum stress intensity factor encompassing casting defect area. The effect of casting defects on fatigue behavior of cast A319-T7 aluminum alloy was discussed based on the fractographic analysis.

4:50 PM
Effect of Weld Tool Geometry on Friction Stir Welded AA2219-T87 Properties: *Joseph Querin*[1]; Judy Schneider[1]; [1]Mississippi State University

In this study AA2219-T87 panels were welded with three different pin tools. The pin tools were tapered and included 0 degree (straight cylinder), 30 degree, and 60 degree angles on the frustum. Friction stir welding (FSW) process parameters for each of the pin tool geometries were optimized to eliminate internal defects. The affects of heat, strain, and strain rate were investigated by maintaining a constant heat input while varying the process parameters of spindle speed and travel speed. This was performed in an attempt to maintain a constant heating for the weld panels while altering the strain rate. Variations in the material flow were investigated by use of microstructural analysis including optical microscopy (OM), scanning electron microscopy (SEM), and orientation image mapping (OIM).

Aluminum Reduction Technology: Process Control

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Aluminium Tech

Tuesday PM — Room: 298
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Geoffrey Bearne, Rio Tinto Aluminium Tech

2:00 PM Invited
Common Behavior and Abnormalities in Aluminium Reduction Cells: *Marco Stam*[1]; Mark Taylor[2]; John Chen[2]; Albert Mulder[1]; Renuka Rodrigo[3]; [1]Aluminium Delfzijl; [2]University of Auckland; [3]Heraeus Electro-Nite Company

The requirement of operating reduction cells at or beyond known performance limits is a new challenge for the aluminium industry. Most existing control systems are based on compensatory control actions with a short-term focus to move the controlled variable(s) back into the optimum region, and have independent strategies for alumina feeding and bath chemistry control. In order to incorporate natural cell behaviour into the control philosophy this paper investigates not only common cell behaviour, but also frequent cell abnormalities and failure mechanisms were studied. Statistical multivariate control surfaces, including alumina feeding, are presented for operating cells using three dimensional Hotelling T2 statistics. This approach allows a holistic decision making process on feeding, heat supply, compositional regulation and on troubleshooting cells. The liquid bath volume is an essential parameter control in control decisions, as is the bath superheat and liquidus temperature. These medium term state variations will be discussed as well.

2:20 PM
Novel Method of Measuring the Liquidus and Bath Temperature of Hall-Héroult Cells: *Hans Bohner*[1]; V. Arora[2]; T.K. Nasipuri[2]; [1]K&T Engineers Ltd; [2]Hamilton Research and Technology Pvt Ltd.

A procedure and equipment for measuring the liquidus and bath temperature of Hall-Héroult cells was developed and tested in PFPB pots at 65 and 185kA. Inconel-sheathed, reusable K-type thermocouples were inserted into stainless steel cups and immersed into the electrolyte for bath temperature measurement and withdrawn to obtain cooling curves. Undercooling was prevented by moving the thermocouple tip forth and back using a magnetically shielded electromotor. The liquidus temperature was determined electronically using the inflection point of the cooling curves or the "Noise" at the inflection point. The results in function of time and alumina feed rate indicate +/- 1°C temperature variation and were valid in 90% of all measurements (min. 16 pots and 120 measurements in each smelter). The cost of thermocouples is minimal as they can be used normally 100 times without deterioration.

2:40 PM
Analysis of a Potroom Performance Drift, from a Multivariate Point of View: *Jayson Tessier*[1]; Carl Duchesne[2]; Gary Tarcy[3]; Claude Gauthier[3]; Gilles Dufour[3]; [1]Alcoa Deschambault; [2]Laval University; [3]Alcoa Inc

Analysis of reduction cells performance could be a hard task for process engineers. One has to handle many highly correlated variables, where time lags and missing values must be covered. Performing an efficient analysis of complex potroom data does not always lead to good answers, for example; when an important production drift occurs. It is often necessary to include anode and alumina quality, which makes the analysis harder. The complications associated with the analysis of such database have limited engineers to analyze few variables at a time using univariate SPC charts, underutilizing the information hidden in extensive databases. In this paper, multivariate statistical analysis techniques are used to perform a combined analysis of potroom process data, anode and alumina quality. In this case study, the analysis is set up to investigate a specific drift in potroom performances, where time lag, anode residence time and correlation between many variables are encountered.

3:00 PM
Statistical Methods for Use in the Aluminum Smelting Industry: *Stephen Rutledge*[1]; [1]Boyne Island Smelters Ltd

The use of designed experiments for process improvement has been well understood since Sir Ronald Fisher's "The Designs of Experiments" published in 1935 [1]. However the application of these designs in the aluminium smelter reduction process has proven to be difficult. This paper details the problems that are relevant to the reduction environment. It also puts forward two techniques to improve the chances of obtaining stastically valid results from process improvement trials. The use of these techniques has allowed the rapid deployment of process changes in a variety of reduction lines and lead to significant improvements in performance, by destroying many of the myths that are common in the black art that is Aluminum smelting.

3:20 PM
Practical Applications of the Continous Measurement of Individual Anode Currents in Hall-Heroult Cells: *Roald Hvidsten*[1]; Ketil Rye[1]; [1]Elkem Aluminum

Traditionally, the anode current distribution is found by manual measurement of voltage drops across a fixed length of the anode rods. These measurements take time and are normally performed once per day or less. Anode currents change rapidly however, due to anode burnoffs, spikes and deformations, instable metal pad or incorrect anode setting height. These deviations will normally not be discovered until the next routine measurement. An instrument for continuous monitoring and display of individual anode currents has been developed and used at Elkem Aluminium Mosjøen for some years. The instrument removes the need for manual measurements and a signal feed into the computer control enables incorporation of the anode current distribution in the pot control system. Some practical applications of the continous measurement of anode currents are presented in this paper.

3:40 PM
Experiments on Wireless Measurement of Anode Currents in Hall Cells: Daniel Steingart[1]; *James Evans*[2]; Paul Wright[2]; Donald Ziegler[3]; [1]Wireless Industrial Technologies, Inc; [2]University of California; [3]Alcoa, Inc

The currents of individual anodes provide information on Hall cells, e.g. the occurrence of "metal roll". These currents are usually measured by voltage drops along anode rods but this is inconvenient for routine monitoring of pots because of the need to change anodes, plus the hazards of draping signal wires around. Experiments have been carried out on wireless measurement anode currents. Hall effect probes measured magnetic fields, reflecting mostly the current in the nearby rod. Signals were relayed wirelessly to a laptop some distance from the pot. Measurements have been carried out on both pots with pair-controlled anodes and pots with anodes on a fixed bridge. For the former, comparison with independent measurement is possible (from voltage drop along the anode flex). Although at the experimental stage, this investigation has served to show that wireless measurements agree roughly with other measurements; wireless measurements reveal metal roll and incipient anode effects.

4:00 PM Break

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Smart Feeders at Alumar Plant: *Haroldo Ferreira*[1]; Nilton Nagem[1]; Carlos Braga[1]; Pedro Leite[1]; Antonio Coimbra[1]; Eliezer Batista[1]; Elisio Bessa[1]; Edison Maia[1]; Ciro Kato[1]; Joel Camara[1]; Camões Pereira[1]; Roberta Camili[1]; Merval Aguiar[1]; [1]Alumar

Alcoa is searching continuously for alternatives to eliminate or reduce the environmental impact caused by operations. Alumar, an Alcoa unit in Brazil has implemented a new project to reduce anode effects. This project is based on the change from the current point feeder to smart point feeder technology. This change in technology is based on the anode effect frequency root cause, which is feeder plunger (more than 85%). The smart feeder is more than a sensor on the feeders, there is a computer routine that alarms failures and compensates if a feeder is not working properly. The test is on six pots and this group of pot shown around 50% anode effect frequency reduction. Other gains such as air consumption and noise reduction were archived.

4:30 PM
Breakthrough in Analysis of Electrolytic Bath Using Rietveld-XRD Method: *Frank Feret*[1]; [1]Alcan Inc

A standardless Rietveld-XRD method was devised for quantification of crystalline phases in bath electrolyte specimens. The phases present in the bath material are: cryolite, chiolite, fluorite, NaCa1.5AlF7, NaCaAlF6, corundum and diaoyudaoite. Two commercial software were employed in Rietveld quantification. Numerous advantages of the Rietveld method are compared with the traditional calibration approach. Departure from the need for calibration curves improves determination reliability and accuracy of all mineralogical phases. 38 Alcan bath standards were quantified for the phase composition by the new method. The CaF2 concentrations were calculated using fluorite, NaCa1.5AlF7 and NaCaAlF6 and compared with CaF2 measured directly. The ExAlF3 concentrations were calculated using chiolite, NaCa1.5AlF7 and NaCaAlF6. The Rietveld-XRD determined ExAlF3 concentrations agree to within ±0.35% with corresponding concentrations determined by wet chemistry. As a primary and universal method for determination of the phase composition in electrolytic bath the Rietveld-XRD method is replacing existing methodology in plant laboratories.

4:50 PM
Model Predictive Control of Superheat for Prebake Aluminum Production Cells: *Zeng Shuiping*[1]; Li Jinhong[1]; Peng Qiangqiang[1]; [1]North China University of Technology

Model Predictive Control (MPC) of superheat was investigated for 300kAprebake aluminum production cells. The control includes a predictive model, error feedback, and timely optimisation actions. The mathematical model was set up based on previous work and industrial experiments. It can predict bath temperature and bath liquidus temperature from the AlF3 additions, aluminum tapping magnitude and cell voltage set-point. The superheat in the production is measured and the difference between the measurement and prediction and its change rate are used to correct the parameters in the predictive model. If the average error and error change rate within a period are above a set value,the optimum program activates. This method has been used in the Hope Aluminum Company in Inner Mongolia for 3 months. Both current efficiency and DC energy consumption have visibly improved.

5:10 PM
The Application of Data Mining Technology in the National Aluminum Test Base: WangXing Li[1]; *QingYun Zhao*[1]; ShiLin Qiu[1]; Qiang Li[1]; [1]ZhengZhou Research Institute of CHALCO

At the National Aluminum Test Base (NATB, which is subordinate to Zhengzhou Research Institute of CHALCO), we have used data mining technology to settle some problems and achieved many improvements. In this paper, the application of this technology to lengthening the life of the cell, intensifying current, lowering cell voltage, reducing AE and increasing CE is described. So far, we have gained many achievements: current is intensified by 7%, energy consumption decreased by 500 kW.h/t-Al, the anode effect rate below 0.03 AE/cell.day, and the CE enhanced by 1% at least. There are many other data mining methods, but in this paper we just use some clear figures and tables to show you the usage and effectiveness of data mining. The use of data mining, combined with the good operating practice, can provide guidance for the optimizing of technical parameters.

5:30 PM
Discussion of Alumina Feeding Control Strategies: *Qi Xiquan*[1]; Li Shujie[2]; Wu Youwei[1]; Ma Shaoxian[1]; Mao Jihong[1]; Wang Dequan[1]; [1]Northeastern University Engineering and Research Institute, Company, Ltd.; [2]Shenyang University, Economy School

For alumina feeding control, many strategies have been adopted to keep a reasonable content range for high current efficiency and low anode effect frequency. Meanwhile, control strategies should be reflected by the historical curve on the supervising computer, which can be used to diagnose and analyze cell condition. In our opinion, a better control strategy should be characterized with lower daily RC times and reasonable feeding cycle duration. Based on this, some new ideas are incorporated into newly upgraded systems which showed rather better results. In this paper, new control philosophies are described systematically. Criteria to judge pot stability and control results as well as optimal daily RC times are put forward too.

5:50 PM
The Impact of Bath Ratio Control Improvements on Current Efficiency Increase: *Thiago Simões*[1]; João Alberto Martins[1]; Marcio Guimarães[1]; João Reis[1]; [1]Novelis

Current efficiency, one of the most important factors for primary aluminum production, is mainly determined by bath composition, bath temperature, current density, anode cathode distance (ACD) and cell design. According to Six Sigma concepts, to reduce the output variation it is necessary to reduce the input variation. Therefore for consistent current efficiency, the bath composition, bath temperature and ACD must have tight control in order to reduce their variation and keep them on target. This paper describes the Novelis process team's experience since 2002 working to improve potline performance. In the last four years the current efficiency has increased by about 1.7% and the current has been increased about 1.3 kA through process control improvements. This paper focuses on the impact of bath ratio control improvements on current efficiency increase and the operational changes that have contributed to drive the percentage of pots in bath ratio target above 85%.

Aqueous Processing - General Session: Aqueous Processing General Abstracts

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Aqueous Processing Committee
Program Organizer: Michael Free, University of Utah

Tuesday PM — Room: 281
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Shijie Wang, Kennecott Utah Copper Corporation

2:00 PM
Alternative Leaching Processes for Cobalt Oxide Minerals: *Cesare Ferron*[1]; Philippe Henry[2]; [1]HydroProc; [2]Hydrometal

Acidic dissolution of cobaltic oxide minerals require reducing conditions. Typically,reducing conditions are generated by using sulphur dioxide in a gaseous form or equivalent. The use of SO2 compounds can lead to serious health/safety issues, and can affect the recovery of other metals, namely copper. Alternative processes to dissolve cobaltic oxide minerals are presented here that overcome these two issues. The first alternative consists in using ferrous iron as the reductant, while the second process involves cathodic reduction of the cobaltic ion in a special design cell. Examples are provided for both alternatives.

2:25 PM
Continuous Circuit Production and Accelerated Ageing of Iron(III)-Arsenic(V) Coprecipitates - Probing Process-Stability Relationships: *Richard De Klerk*[1]; George Demopoulos[1]; [1]McGill University

The standard industrial method of arsenic removal from acidic aqueous processing effluents involves coprecipitation of arsenic with ferric iron (Fe/As>3) by lime neutralization in continuous circuits. To better understand current industrial practice, continuous circuit coprecipitation experiments were performed to identify industrially relevant process parameters and admixtures that contribute to improved arsenic removal and retention. The coprecipitated solids were subjected to accelerated ageing at 20°, 40° and 70°C and arsenic behavior was monitored and modeled. Staging and the presence of calcium ions were found to have the most significant impact on arsenic retention. In this paper the techniques used and major results obtained are discussed.

2:50 PM
Field-Deployable Aqueous Lead Analysis Using Nanoband Electrode System and Use of Green Rust for Its Removal: *Jewel Gomes*[1]; David Cocke[1]; Manohar Miryala[1]; Hector Moreno[1]; Eric Peterson[1]; Dan Rutman[1]; [1]Lamar University

Lead has long been an USEPA priority pollutant and an important environmental and health concern because of its toxicity at ppb levels. In light of health and exposure data for lead, the EPA has set standards for Maximum Contaminant Level (MCL) and MCL Goal (MCLG) as 15 ppb and 0 ppb, respectively. An accurate and rapid measurement of lead in the field remains still a technical challenge. In this work, a relatively new method deploying a nanoband electrode system using anodic stripping voltammetry has been optimized by changing deposition potential, electrolyte, and plating time used for the measurement of Pb(II) in water samples. Green rust has been proven to be an efficient intermediate matrix for removal of lead contaminants. We also reported here the removal efficiency of lead using green rust produced by electrocoagulation directly and indirectly. Lead containing floc was also characterized through XRD, SEM, and EDAX techniques.

3:15 PM Break

3:35 PM
The Influence of Chloride Ion to the Biooxidation of Arsenic Bearing Gold Concentration: *Dawen Wang*[1]; Hongying Yang[1]; Changliang Zhu[1]; Huanjie Jiang[1]; [1]Northeastern University

During the process of arsenic bearing gold concentration biooxidation, the content of chloride ion in water is a very important factor to the activity of bacteria and the oxidation of sulfide minerals. In this paper, the influence of underground mine water with different chloride ion content to the biooxidation of arsenic bearing gold concentration and the limit of chloride ion content to bacteria were summarized. These results indicated if the content of chloride ion was less than 1.2g/L, the bacteria's ability to oxidize sulfide minerals was little affected, and if the content of chloride ion were increased to 2g/L or more, the activity of bacteria was seriously restrained, and if the content of chloride ion were increased to 4g/L, the activity of bacteria was absolutely restrained.

4:00 PM
Hydrochloric Acid Leaching of Panzhihua Ilmenite for Preparation of Synthetic Rutile: *Jilai Xue*[1]; Zengjie Wang[1]; Haibei Wang[2]; Xunxiong Jiang[2]; [1]University of Science and Technology; [2]Beijing General Research Institute of Mining and Metallurgy

Investigation on hydrochloric acid leaching of the ilmenite from Panzhihua, China was carried out to prepare synthetic rutile. Various leaching parameters were optimized to obtain high content of TiO_2 in the products. The experimental data suggest that an optimum leaching can be realized with 30% acid, a solid/liquid ratio of 1/4 at 95°C, and ore ground for -400 mesh size. Kinetic data of the leaching was found to follow the spherical model f(a)=1-(1-a)1/3, and the apparent activation energy was calculated as 47.21 KJ/mol. After the acid leaching, an additional alkaline leaching process using hot NaOH solution was performed, and then the products was heat treated at 900°C in order to extract Si and increase the particle size of synthetic rutile. The final product has a high content of TiO_2 (about 95%), a low content of chlorine consuming elements (total CaO+MgO =0.07%) and a suitable particle size distribution for chlorination process.

Biological Materials Science: Scaffold Biomaterials

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Ryan Roeder, University of Notre Dame; Robert Ritchie, University of California; Mehmet Sarikaya, University of Washington; Lim Chwee Teck, National University of Singapore; Eduard Arzt, Max Planck Institute; Marc Meyers, University of California, San Diego

Tuesday PM — Room: 390
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Roger Narayan, University of North Carolina and North Carolina State University

2:00 PM Invited
Composites and Scaffolds for Calcified Tissue Regeneration: *Antoni Tomsia*[1]; [1]Lawrence Berkeley Laboratory

Despite extensive efforts in the development of fabrication methods to prepare porous ceramic scaffolds for osseous tissue regeneration, all porous materials have a fundamental limitation - the inherent lack of strength associated with porosity. Shells (nacre), tooth and bone are frequently used as examples for how nature achieves strong and tough materials made out of weak components. The objective of this study was to mimic the architecture of natural materials in order to create a new generation of strong hydroxyapatite-based porous scaffolds. The porous inorganic scaffolds were fabricated by the controlled freezing of water-based hydroxyapatite slurries. The scaffolds obtained by this process have an ordered and homogeneous lamellar architecture that exhibits striking similarities with the meso- and micro- structure of the inorganic component of nacre. These biomimetic scaffolds could be the basis for a new generation of porous and composite biomaterials.

2:30 PM Invited
Novel Scaffolds for Bone Tissue Engineering: *Fergal O'Brien*[1]; Matthew Haugh[1]; Claire Tierney[1]; [1]Royal College of Surgeons in Ireland

Tissue engineering relies extensively on the use of porous scaffolds to provide the appropriate environment for the regeneration of tissues and organs. Collagen-GAG scaffolds have shown excellent potential as a scaffold on which to culture cells and produce extracellular matrix. However, the scaffolds have limitations for bone tissue engineering, most notably, the mechanical properties of the scaffold are currently not suitable for use as a bone graft. In this study we sought to develop the optimal scaffold for bone tissue engineering by varying: (i) the crosslinking density to improve the mechanical properties of the scaffolds and (ii) the collagen and GAG content to investigate their effect on osteoblast viability. The results indicate that the use of crosslinking leads to a substantial increase in scaffold modulus while a 1% collagen and 0.2% GAG scaffold composition provides the best environment for osteoblast proliferation and bone formation. Acknowledgements: Science Foundation Ireland.

3:00 PM
Porous PLLA:PGA:PVA Scaffolds for Tissue Engineering: Mayur Uttarwar[1]; Ashwin Nair[1]; Liping Tang[1]; *Pranesh Aswath*[1]; [1]University of Texas at Arlington

In this study highly porous scaffolds were prepared by emulsion freeze dry method. PLLA: PGA (85:15) in chloroform solution was blended with PVA in a water solution. Four combinations of the scaffolds were obtained by varying blender speed and PVA concentration. The resultant scaffolds exhibited highly porous, interconnected structure with porosity more than 90%. The viability of scaffolds for cells attachment and growth was studied in vitro by culturing mouse 3T3 fibroblast cells for one week. The cells were quantitatively determined by following MTT assay. The attachment and location of the cells on the scaffolds was determined by florescent microscopy and SEM. The study gave important information about the pore size, porosity, pores distribution and the relative effect of these variables on cells behavior with scaffolds. The potential use of scaffolds fabrication method for tissue engineering with emphasis on growth factor release and controllable design is also discussed.

3:20 PM
Hydroxyapatite Whisker Reinforced Polyetherketoneketone for Bone In-Growth Scaffolds: *Gabriel Converse*[1]; Ryan Roeder[1]; [1]University of Notre Dame

Porous scaffolds are of interest for orthopaedic implant fixation. The scaffold should have porosity that promotes bone in-growth and mechanical properties sufficient to withstand physiological loads. Additionally, the scaffold biomaterial should be biocompatible, bioactive and amenable to providing osteoinductive growth factors. In order to meet these criteria, polyetherketoneketone (PEKK) was reinforced with 20-40 vol% hydroxyapatite (HA) whiskers. 40 vol% reinforcement resulted in an elastic modulus (17.9 GPa) and ultimate tensile strength (82.0 MPa) within the ranges reported for human cortical bone. Composites were loaded in four-point bending fatigue to 30 MPa for one million cycles without failure. Finally, HA whisker reinforced PEKK scaffolds with 75-90% porosity and 0-40 vol% HA whiskers were prepared using a particle leaching technique. HA whiskers were embedded within the polymer struts to act as reinforcement and were also exposed on the surface of polymer struts to promote growth factor retention and tissue attachment.

3:40 PM Break

3:50 PM Invited
Novel Polymer-Reinforced Calcium Phosphate Cement as Mesenchymal Stem Cell Carrier: Daniel Alge[1]; W. Goebel[2]; *Tien-Min Chu*[2]; [1]Purdue University; [2]Indiana University

Novel hydroxyapatite-based calcium phosphate cements reinforced with PLLA-based monomers have shown significant improvements in mechanical properties. In this study, we investigated the feasibility of using polymer-reinforced calcium phosphate cement (PRCPC) as a potential carrier for mesenchymal stem cells (MSCs). Rat MSCs were isolated from the femur bone marrow of Sprague-Dawley rats. CPC with starting ceramic powder ratio of hydroxyapatite: monocalcium phosphate monohydrate = 2:0.5, 2:1, 2:3, and 2:5 were reinforced with 5% PLLA and were seeded with MSCs. The cell proliferation profiles of the MSCs on the CPC with and without reinforcement were characterized. Three-dimensional PRCPC cubes with interconnected channels were seeded with MSCs and implanted subcutaneously on the backs of Sprargue-Dawley rats for two weeks. The in vitro characterization through MTT assay and in vivo characterization by histology and micro-CT will be presented. The potential of using three-dimensional PRCPC as scaffold for MSC carrier will be discussed.

4:20 PM
Porous Chitosan Scaffolds and Chitosan-Clay Nanoparticle Carrier for Advanced Drug Delivery Systems: *Wah Thein-Han*[1]; Y. Kitiyanant[1]; Devesh Misra[2]; [1]Mahidol University, Institute of Science and Technology; [2]University of Louisiana

In the advanced drug delivery and in tailored tissue engineering, modified chitosan nanoparticles and porous biodegradable three dimensional chitosan scaffolds are potential biomaterials for the improvement of drug and cell delivery systems. Chitosan nanoparticles incorporated with medical clay, montmorrillonite (MMT) and chitosan scaffolds of different degree of deacetylation (DD) were prepared by emulsion/solvent evaporation approach and combination of freezing and lyophilization methods, respectively. We describe here the physico-chemical analysis of nanoparticles and scaffolds. The studies suggest that the properties of chitosan-MTT nanoparticles and chitosan scaffolds have potential for drug delivery system.

4:40 PM
Resorbable Polymer Nanocomposites as Structural Tissue Engineering Constructs and Bone Graft Substitutes: *Kevin Baker*[1]; Rangaramanujam Kannan[2]; [1]William Beaumont Hospital; [2]Wayne State University

The development of biocompatible scaffolds is an area of intense research as tissue engineering-based solutions to orthopaedic surgical procedures gain popularity. Several techniques, including thermal induced phase separation, particulate leeching and gas foaming have been used to create porous polymer constructs. However, the maximum compressive strengths reported by researchers, render these constructs unsuitable for most load bearing applications. Addition of bioceramic fillers to the scaffolds have resulted in only modest gains in mechanical strength. Utilizing a supercritical-CO_2 ($scCO_2$) process, we have synthesized a novel, resorbable polymer nanocomposite (RPN), which consists of nanostructured Montmorillonite clay particles dispersed in a resorbable polymer matrix. Synthesis by the $scCO_2$ process imparts a porous architecture to the RPN constructs with a bimodal distribution of pore sizes (100-250µm, 10-50µm). The uniform dispersion of nanostructured clay particles achieved by $scCO_2$ processing reduces polymer chain mobility, leading to enhanced static and dynamic mechanical properties.

5:00 PM
Biological and Physicochemical Study of Tricalcium Phosphate-Silicone Rubber Composite: An Implantable Biomaterial System: *Qiang Yuan*[1]; Devesh Misra[1]; [1]University of Louisiana

We describe here the processing routes to successfully develop a new tricalcium phosphate-silicone rubber composite system that can be reliably used as an implantable biomaterial system. The objective here is to uniquely combine the chemical stability of silicone rubber with the superior biological characteristic of new bone formation by self degradation property of the tricalcium phosphate. A relative comparison of biological and physicochemical functions is made with silicone rubber to evaluate the viability of the implantable biomaterial.

5:20 PM
Fabrication of Porous Titanium Materials for Orthopedic Implant Applications: *Xing Yang Liu*[1]; Jianfeng Wang[1]; Ben Luan[1]; [1]IMTI, NRC

Titanium has been widely used in orthopedic implants due to their good biocompatibility, corrosion resistance, and mechanical properties. However, the elastic modulus of titanium alloys is still too high as compared with human bones. It is desired to further reduce the elastic modulus of the titanium alloys so as to minimize the stress-shielding effect of the implants. This paper presents the research work on the mechanical properties and microstructures of porous titanium produced by a powder metallurgy method so as to produce titanium materials with bone-matching modulus and good mechanical strength. The effect of porosity, particle size and impurity content on the bending strength and elastic modulus is discussed.

Bulk Metallic Glasses V: Structures and Mechanical Properties III

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter K. Liaw, University of Tennessee; Wenhui Jiang, University of Tennessee; Guojiang Fan, University of Tennessee; Hahn Choo, University of Tennessee; Yanfei Gao, University of Tennessee

Tuesday PM — Room: 393
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: T.G. Nieh, University of Tennessee; Y. Yokoyama, Tohoku University

2:00 PM Invited
On the Interpretation of Ductility Improvement in Bulk Metallic Glasses: *T. G. Nieh*[1]; Shuangxi Song[1]; [1]University of Tennessee

Reports on the ductility improvement in bulk metallic glasses (BMGs) will be reviewed first. The commonality of mechanical and structural characteristics will be noted and summarized. We will then present our recent results on the deformation of several bulk metallic glasses (BMGs). Some mechanical features such as yielding, flow serration, strain hardening, and perfectly plastic flow will be carefully analyzed and described. Several artifacts which can cause data misinterpretation will be especially pointed out. Finally, we will discuss the dominant deformation mode that occurs in the case of "ductility improvement".

2:20 PM Invited
Evolution of Large Plastic Strains in Metallic Glasses: In Situ Monitoring in a TEM: *Evan Ma*[1]; [1]Johns Hopkins University

For metallic glasses (MGs), the initial evolution of plastic strains in time and space has not been resolved. Here I discuss in situ tests, in tension and compression, of monolithic Zr-based and Cu-Zr-Al MGs in a TEM, employing samples with dimensions of the order of 100 nanometers. The tensile experiments were carried out in collaboration with H. Guo and M.L. Sui at SYNL (H. Guo et al., Nature Mater. 2007), and the nanocompression work was a joint project with Z.W. Shan of Hysitron and LBNL and J. Li at OSU (Z.W. Shan et al., submitted, 2007). Large ductility was observed, including uniform deformation and extensive necking or slow growth of the shear offset. The MGs deformed in a manner similar to their crystalline counterparts, without deflection/branching of shear bands or nanocrystallization. The sample size effects have implications for the application of MGs in thin films and micro-devices.

2:40 PM Invited

Fatigue Damage in Bulk Metallic Glasses: *Reinhold Dauskardt*[1]; [1]Stanford University

We present experimental and computational studies of the initiation of fatigue damage obtained from stress-life experiments and the growth of fatigue cracks measured under stable and transient cyclic loading conditions. The early stages of damage initiation and propagation result in low fatigue endurance limits. The effect of fatigue load ratio was investigated for a range of tension-tension, compression-tension and fully compressive loadings. Simulation of the effects of cyclic loading on the nature and propagation of shear bands was conducted using molecular dynamics computational models. The formation and evolution of shear bands under the influence of alternating loads will be described. The evolution of fatigue damage was experimentally characterized in terms of both "small" and "long" fatigue crack growth rate behavior to elucidate the mechanism of crack growth. A focused ion beam was used to introduce 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. Implications for the fatigue properties of metallic glasses are considered.

3:00 PM Invited

Modeling the S-N Behavior of Bulk-Metallic Glasses: *D. Harlow*[1]; Gongyao Wang[2]; Peter Liaw[2]; [1]Lehigh University; [2]University of Tennessee

Modeling the S-N behavior of bulk-metallic glasses (BMG) is critical for their acceptance in engineering applications as the material for structural members. Typically the S-N response of materials is characterized through empirically based statistical analyses of experimental data. This approach requires extensive amounts of data, and frequently laboratory testing does not adequately reflect long-term performance required of service conditions. Because of the inherent microstructural properties of BMG, experimental testing cannot identify all of the key sources and the extent of their contributions to the randomness in fatigue life, especially for the very high cycle regime. The primary sources of variability arise from the microstructure, environment, and loading. An approach for modeling the S-N response using standard fatigue crack growth modeling 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. Variability associated with manufacturing and material variables are considered. The effectiveness of this approach is demonstrated with an amalgamated set of S-N data for bulk-metallic glasses. Further consideration is given to three classes of BMG, *i.e.*, Zr, Cu, and Fe based BMG, in order to evaluate the effect of the different microstructural materials. Adoption of the proposed approach is recommended for sound scientific and probabilistic life prediction.

3:20 PM Invited

Fatigue of Zr-Based Bulk Metallic Glass: *Yoshikazu Nakai*[1]; [1]Hyogo University

Fatigue tests of smooth specimens and sharp-notched specimens of Zr-based BMG were conducted. The fatigue notch factor was identical to the stress concentration factor. In either specimen, shear step was formed just before fatigue crack initiation. The fatigue crack propagation tests were also conducted, and it was found that the fatigue crack propagation in air was cycles dependent and the rate was controlled by the stress intensity range or its effective component, independent of the stress ratio and the loading frequency. The crack growth rate in deionized water was almost identical to that in air. The rates in NaCl solutions, however, were much higher than that in air. In NaCl solution, the time-based crack propagation rate was determined by the maximum stress intensity factor independent of the loading frequency and the stress ratio, and the growth rate was almost identical to that of environment-assisted cracking under a sustained load.

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Microstructural Investigation and Mechanical Behavior of a Zr-Based Bulk Metallic Glass: *Matthew Freels*[1]; Y. Wang[1]; J. Jang[2]; D. Du[3]; K. Hsieh[3]; J. Huang[3]; Peter Liaw[1]; [1]University of Tennessee; [2]I-SHOU University; [3]National Sun Yat-Sen University

The microstructure of a (Zr53.1Cu29.8Ni9Al8.1)99.75Si0.25 bulk metallic glass (BMG) was investigated by x-ray diffraction (XRD), synchrotron high-energy x-ray diffraction (HEXRD), and transmission electron microscopy (TEM). XRD patterns showed a broad maximum, with no evidence of any obvious peaks. HEXRD patterns, however, exhibited distinct crystalline peaks. Thermodynamic calculations can predict the type of crystalline phase. Moreover, uniaxial compression and cyclic compression tests were performed. After failure, fracture surfaces were investigated via scanning electron microscopy (SEM). Proposed deformation mechanisms are discussed. 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.

3:55 PM

Study on the Metallic-Glass Coatings for Fatigue-Resistance Enhancements: *F. X. Liu*[1]; F. Q. Yang[2]; Y. F. Gao[1]; W. H. Jiang[1]; J. P. Chu[3]; P. D. Rack[1]; P. K. Liaw[1]; [1]University of Tennessee; [2]University of Kentucky; [3]National Taiwan Ocean University

Multicomponent metallic-glass coatings were deposited on structural substrates by magnetron sputtering. X-ray diffraction and high-resolution transmission electron microscopy were utilized to study the structure of the coatings. Mechanical properties of the coatings were investigated by nanoindentation and microscratch tests. Four-point-bend fatigue tests were performed on the coated materials. It was shown that the application of the metallic-glass coatings could improve the fatigue life and fatigue-endurance limit of the substrates. The high hardness and strength of the amorphous coatings, and the good adhesion to the substrates are the beneficial factors for the enhancement. The effects of various films, and different film thicknesses on the fatigue resistances of different substrates were discussed by a hypothetical micromechanical model. It was suggested that the suppression of the slip offset by the metallic-glass film (with the enhanced ductility by shear-band confinement) may delay fatigue-crack-initiation process, and therefore extend the fatigue life.

4:10 PM Break

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Effects of Partial Crystallization on Bending Fatigue Behavior of Zr-Based Bulk Metallic Glasses: *Gongyao Wang*[1]; Peter Liaw[1]; Y. Yokoyama[2]; Matt Freels[1]; A. Inoue[2]; [1]University of Tennessee; [2]Tohoku University

$Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Al_{10}Ni_{10}$, and $Zr_{50}Cu_{37}Al_{10}Pd_3$ (in atomic percent) are arc-melt tilt-casting bulk-metallic glasses (BMGs). Plate and rod specimens are fabricated. However the cooling rate of plate specimens was lower than that of rod specimens. Thus, the X-ray diffraction results exhibited that the rod specimens are fully amorphous alloys, but the plate specimens could be subjected to the partial crystallization. Four-point-bend fatigue experiments were performed on these zirconium (Zr)-based BMGs in air. The experiments were conducted at a frequency of 10 Hz, using an electrohydraulic machine with a R ratio of 0.1, where $R = \sigma s_{min.}/\sigma s_{max.}$, $\sigma s_{min.}$ and $\sigma s_{max.}$ are the applied minimum and maximum stresses, respectively. The fatigue-endurance limits of these BMGs with the partial crystallization were much lower than those of fully-amorphous alloys. These results suggested that the fatigue behavior of a bulk-metallic glass is very sensitive to the microstructure. A mechanistic understanding of the fatigue behavior is suggested.

4:30 PM
Mechanical Behavior of a Cu-Zr-Al-Ag Bulk Metallic Glass: *Matthew Freels*[1]; Peter Liaw[1]; Gongyao Wang[1]; W. Zhang[2]; A. Inoue[2]; [1]University of Tennessee; [2]Tohoku University

The mechanical behavior of a Cu45Zr45Al5Ag5 bulk metallic glass (BMG) was investigated under both uniaxial compression and compressive cyclic loading. The effect of frequency on fatigue behavior was studied. The fatigue experiments were performed using an electrohydraulic machine under a load control mode at frequencies of 10 and 25 Hz (sinusoidal waveform) with R = 0.1. Preliminary results show a high compressive strength and a fatigue endurance limit as high as 1550 MPa. Fracture surfaces were examined via scanning electron microscopy (SEM). Proposed deformation mechanisms are discussed. 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.

4:45 PM
Temperature and Strain-Rate Dependence of Shear Band Propagation in Zr-Based Bulk Metallic Glasses: *Florian Dalla Torre*[1]; Alban Dubach[1]; Jörg Löffler[1]; [1]Swiss Federal Institute of Technology

In this study the inhomogeneous flow behavior of Zr-based bulk metallic glasses (BMGs) is investigated as function of strain rate and temperature. In contrast to other BMGs the plastic strain exceeds 5% in these materials, which makes possible accurate measurement of the flow behavior under various conditions. At low temperatures the deformation kinetics is characterized by a positive strain rate sensitivity and smooth yielding, while above a critical temperature and strain rate the flow becomes serrated and the strain rate sensitivity is negative. A constitutive strain rate equation is proposed, which accounts for this flow behavior. Here serrated flow is understood to result from structural relaxation which occurs within shear bands; the propagation speed of the shear bands is strongly related to the applied temperature. TEM observations of samples deformed at various temperatures are also presented.

5:00 PM
Probe the Tensile Plasticity in Nanoscale Metallic Glasses: *Yinmin (Morris) Wang*[1]; J. Li[2]; A. Hamza[1]; T. Barbee[1]; [1]Lawrence Livermore National Laboratory; [2]Ohio State University

We present the experimental evidence that nanoscale metallic glasses may sustain appreciable tensile plasticity under certain confined conditions. We observe such phenomenon in tensile deformations of crystalline-amorphous nanolaminates with nanometer-sized (<10nm) Cu/Zr glass layers sandwiched between nanoscale copper crystalline layers. The appreciable amount of tensile plasticity developed in nanoscale amorphous layers and unusual overall high tensile ductility observed in nanolaminates are discussed in terms of the suppression of shear-banding mechanisms in nanometer-sized amorphous layers and the unique capability of amorphous phases that help to sink dislocations evolved from large plastic deformations. This work was performed under the auspices of the US Department of Energy by University of California, Lawrence Livermore National Laboratory under contract of No. W-7405-Eng-48.

5:15 PM
Nanoscale Heterogeneity Correlated with Shear Initiation in Monolithic Metallic Glasses: *Yongqiang Cheng*[1]; Hongwei Sheng[1]; En Ma[1]; [1]Johns Hopkins University

Monolithic glasses are usually considered homogeneous. However, on nanoscale, they may exhibit obvious heterogeneity, in terms of both structure and dynamics. This heterogeneity originates from the diversified configurations and spatial correlations of atoms at this scale. We discuss the impact of the nanoscale heterogeneity on the mechanical properties of amorphous materials. By computer simulation, we study the nanoscale heterogeneity of model metallic glasses by structural and dynamic analysis. Typical length-scale of heterogeneity, effect of cooling rate on the degree of heterogeneity, and fertile sites consisting of extraordinarily mobile atoms are investigated and characterized. To establish statistic correlations between shear initiation and structural/dynamic heterogeneity, we simulate early stage of plastic deformation with difference loading configurations. Triggering events, shear transformations, and collective excitation of shear transformation zones are observed and illustrated. Analytical models regarding shear initiation in monolithic metallic glasses are proposed and discussed.

5:30 PM
The Effect of Hydrogen Charging on Ln-Based Amorphous Materials: *ChihPin Chuang*[1]; P. Liaw[2]; R. Kuo[3]; J. Huang[1]; G. Yu[1]; W. Dmowski[4]; R. Li[5]; Tao Zhang[5]; R. Yang[6]; [1]National Tsing-Hua University, Department of Engineering and System Science; [2]Department of Materials and Engineering, University of Tennessee; [3]Division of Nuclear Fuels and Materials, Institute of Nuclear Energy Research; [4]University of Tennessee, Department of Materials and Engineering; [5]Beijing University of Aeronautics and Astronautics, Department of Materials and Engineering; [6]Argonne National Laboratory, Advanced Photon Source

In the present work, the effects of hydrogen charging on Ln-based amorphous alloys had been studied. The $(La_{0.5}Ce_{0.5})_{65}Al_{10}Co_{25}$ bulk metallic glasses (BMG) were charged with hydrogen by an electrochemical method in an alkali solution. The hydrogen concentration in the sample after a 36-hours charge can reach as high as 1,904 ppm. With the presence of hydrogen atoms, the hardness of Ln-based BMGs increased 80% comparing with the as-cast samples. The structural evolution of the amorphous matrix due to the high fugacity of hydrogen gas during the hydrogen-uptake process was investigated by the high-energy synchrotron X-ray scattering technique. The sample surface was crystallized after hydrogen charging. X-ray diffraction (XRD) measurements revealed broad crystalline peaks superimposed on an amorphous-scattering pattern. The crystalline phase grew through the surface to several hundred microns deep into the amorphous matrix. The atomic arrangements of both amorphous and crystalline phases were described by the atomic pair-distribution-function (PDF).

5:45 PM
The Effects of Surface Nanocrystallization and Hardening Process on the Mechanical Behaviors of Zr-Based Bulk-Metallic Glasses: *J. W. Tian*[1]; L. L. Shaw[2]; Y. Yokoyama[3]; P. K. Liaw[1]; [1]University of Tennessee; [2]University of Connecticut; [3]Tohoku University

A surface-treatment process, which can generate severe plastic deformation in the near-surface layer of crystalline materials, is applied on the Zr50Cu40Al10 bulk metallic glasses (BMGs). The experiment is implemented using twenty high-speed WC/Co balls to bombard the surface of the samples in an argon atmosphere. Plastic-flow deformation in the unconstrained sample edge was observed, which exhibits the good intrinsic ductility of the BMG materials in this experimental condition. Differential-scanning-calorimetry tests on the specimens show that possible crystallization may occur during the process. Surface hardness was improved by about 15% after three hours of the treatment, which may be attributed to the partial crystallization in the material and/or the reduced free volume. Four-point-bending fatigue behavior has been characterized and related to the modified surface structure and the compressive residual stress induced by the surface treatment. This work is support by the NSF combined research-curriculum development (CRCD) program under DGE-9987548.

6:00 PM
Instrumented Spherical Indentation of Bulk Metallic Glasses: Byung-Gil Yoo[1]; Byung-Wook Choi[1]; *Jae-il Jang*[1]; [1]Hanyang University

To analyze the unique low-temperature deformation in bulk metallic glasses (BMGs), here we performed the instrumented spherical indentation tests on BMGs using the various spherical indenters (having different tip radii from 1 to 250 μm). Unlike indentation with a geometrical self-similar sharp indenter (such as Berkovich and Vickers indenters), spherical indentation can induce the sequential development of stresses and strains in a material under the indenter, which means that one might observe the flow behavior by single indentation experiment. The results are discussed in terms of the evolution of plasticity during spherical indentation. This research was supported by the Korea Research Foundation Grant funded by the Korean Government, MOEHRD (Grant # KRF-2006-331-D00273).

Carbon Dioxide Reduction Metallurgy: Ferrous Industry

Sponsored by: National Materials Advisory Board, Metallurgical Society of the Canadian Institute of Mining Metallurgy and Petroleum, The Minerals, Metals and Materials Society, American Iron and Steel Institute, TMS Light Metals Division, TMS Extraction and Processing Division, TMS: Reactive Metals Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Neale Neelameggham, US Magnesium LLC; Masao Suzuki, Al Tech Associates; Ramana Reddy, University of Alabama

Tuesday PM Room: 294
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Lawrence Kavanagh, American Iron and Steel Institute; Kanchan Mondal, Southern Illinois University, Department of Mechanical Engineering and Energy Processes

2:00 PM
UCLOS: The European Steel Industry's Effort to Find Breakthrough Technlologies to Cut CO_2 Emmissions Significantly: *Jean-Pierre Birat*[1]; J. Borlée[1]; [1]ArcelorMittal Maizières

The European Steel Industry has been engaged since 2004 in an extensive, 5-year and 50M€+ program to develop breakthrough process technologies to produce Steel with a reduction of specific CO_2 emissions by at least a factor 2. This program, called ULCOS (Ultra Low CO_2 Steelmaking), will deliver a few process route concepts that meet this target and are ready for scaling up to a commercial size pilot by 2009. After screening a large number of potential routes in an extensive future studies, the program now focuses on 5 concepts: the oxygen blast furnace with top gas recycling and CCS, a bath smelting reduction process, also using pure oxygen and CCS, a new direct reduction, also oxygen and CCS-based, and two processes to carry out the electrolysis of iron ore; they will now be tested at fairly large scales. The approach may be of interest to other metallurgies than steelmaking.

2:20 PM
Recent Developments in Technology Management for Reduction of CO_2 Emissions in Metal Industry: *Malti Goel*[1]; [1]Department of Scienceand Technology

Ferrous and Non-ferrous industry in India is undergoing a transition to meet the growing demand and a new thrust to Research and Development (R&D) is mounting. On one hand, to meet the target laid by the policy in the Iron and Steel sector for 110 million tones production by 2020; on the other, innovations taking place in light metal production technology; both have necessitated greater attention to development of industrial ecological framework. Industrial ecology provides a number of new challenges as well as opportunities for the industry. The development and growth in a particular industry should have a trend towards reduced energy consumption, reduced resource consumption, thereby reducing the cost of production. Technology management strategies will have a key role in achievement of these targets. The paper describes recent developments in technology management, importance of R&D and case studies on adoption of energy efficient and CO_2 reduction technologies in metal industry in India. In the end suggestions for future are made. * The views expressed here do not represent organization's viewpoint.

2:40 PM
Impact on Greenhouse Gas Emissions of a Switch from Carbon to Hydrogen as the Principal Reducing Agent in Producing Metals: *James Evans*[1]; Brian Wildey[2]; [1]University of California; [2]Pacific Consolidated Industries

Carbon has been the principal reducing agent in producing metals for centuries. Carbon is an inexpensive reducing agent, but also an effective one, as any undergraduate who knows her Ellingham diagrams is aware. Even in the production of aluminum, an electrolytic process, carbon consumed at the anodes serves to reduce the voltage of the Hall-Héroult cell. The interest in reducing the amount of anthropogenic CO_2 has led to this examination of whether a switch to hydrogen as a major reductant is feasible and economic. The main source of hydrogen is natural gas and its production entails the generation of CO_2; however, that CO_2 is more easily captured than, say, the CO_2 leaving an iron blast furnace. Calculations and speculations lead to conclusions about whether there is significant benefit to be obtained from such a radical change in our way of producing metals and the approximate cost of such change.

3:00 PM
Sequestration of Carbon Dioxide by Steelmaking Slag: Process Phenomena and Reactor Study: *Von Richards*[1]; Simon Lekakh[1]; Charles Rawlins[1]; Kent Peaslee[1]; [1]University of Missouri

Steel-making processes generate carbon dioxide air emissions and a slag co-product. The aim of this project was to develop a functional sequestration system using steelmaking slag to permanently capture carbon dioxide emitted in steelmaking offgas. A possible parallel benefit of this process would be rapid chemical stabilization of the slag minerals with reducing swelling or leaching. This paper summarizes the original results of the project, including mineralogical and structural features of carbon sequestration with steel making slag, mathematical modeling of reaction phenomena using a modified shrinking core model, METSIM modeling of several possible industrial applications, a thermo-gravimetrical study of the reaction between slags and different gases, and design and testing for a lab scale apparatus consisted of two reactors.

3:20 PM Break

3:35 PM
Canadian Green Steel: Adopting Best Practices: *Jon Feldman*[1]; Edmund Smith[2]; Stephen Gale[1]; David Clarry[1]; [1]Hatch; [2]Hatch Energy

In 2006, Canadian independent steel maker, Algoma Steel Inc. embarked on an ambitious initiative to become "Best in Class in Energy Management". The initiative focussed on identifying energy/GHG management improvements leading to low-cost savings. They key components of the approach were: [1] Review site energy management practices and benchmark internationally. [2] Review best technical practices and benchmark against public energy intensity data. [3] Conducted a site-wide Energy Assessment and physical review of operating areas. [4] Facilitation of on site-workshops with staff to prioritise savings opportunities, identified and removed implementation barriers. [5] Development of Energy Management Action Plan for implementation of improvementsMost organisations can produce, with relative ease, a list of ideas for saving energy, implementing them is the challenge. This paper addresses these issues as well as the profitability of the energy efficiency improvements and the consequences for GHG emissions.

3:55 PM
Reduction of Ti-V-Magnetite with Microwave: Chenguang Bai[1]; *Liangying Wen*[1]; Feng Xia[1]; [1]Chongqing University

The situation of the Ti-V-Magnetite samples reduced with microwave has been investigated in lab. scale. The relationship between carbon consumption and microwave energy consumption for magnetite reducing processing has been studied. In different microwave power level the carbon usage for unit ore is different. While the output of power increase and irradiation time increase the carbon consumption will dicrease. The result approve that microwave is a good energy resourse that can be used in magnetite extractive procedure in order to reducing carbon dioxide emission.

4:15 PM Panel Discussion

Cast Shop Technology: Melt Handling and Treatment

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Hussain AlAli, GM Casthouse and Engineering Services, Aluminium Bahrain Company (ALBA); David DeYoung, Alcoa Inc

Tuesday PM — Room: 295
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Jonathan Prebble, Euroservice, Pyrotek Engineering Materials Ltd.

2:00 PM Keynote
Melt Treatment - Evolution and Perspectives: *Pierre Le Brun*[1]; [1]Alcan Inc

The use of aluminium alloys is increasingly oriented towards high quality end products, where aluminium enters into competition with other materials on a property/quality/cost basis. The aluminium industry has developed several technologies to provide the required quality of the aluminium alloy as function of the end application. Furnace treatment, in line treatment, and filtration are mainly used to tailor the quality. This paper summarises the evolution of these technologies in the last years, and illustrates the link with quality and product properties. The following aspects are integrated in the discussion: environment, productivity, recycling, flexibility, quality measurement. Finally some areas were developments are required or would be beneficial are discussed.

2:30 PM
Optimization of the Melt Quality in Casting/Holding Furnaces: *Bernd Prillhofer*[1]; Helmut Antrekowitsch[1]; Holm Böttcher[2]; [1]University of Leoben; [2]AMAG Rolling GmbH

There are several criteria which characterize melt cleanliness, e.g. hydrogen, alkali metal and the non-metallic inclusion content. According to the increasing quality demand of materials for high end applications (e.g. the aerospace industry) melt cleanliness has to be improved. Beside the in-line melt treatment, which is a very important step for rising melt quality, the content of impurities in casting/holding furnaces have to be reduced in front of for enhanced efficiency of in-line operations. This paper describes the optimisation possibilities for the casting furnace process. Based on CFD-calculations a methodology for melt treatment of AA 7075 has investigated and applied to the real process. Out of this, key factors can be derived and will be discussed.

2:50 PM
Variations in Aluminium Grain Refiner Performance and the Impact on Addition Optimization: *Rein Vainik*[1]; [1]Opticast Aluminium AB

A prerequisite for the optimization of the use of aluminium grain refiners is that the refiners exhibit a constant efficiency. Differences in efficiency will have a large impact on the possibility to optimize the use of grain refiners and will therefore lead to high costs for grain refinement. Furthermore, the risk of unwanted cast defects, e.g. cracking of ingots and billets, is greatly increased. A large number of aluminium grain refiners of different compositions have been investigated and the results show that there are large variations in performance, even if the nominal composition is the same. The test procedures used today are not discriminative enough and a new technique has been developed which can differentiate a good grain refiner from a bad one. The new procedure has been developed as an integral step in the implementation of the Opticast technology for grain refiner addition optimization.

3:10 PM
Improved Understanding of the Melting Behavior of Fused Magnesium Chloride-Potassium Choloride Based Refining Fluxes: *John Courtenay*[1]; [1]Melt Quality Partnership Limited

Fused magnesium chloride-potassium chloride refining fluxes have become widely adopted as a more environmentally acceptable means for removing alkali metals and oxides from molten aluminium than injection of chlorine gas. Refining fluxes were initially designed based on the binary system magnesium chloride-potassium chloride which exhibits two low melting point eutectics; one at 60% and one at 40% magnsesium chloride. In a further development products containing 25% magnesium chloride have found widespread adoption exhibiting equal removal efficiencies and satisfactory melting behaviour. The objective of the current work was to investigate the effect of fluoride additions and to reconcile the observed performance with the phase equilibria by conducting differential thermal analysis on industrial compositions and relating data collected to known binary and ternary systems. The findings indicate a significant beneficial effect from fluoride addition and the existence of a third low melting point eutectic in industrially produced compositions at 30 mole %.

3:30 PM
Development of an Improved System for the Filtration of Molten Aluminium Based on a Three Stage Reactor Employing a Cyclone as the Final Stage: *John Courtenay*[1]; Frank Reusch[2]; Andrey Turchin[3]; Dmitry Eskin[3]; Laurens Katgerman[4]; [1]Melt Quality Partnership Limited; [2]Drache Umwelttechnik GmbH; [3]Netherlands Institute for Metals Research; [4]Delft University of Technology

The developement of a new prototype multi stage filter is described in which a ceramic foam filter is applied in the first chamber operating in cake mode; grain refiner is added in a second chamber and in a final chamber a cyclone is deployed to ensure removal of any oxides or agglomerates arising from the grain refiner addition or release events from the ceramic foam filter. The concept of using a cyclone has been verified as an effective means of flow modification and inclusion removal in a flow modelling study prented seperately by A.N. Turchin et al. The first industrial prototype has been installed at Trimet Aluminium in Essen in Germany and the initial trial results with respect to inclusion removal efficiency and operational performance are reported.

3:50 PM Break

4:00 PM
Mechanisms of Inclusion Removal from Aluminum through Filtration: Lucas Nana Wiredu[1]; *Lifeng Zhang*[2]; [1]Norwegian University of Science and Technology; [2]Missouri University of Science and Technology

Alumina Ceramic Foam Filters (CFF) are used to remove inclusions from aluminium in laboratory experiments. Morphology and composition of inclusions in aluminium are identified using before and after filtration using optical microscope, SEM, XRD and EPMA. Qualitative analysis of results form various filtration experiments indicates that CFF filtration is an effective process to remove inclusions from molten aluminium. Interaction between inclusions and filter materials is theoretically investigated, which includes the effects of surface energy change before and after attachment, inclusion size, and fluid flow. Mechanisms of inclusion removal through filtration are then proposed.

4:20 PM
The Effect of Solid Particles on Foaming Process of Aluminum Melt: Hongjie Luo[1]; *Yihan Liu*[1]; [1]Northeastern University

The effects of endogenetic or introduced solid particles on bubble nucleation and its Stabilization was studied in this paper in producing aluminum foam by directly foaming in the melt. It was also analyzed the state of solid particles in aluminum melt and their distribution remained in foam block. The results show that the existence of solid particles, which are generated by adding Ca into aluminum melt and introduced through using TiH_2 particles as foaming agents, provides necessary conditions of heterogeneous nucleation of bubbles. These solid particles can suspended in the melt and be trapped in the course of bubble nucleation. The solid particles that can't act as nucleating agent may adhere to the generated bubble surface spontaneously and increase its Stabilization in aluminum melt. In the interior of obtained foam block, solid particles are mainly concentrated in foam layer and few of them appear in bubble-free layer.

4:40 PM
Predicting of Effect of Delivery System Configuration on Flow and Solidification in the Pool of Twin-Roll Strip Casting: *Bo Wang*[1]; Jieyu Zhang[2]; [1]Inner Mongolia University of Science and Technology; [2]Shanghai University

A wedge type metal delivery system has been designed for the twin-roll strip casting process. In this study, a three-dimensional mathematical model has been developed for the coupled analysis of fluid flow, heat transfer and solidification in the pool using finite difference method. The effect of difference delivery system configuration on flow pattern and solidification was predicted. The simulation results showed that it was desirable for the wedge metal delivery system to not only gain the uniform of flow and temperature in the pool, but also improve strip quality and ensure casing process.

5:00 PM Panel Discussion

Characterization of Minerals, Metals, and Materials: Characterization of Microstructure and Properties of Materials II

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee
Program Organizers: Jian Li, Natural Resources Canada; Toru Okabe, University of Tokyo; Ann Hagni, Intellection Corporation

Tuesday PM — Room: 284
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Jiann-Yang Hwang, Michigan Technological University; Tetsuya Uda, Kyoto University

2:00 PM
FSP Induced Structure Evolution in the HAZ/TMAZ of the Ni-Based IN738 Superalloy: *Oleg Barabash*[1]; Zhili Feng[1]; Stan David[1]; Rosa Barabash[1]; [1]Oak Ridge National Laboratory

The range of the application of the FSP is fast expanding to include IN738 superalloy strengthened by 50 vol % Ni_3AlTi-particles. By means of X-ray microbeam, OM, SEM and OIM structural transformations in the HAZ and TMAZ of this alloy due to FSP were studied. In the HAZ the dendrite orientation remains same. In the TMAZ rotation and bending of the dendrites take place. The character of Laue patterns changes from spot-like in the HAZ to the streaked ones in the TMAZ. In the immediate vicinity of the SZ (within 100 microns) recovery with the formation of the cellular substructure with low angle boundaries takes place. Partial coagulation of Ni_3AlTi-particles takes place in both zones. These processes increase approaching the boundary with SZ. At the boundary the complete dissolution of the Ni_3AlTi-particles takes place resulting to the transition of the alloy into ductile state.

2:20 PM
Laser Shock Peening of IN718 Superalloy: *Amrinder Singh Gill*[1]; Vijay Vasudevan[1]; S. R. Mannava[1]; [1]University of Cincinnati

LSP enhances service lifetimes of critical metal parts like aircraft engine fans and compressor blades.LSP dramatically improves fatigue strength, life and crack propagation resistance with shock wave-induced generation of deep compressive residual stress and microstructural changes. This study aims to understand effects of LSP parameters on residual stress distributions and microstructural changes in an important aero-engine material, IN718. Coupons of alloy with and without sacrificial layer were LSP-treated with varying energy densities using the GENIV system at GE Aircraft Engines. Depth-resolved characterization of macro residual strains and stresses and degree of cold work in peening direction and transverse to it was achieved using high-energy synchrotron x-ray diffraction at the Advanced Photon Source. Property changes were also studied using EBSD in SEM and TEM. Local property changes were examined using micro and nano-indentation measurements. Results showing the relationship between LSP processing parameters, microstructure, residual stress distributions and hardness are presented.

2:40 PM
Characterization of δ-Phase in the Nickel Based Alloy Allvac 718 Plus™: *Christof Sommitsch*[1]; Christoph Stotter[1]; Harald Leitner[1]; Julian Wagner[2]; Stephan Mitsche[2]; Martin Stockinger[3]; [1]University of Leoben; [2]Austrian Centre for Electron Microscopy; [3]Boehler Schmiedetechnik GmbH

Aerospace gas turbine disks operate in an environment of relatively high stresses caused by centrifugal forces and elevated temperatures. These severe conditions necessitate the need for materials with high temperature strength and good low cycle fatigue resistance. A new nickel based alloy Allvac 718 Plus™ shall further enhance the high temperature properties and is characterized. During hot forming, the grain structure development is strongly influenced by plate-like δ-phase (Ni_3Nb) with orthorhombic ß-Cu_3Ti structure. These plates look different from δ-Ni_3Nb investigated in Inconel 718. The phase appearance and fraction as a function of temperature was predicted both by thermodynamical calculations measured by DSC. The detailed phase identification was conducted by FIB, EBSD as well as TEM technique to investigate its morphology, volume fraction, crystallography and chemical composition.

3:00 PM
Microstructural Evolution and Mechanical Properties of IN740 Superalloy: *Hyojin Song*[1]; Quanyan Wu[1]; Manuj Nahar[2]; John Shingledecker[3]; Vijay Vasudevan[1]; [1]University of Cincinnati, Department of Chemical and Materials Engineering; [2]Indian Institute of Technology; [3]Oak Ridge National Laboratory

The IN740 Ni-base superalloy is a relatively new alloy that is being evaluated as a potential boiler material for ultra supercritical steam power systems under a ultra-supercritical condition Steam Boiler Consortium composed of industry, government laboratories and academia. In this study, microstructural evolution during aging of this alloy between 700 to 800°C for times to 3000 h and corresponding changes in tensile mechanical properties and fracture behavior are reported. Aging leads to rapid and substantial increase in strength, whose magnitude depends strongly on the aging temperature and time. Microstructure evolution was studied principally using SEM and TEM and the observations indicated that the strengthening is related to the formation of γ' precipitates. The fine structure of defects and their interactions with the γ' precipitates were characterized by TEM to disclose the deformation mechanisms and these results, together with the size and volume fraction data, was used to model strength.

3:20 PM Break

3:40 PM
Mechanical Behavior of Epoxy Matrix Composites Reinforced with Piassava Fibers: *Sergio Monteiro*[1]; Regina Coeli Aquino[2]; Denise Cristina Nascimento[1]; Luiz Fernando dos Santos[1]; [1]State University of the Northern Rio de Janeiro - UENF; [2]Centro Federal de Educação Tecnológica - CEFET–Campos

Natural fibers are being considered as reinforcement for polymeric composites owing to low cost, as compare to synthetic fibers, as well as environmentally correct renewable and biodegradable characteristics. This work investigates the mechanical behavior of epoxy composites reinforced with piassava fibers. These are stiff lignocellulosic fibers obtained from a Brazilian tropical palm tree, scientifically designated as Attalea funifera Mart. Composites with up to 40wt.% of piassava fibers embedded in epoxy matrix with different percentages of hardener were bend tested after 24 hours cure at room temperature. The fracture was analyzed by SEM. The results showed that the piassava fibers significantly increase the strength of the composite. A relatively strong fiber/matrix interface is partially responsible for the performance of the composite.

4:00 PM
Texture Transformation in Annealed Wire-Drawn OFHC Copper: *Daudi Waryoba*[1]; Ana Erb[1]; Peter Kalu[1]; [1]Florida A&M University-Florida State University College of Engineering

It is well documented that the texture of medium to high stacking fault energy (SFE) fcc materials, such as copper, consists of strong<111>+weak<100> fiber texture when deformed by wire drawing. The texture transformation on annealing, however, is not as clear. Although it is recognized that at low annealing temperature the wire-drawn texture evolves during recrystallization to a strong<100>+weak<111>, which later changes to a dominant <111> texture at higher temperatures, the effect of both annealing temperature and time on this transformation is not well understood. The objective of this investigation is to present a quantitative temperature-time-dependence of texture transition in annealed wire drawn OFHC copper. Two annealing procedures were performed: the first involves variable temperature at constant time, and the second involves constant temperature at variable time. Microtexture results from both experiments show that indeed the strong<100>+weak<111> is a metastable recrystallization texture, whereas the <111> texture is the stable orientation.

4:20 PM
Microstructural and Hardness Evolution of Roll-Bonded Cu-Nb Composite that Undergoes Annealing: *Chao Voon Samuel Lim*[1]; Anthony Rollett[1]; [1]Carnegie Mellon University

A metal-metal composite of Cu and Nb was made using the roll-bonding technique with layer thicknesses as small as 200 nm. Samples were annealed at temperatures of 300°C to 800°C and characterized with high resolution EBSD scans. This study reports the microstructural evolution with particular focus in the Cu layers and the hardness of the composite as a function of layer thickness and annealing temperature. The elongated grain structures observed in the as-deformed state were found to evolve into "bamboo-like" structures with increasing annealing temperature. No evolution was evident in the Nb layers below 600°C, although recovery may well take place. The growth of the grains in the thin layers was also found to be restricted by the phase boundaries. The hardness of the composite decreased with increasing annealing temperature but the total softening was the least for the composite with the thinnest layers, about 200 nm.

4:40 PM
Properties and Microstructural Characterization of Synthesized Brazilian Carbonado Diamond: Guerold Bobrovnitchii[1]; *Sergio Monteiro*[1]; Ana Lucia Skury[1]; Alan Monteiro[1]; [1]State University of the Northern Rio de Janeiro - UENF

Carbonado is the original denomination of a polycrystalline natural diamond found in Brazil and in the Central Republic of Africa. Carbonado-like diamonds can be synthesized at high pressure and high temperature from bulk graphite transformation in the presence of a molten solvent/catalyst metallic alloy. In the present work, the properties and microstructure of the first synthetic carbonado produced in Brazil were evaluated. The carbonado was transformed from Brazilian graphite inside a toroidal anvil chamber of a 6300 ton press at 1800°C and 7 GPa of pressure. The properties were evaluated by Raman spectroscopy, X-ray diffraction and microhardness determination. The microstructure was analyzed by SEM. The results showed typical properties of diamonds with a non-uniform distribution of hardness, which is a consequence of a temperature gradient inside the high pressure chamber. The microstructure was associated with metallic segregation at the grain boundaries in addition to polyhedral-shaped diamond crystals.

Complex Oxide Materials - Synthesis, Properties and Applications: Epitaxial Oxides: Ferroelectric, Dielectric, and (Electro-)Magnetic Thin Films

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division
Program Organizers: Ho Nyung Lee, Oak Ridge National Laboratory; Zhiming Wang, University of Arkansas

Tuesday PM Room: 277
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Susanne Stemmer, University of California, Santa Barbara; Paul Evans, University of Wisconsin, Madison

2:00 PM Invited
Effect of Strain and Crystal Structure on the Ferroelectric Properties of $BiFeO_3$: *Chang-Beom Eom*[1]; [1]University of Wisconsin-Madison

With a spontaneous polarization (Ps) over 100 μC/cm2 and its lead-free ferroelectric nature, $BiFeO_3$ has made it an extremely important material for non-volatile memory applications. Its simultaneous magnetism puts it in the rare class of magnetic ferroelectrics. Since the discovery of its incredible properties in thin-film form, the question of how strain and crystal structure affect the ferroelectric properties of $BiFeO_3$ still remains unclear, considering that bulk $BiFeO_3$ yields relatively small Ps values. Here we answer this question through measurements on the same epitaxial (001) $BiFeO_3$ thin-film capacitors before and after releasing them from the underlying Si substrate to which they are strained. Our measurements reveal that the Ps of $BiFeO_3$ is intrinsically large and the remanent polarization (Pr) has a strong strain dependence for strain in the (001) $BiFeO_3$ plane.

2:30 PM Invited
Stability of the Ferroelectric Polarization in Strained $BiFeO_3$ and Related Materials: *Hans Christen*[1]; [1]Oak Ridge National Laboratory

In epitaxial films of many ferroelectrics ($BaTiO_3$, $KNbO_3$), epitaxial in-plane strain results in a fundamental modification of the polar behavior (such as increases in the transition temperature). However, in this presentation we show results confirming theoretical predictions that this behavior is far from being universal. In fact, in $BiFeO_3$, the magnitude of the polarization changes only insignificantly over a very wide range of epitaxial strain (achieved by varying the thickness of the films on a given substrate), similar to the behavior in $PbZr_{0.2}Ti_{0.8}O_3$ [Lee et al., PRL v98 p217602 (2007)]. To further investigate this surprising stability of the A-site driven ferroelectricity, alloys between $BiFeO_3$ and the antiferroelectric $BiCrO_3$ were grown. In these $BiFe_{1-x}Cr_xO_3$ films, the ferroelectric phase remains stable even at x = 7/8. Research sponsored by the Division of Materials Sciences and Engineering and the Division of Scientific User Facilities (MDB), BES, US-DOE.

3:00 PM
Ab Initio Study of Surface and Surface Oxygen Diffusion Properties of $LaMnO_3$: *Dane Morgan*[1]; Yueh-Lin Lee[1]; [1]University of Wisconsin

The rate of the oxygen reduction reaction on $(La,Sr)MnO_3$ is a major limitation in developing lower temperature solid oxide fuel cells (SOFCs). To understand the relationship between surface structure and oxygen reduction we use ab initio methods to study the energetics of surfaces, near surface vacancies, and oxygen transport in $LaMnO_3$ (LMO). Vacancy formation energies near the most stable (100) surface are found to deviate dramatically from their bulk values, leading to orders of magnitude differences in vacancy concentrations between bulk and surface. The oxygen vacancy and surface binding energetics have been incorporated into a thermokinetic model to predict the oxygen coverage on the surface, near surface vacancy concentrations, and oxygen diffusivity on the surface and in near surface layers.

3:20 PM
Field-Assisted Reaction Sintering and Magnetic Properties of $FeCr_2S_4$: *Dat Quach*[1]; Veaceslav Zestrea[2]; Vladimir Kodash[1]; Vladimir Tsurkan[2]; Joanna Groza[1]; [1]University of California, Davis; [2]Academy of Science of Moldova

The novel Field-Assisted Sintering Technique (FAST) was successfully utilized to prepare bulk $FeCr_2S_4$ from a mixture of FeS and Cr_2S_3 powders. This technique significantly reduces the processing time from days in conventional solid-state synthesis to 10 minutes in FAST. FAST samples have high density (almost fully dense) compared to more porous ones synthesized by the conventional technique. Even though both types of samples have some similar magnetic properties, residual structural disorder in FAST specimens has a pronounced impact on the ferrimagnetic-paramagnetic transition and the orbital ordering at low temperatures.

3:40 PM Break

4:10 PM Invited
Materials Design for Piezoelectric Thick Films from Study on MOCVD-Grown Epitaxial Films: *Hiroshi Funakubo*[1]; Shintaro Yokoyama[1]; Satoshi Okamoto[1]; Keisuke Saito[2]; Takashi Iijima[3]; Ken Nishida[4]; Takashi Katoda[5]; Joe Sakai[6]; Takashi Yamamoto[7]; Hirotake Okino[7]; [1]Tokyo Institute of Technology; [2]Bruker AXS; [3]Advanced Institute of Science and Technology; [4]National Defence Academy and Kouchi University of Technology; [5]Kouchi University of Technology; [6]Japan Advanced Institution of Science and Technology; [7]National Defence Academy

Epitaxial Pb(Zr1-xTix)O3 [PZT] and (1-x)Pb(Mg1/3Nb2/3)O3-xPbTiO3 [PMN-PT] films, above 2 μm in thickness, were grown on (100)cSrRuO3//(100)SrTiO3 substrates by MOCVD. PbTiO3 content (x) dependencies of the crystal structure, dielectric and piezoelectric properties were systematically investigated for these films. The longitudinal electric-field-induced strain Δx33 and transverse piezoelectric coefficient e31,f for PZT films were also maximum at the mixed phase region, on the other hand, that for PMN-PT films were maximum at larger x edge of rhombohedral (pseudocubic) region. Almost the same order of Δx33 was observed under applied electric fields up to 100 kV/cm, while larger e31,f was observed in PMN-PT films compared with the case of PZT films. e31,f coefficients of ~-8.9 C/m2 and ~-11.0 C/m2 were calculated for the PZT film with x=0.46 and for the PMN-PT film with x=0.39, respectively. Based on these research, we discuss on the material design concept of piezoelectric thick films.

4:40 PM Invited

Combinatorial Discovery of a Morphotropic Phase Boundary in a Lead-Free High Tc Piezoelectric Perovskite: Shige Fujino[1]; *Nagarajan Valanoor*[2]; Makoto Murakami[1]; Sung Hwan Lim[1]; Anbusathaiah Varatharajan[2]; Lourdes Salamanca-Riba[1]; Manfred Wuttig[1]; Ichiro Takeuchi[1]; [1]University of Maryland; [2]University of New South Wales

The lead (Pb) content of Pb-based piezoceramics has recently raised environmental concern, and has created motivation to find a Pb-free replacement with comparable characteristics and simple perovskite structure. Though some guidelines which predict the presence of MPBs do exist, comprehensive mapping of compositions requires synthesis of an enormously large number of individual samples. We overcome this challenge by implementing a combinatorial synthesis strategy, where we have identified a property enhancing morphotropic phase boundary (MPB) in the $(Bi,Sm)FeO_3$ system, whose electromechanical properties represent substantial enhancement over those of simple $BiFeO_3$. We show a rhombohedral to triclinic to orthorhombic structural transition which exhibits a ferroelectric (FE) to antiferroelectric (AFE) transition at the MPB with piezoelectric properties comparable to those of PZT. Thus the newly discovered MPB sits at an interesting ferroelectric/antiferroelectric transition, and as a consequence we also demonstrate characteristics of a new Pb-free antiferroelectric.

Computational Thermodynamics and Kinetics: Functional Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, ASM Materials Science Critical Technology Sector, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Phase Transformations Committee

Program Organizers: Yunzhi Wang, Ohio State University; Long-Qing Chen, Pennsylvania State University; Jeffrey Hoyt, McMaster University; Yu Wang, Virginia Tech

Tuesday PM — Room: 288
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Long Qing Chen, Pennsylvania State University; Gunther Eggeler, Ruhr University

2:00 PM Invited

Domain Mechanisms in Giant Magnetostrictive Materials: *Yongmei Jin*[1]; Yongxin Huang[1]; [1]Texas A&M University

A phase field micromagnetic microelastic model is employed to study the domain mechanisms in giant magnetostrictive materials. The model explicitly treats magnetic-elastic domain microstructures, takes into account various magnetization-strain coupling mechanisms (intrinsic direct coupling through magnetostriction and extrinsic indirect coupling through magnetocrystalline anisotropy), and calculates multiple thermodynamic driving forces (magnetostatic, elastostatic, magnetocrystalline, exchange, chemical, twin boundary, applied magnetic and mechanical loading). Simulations of the coupled magnetic and elastic domain microstructure evolution are performed for two distinct giant magnetostrictive materials, namely, ferromagnetic shape memory alloy Ni-Mn-Ga and magnetostrictive Terfenol-D. The results identify domain mechanisms in these two types of materials and clarify their respective advantages and disadvantages, providing insight for new giant magnetostrictive materials development.

2:25 PM Invited

The Effect of Microstructure on the Local and Macroscopic Ferroelectric Switching of Polycrystalline Ferroelectric Films: *R. Edwin Garcia*[1]; [1]Purdue University

Ferroelectric Lead Zirconate Titanate (PZT) films display physical behavior that makes them an important candidate for several device applications. In such devices, polarization domains are locally switched by the application of an electric field, and the microstructural features are very important. In particular, large spatial variations of the fields arise as a combined result of the stresses that develop due to the thermal expansion and lattice mismatch of the film-substrate system, the anisotropy of the properties, and the processing conditions. Simulations show that in-plane electromechanical interactions electrically shield some of the grains, while enhancing the polarization response in others. Additionally, grain corners and boundaries become domain nucleation sites as well as domain pinning locations. The local phenomenology observed in these simulations agrees macroscopically with the observed experimental behavior, and is used as a basis to propose microstructural mechanisms that explain the local in-plane and out-of-plane ferroelectric switching behavior.

2:50 PM

Computer Simulations of Ferroelectric Domains and Switching Using Phase-Field Approach: *Samrat Choudhury*[1]; Yulan Li[2]; Long Chen[1]; [1]Pennsylvania State University; [2]Los Alamos National Laboratory

Application of ferroelectrics in information storage devices, involves switching of ferroelectric domains by an applied electric field. Conventional thermodynamic approach to describe switching behavior typically assume a material to be a perfect crystal while a real ferroelectric material is generally inhomogeneous and contains defects such as domains and domain walls, surfaces, grain boundaries, dislocations and dipolar defects. In this work, we developed a three-dimensional (3-D) phase-field model for predicting the domain structures and ferroelectric properties in presence of structural inhomogenities. The model takes into account realistic polycrystalline grain structures as well as various energetic contributions including elastic energy, electrostatic energy, and domain wall energy. It is shown that the defects such as existing domain walls and grain boundaries play a critical role in domain switching and the magnitude of coercive field. It will be demonstrated that ferroelectric properties obtained from phase-field simulation are in excellent agreement with experimental measurements.

3:10 PM

Phase Field Modeling of Morphotropic Phase Boundary Ferroelectrics: Weifeng Rao[1]; *Yu Wang*[1]; [1]Virginia Tech

Phase field model is employed to study some fundamental aspects of morphotropic phase boundary (MPB) ferroelectrics. MPB ferroelectrics offer the best electromechanical properties among all ferroelectric and piezoelectric materials. This talk will present some new insights into the underlying mechanisms responsible for the advanced piezoelectric behaviors around MPB. The new findings are domain wall broadening mechanism for domain size effect of enhanced piezoelectricity, bridging domain mechanism for phase coexistence, ferroelectric shape memory effect, composition-induced inter-ferroelectric phase transition, nanodomain microstructures of coherent ferroelectric phase decomposition, and adaptive diffraction phenomenon peculiar to nanodomains. Application of the gained understanding to other ferroic functional materials is also discussed.

3:30 PM Break

4:00 PM Invited

On the Role of Microstructure on the Martensitic Transformation of NiTi Shape Memory Alloys: *Gunther Eggeler*[1]; [1]Ruhr-University Bochum

NiTi shape memory alloys show fascinating properties like the one way effect and pseudoelasticity. These rely on thermally or mechanically induced martensitic transformations. Many atomistic, crystallographic and micromechanic aspects of the martensitic transformations are reasonably well understood. But this is not enough for making good shape memory alloys. There is a need to understand the role of microstructure during the martensitic transformation. Microstructures of shape memory alloys evolve during processing and consist of crystal defects including vacancies, dislocations, internal boundaries and particles which can all interact with each other and with the martensitic transformation. The first part of this overview presentation will recall some shape memory basics. It will then be shown how indiviual defects can interact with the martensitic transformation and what implications this has for shape memory properties. Challenges for modellers in view of experimental results will finally be highlighted.

4:25 PM
Effect of Ni_4Ti_3 Precipitation on Martensitic Transformations in Ti-Ni Based Shape Memory Alloys: *Ning Zhou*[1]; Chen Shen[1]; Martin Wagner[2]; Gunther Eggeler[2]; Yunzhi Wang[1]; [1]Ohio State University; [2]Ruhr-University Bochum

Precipitation of Ni_4Ti_3 plays a critical role in determining the martensitic transformation path and Ms temperature in Ni-Ti based shape memory alloys. In this study the concentration and stress fields around growing Ni_4Ti_3 precipitates are simulated quantitatively using the phase field method. Experimental data for lattice parameters of the parent and precipitate phases, precipitate/matrix orientation relationship, elastic constants and thermodynamic database are used as model inputs. Simulation predictions of the concentration and stress fields as function of precipitate size and spatial distribution are compared with experimental observations. The effects of the concentration and stress fields on subsequent martensitic transformations are investigated using a combination of the phase field method and the nudged elastic band method. These quantitative simulation results could be used for designing alloy composition and heat treatment schedule to precisely control the martensitic transformation temperature through manipulation of the size and density of Ni_4Ti_3 precipitates.

4:45 PM Invited
A Comparative Phase Field and Lagrangian Analysis of the Dynamics of the Martensitic Transition: *Alphonse Finel*[1]; Umut Salman[2]; [1]ONERA; [2]CNRS

When slowly driven by an external force, a martensitic transition is often characterized by a random sequence of burst or avalanches whose amplitudes display power-law distributions other several decades. This indicates that the martensitic volume fraction evolves without characteristic length and time scales, which is the signature of criticality. We present a comparative investigation of this critical dynamics in athermal martensite using both a Time-Dependant-Ginzburg-Landau modeling and a lagrangian method that takes into account inertial effects.

5:10 PM
Calculation of Impurity Free Energies by Ab-Initio Molecular Dynamics: *Chris Retford*[1]; Mark Asta[1]; Ben Haley[2]; Niels Gronbech-Jensen[1]; Michael Manley[3]; Christopher Woodward[4]; Dallas Trinkle[5]; [1]University of California, Davis; [2]Purdue University; [3]University of California, Davis/Lawerence Livermore National Laboratory; [4]Northwestern University/Air Force Research Laboratory; [5]University of Illinois, Urbana-Champaign

In first-principles calculations of the thermodynamic properties of solids, vibrational contributions are typically calculated within the framework of quasi-harmonic theory. This talk discusses approaches for calculating vibrational thermodynamic properties of solids when the quasi-harmonic theory is not applicable, namely for crystalline solids which are harmonically unstable at zero temperature. We discuss in particular an application to the calculation of site preference for ternary additions in the austenitic phase of the shape-memory NiTi compound. The formation free energy for Fe additions to NiTi alloys are computed employing a semigrand-canonical-ensemble relation, implemented within the framework of ab-initio molecular dynamics, which includes naturally electron-phonon coupling. The results of these calculations are discussed in the context of recent experimental studies of the effect of Fe additions on structural transitions in the NiTi(Fe) system.

Deformation Twinning: Formation Mechanisms and Effects on Material Plasticity: Experiments and Modeling: Twin Effects on Material Deformation II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: George Gray, Los Alamos National Laboratory; Subhash Mahajan, Arizona State University; Ellen Cerreta, Los Alamos National Laboratory

Tuesday PM — Room: 383
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Ellen Cerreta, Los Alamos National Laboratory

2:00 PM Invited
The Influence of Deformation and Microstructure on Twinning Processes: *Ian Robertson*[1]; Bryan Miller[1]; Cynthia Smith[1]; [1]University of Illinois Urbana-Champaign

Dislocation slip and twinning are the two common modes of deformation in metals. The latter is more common in metals with limited slip system availability, and under quasi-static loading rates at low temperature or high strain rate loading at higher temperature. In addition, deformation twinning has been reported to occur in high stacking-fault energy face-centered cubic metals with a nanometer grain size, although it does occur in large grains of these same materials in regions of high stress. Deformation twinning can also occur after extensive dislocation slip, which raises the question of how the twinning dislocations penetrate the existing dislocation structure. In this talk, the different conditions under which twinning occurs in face-centered cubic metals, the effectiveness of twins as dislocation sources and dislocation sinks, and a new mechanism for twinning in the presence of extensive slip will be presented and discussed.

2:30 PM
A Simple Non-Hardening Rate-Independent Constitutive Model for HCP Polycrystals Deforming by Slip and Twinning: *Babak Kouchmeshky*[1]; Nicholas Zabaras[1]; [1]Cornell University

A continuum approach is presented for predicting the constitutive response of HCP polycrystals using a simple non-hardening constitutive model incorporating both slip and twinning. A physically based methodology is introduced for restricting the amount of the twinning activity. A continuum approach is used for modeling the texture evolution that eliminates the need for increasing the number of discrete crystal orientations to account for new orientations created by twinning during deformation. The polycrystal is represented by an orientation distribution function using the Rodrigues parameterization. Numerical examples are used to show the application and accuracy of the methodology for modeling deformation processes.

2:50 PM
Microscopic Phase Field Modeling of Microtwinning in Ni-Base Superalloys during Creep Deformation: *Chen Shen*[1]; Ju Li[1]; Michael Mills[1]; Yunzhi Wang[1]; [1]Ohio State University

Recent investigations of creep deformation of Ni-base superalloys have demonstrated clearly the importance of twinning and reordering in determining dislocation-precipitate interactions and the corresponding kinetic pathways of the deformation process. Guided by concurrent experimental characterization we apply phase field modeling at different length scales to study the motion of dislocations in the g/g' microstructure and the formation of various planar faults in both precipitate and matrix under different loading conditions. In combination with ab initio calculations of generalized stacking fault (GSF) energy and the nudged elastic band (NEB) method, the effects of key material parameters and microstructural features on the minimum energy paths and activation energies of various deformation modes including microtwinning and isolated faulting are investigated. This work is supported by AFOSR through the MEANS program.

3:10 PM
Twinning during Low-Temperature Deformation of Sub-Microcrystalline Pulsed-Electrodeposited Nickel: *Lutz Hollang*[1]; Klemens Reuther[1]; Werner Skrotzki[1]; [1]Dresden University of Technology

Pure sub-microcrystalline nickel was produced by pulsed electro-deposition without additives for grain refinement. The average grain size of the material is d_{EBSD} = 150 nm and d_{XRD} = 25 nm if determined by electron backscatter diffraction (EBSD) and by X-ray diffraction (XRD), respectively. Tensile tests between 4 K and 320 K reveal that the material is ductile in the whole temperature range. Above a characteristic temperature T^* = 7 K the stress–strain curves are parabolic and the stress reaches its maximum after about two percent plastic strain, with the ultimate stress strongly increasing with decreasing temperature. However, during deformation below T^*, at a stress level of 2400 MPa the deformation mode suddenly changes to twinning. The twinning events are characterized by substantial stress drops accompanied by acoustic emissions. The microstructural changes connected with twinning will be discussed on the basis of results obtained by scanning and transmission electron microscopy.

3:30 PM
Size-Effects in Materials Deforming by Mechanical Twinning: *Javier Gil-Sevillano*[1]; Jon Alkorta[1]; Jon Molina[1]; [1]University of Navarra, CEIT and TECNUN

Size-effects associated to plastic strain gradients in materials deforming by dislocation-mediated slip have been thoroughly discussed in the recent past. By contrast, similar effects in materials deforming by mechanical twinning have been up to now ignored, despite the technological importance of some advanced twinning-deforming materials. This paper is a first attempt to analyze such effects both theoretically and experimentally. Results of the indentation size effect (ISE) in a high-Mn low stacking-fault energy TWIP will be presented.

3:50 PM Break

4:20 PM Panel Discussion: Carlos Tome, Moderator

Electrode Technology Symposium (formerly Carbon Technology): Anode Manufacturing and Developments

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Carlos Zangiacomi, Phelps Dodge International Corporation; John Johnson, RUSAL Engineering and Technological Center LLC

Tuesday PM — Room: 297
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Markus Meier, R&D Carbon Ltd; Alan Tomsett, Rio Tinto Alcan

2:00 PM
Anode Butts Automated Visual Inspection System: *Jean-Pierre Gagne*[1]; Marc-André Thibault[1]; Gilles Dufour[2]; Claude Gauthier[2]; Michel Gendron[2]; [1]STAS; [2]Alcoa, Inc

Anodes consumed in the smelting process for the production of primary aluminium need to be replaced regularly. Inspecting anode butts properties can be helpful to optimize the electrolysis process, reduce the anode cycle and optimize anode fabrication. In most of the aluminium smelters, manual measurements are taken on a sampling basis in order to obtain minimum feedback information. STAS-Alcoa's R&D team has been working to develop an automated inspection system; the intention was to design a low cost solution to automatically obtain a series of measurements on 100% of the anode butts. The prototype has been implemented at "Alcoa, Aluminerie de Deschambault", Canada, on May 2006. The inspection system, based on artificial vision is integrated within the existing conveyor system of the rod plant. All anode butts are inspected in line while they are transported on the conveyor. This paper presents some of the results obtained.

2:25 PM
New Rodding Shop Solutions: *Nicolas Dupas*[1]; [1]ECL

With more than 25 years of experience in rodding shop equipment design and commissioning, ECL is the leading supplier for modern smelters. All types of technology (2, 3, 4, or 6 stubs) are catered for, from individual machines to turnkey solutions: - Overhead conveyors, - Loading/unloading stations with unique centering mechanism, - Bath breaking and removal stations, - New hot-bath cleaning solutions, - Shot Blasting stations, - Butt stripping machines, - Thimble press, new simultaneous breaking design, - New butt stripping and thimble combination, - Stem control and preparation stations, - Casting stations: standard carriage, articulated carriage, casting crane, mating tables and carrousels. "Level 2" solutions allow a centralized computer control of the rodding shop to increase productivity, efficiency and facilitate maintenance. As for all of the smelter's equipment range, environment, health and safety are the top priorities in the design and commissioning of the rodding shop.

2:50 PM
Safe Operation of Anode Baking Furnaces: *Inge Holden*[1]; [1]Hydro Aluminium AS

The baking of anodes is a process in which combustible substances are released. Ring main fires do occur and even explosions in the ring main or fume treatment plant have happened in carbon plants. The risks associated with different process deviations and the possible concequences of these, can be evaluated for both existing and new furnaces to be built. European and IEC safety standards give useful guidelines for the design of process control and safety systems applicable to the baking process. This paper will present and discuss methods for evaluating the risks and consequences, and give examples for how operational procedures and the design of safety systems can reduce the occurence of unwanted events for open as well as closed top furnaces.

3:15 PM
Baking Furnace Optimisation: *Amer Al Marzouqi*[1]; Tapan Sahu[1]; Saleh Rabba[1]; [1]Dubai Aluminium Company Limited

Dubai Aluminium Company Limited (Dubal) began its operation in 1979 as a 135 000 tons/annum of primary aluminium smelter. The electrode requirements for its three pot lines were supplied by two closed-top baking furnaces. Today, Dubal is a 890 000 tons/annum producer of primary aluminium operating four open top Alcan-Alesa baking furnaces beyond their design capacity, to meet its continuously expanding anode requirements. Recent increases in pot line amperage have demanded the use of larger anodes of varying configuration which has made the furnace operation extremely complicated. For the past three years Dubal has optimised its baking furnace operations to improve baked anode quality and increase productivity, with the support of R&D Carbon. This paper describes the improvement measures taken during these three years, through systematic implementation of various innovations in baking furnace practices, which have resulted in increased production and improved anode quality at Dubal.

3:40 PM Break

3:50 PM
New Concept for a Green Anode Plant: *Michael Kempkes*[1]; Werner Meier[1]; [1]Buss ChemTech AG

Buss ChemTech, the leading supplier for anode production since 1950's producing about 80% of world's anodes with individual machines to turn-key solutions. Moreover, with increasing smelter capacity from 50'000 t/a to today's smelters reaching above 1 million t/a the throughput of anodes increased while quality of raw materials steadily decreases requiring higher attention in production to archive stable results. Success of any smelter depends on the performance of carbon, green carbon and anode baking from start-up through its operation. Furthermore, future changes in all regulations need to be incorporated into new designs to reduce construction time, assure safe, efficient and stable operation, address present and future environmental regulations while still being most cost effective. This paper outlines the future green anode plant design build around highly reliable equipment, avoidance of unneeded equipment and simplification of process steps to formulate the concept for producing the perfect quality anodes.

4:15 PM
Modelling of Anode Thermal Cracking Behavior: *Odd Einar Frosta*[1]; Trygve Foosnæs[1]; Harald Øye[1]; [1]Norwegian University of Science and Technology

Carbon anodes used in the aluminium reduction process are consumed and replaced with new ones. The new anode is exposed to a severe thermal shock as it is submerged in the molten bath. The thermal shock induces thermal stress that sometimes initiates cracks in the carbon anode. Temperatures and stresses in a carbon anode have been modelled using three-dimensional finite element methods through the modeling program ANSYS. The first model grid was based on a homogeneous anode. Inhomogeneity was introduced stepwise by giving the grid varying properties according to analyses of core samples drilled throughout industrial anodes. Modeling gradients of several material properties through the anode can now be done. The result is an anode model in which certain parameters can be altered in order to determine optimal thermal shock resistance (TSR).

4:40 PM
Characteristic and Development of Production Technology of Carbon Anode in China: *Guanghui Lang*[1]; Rui Liu[1]; Kangxing Qian[1]; [1]Sunstone Carbon

With rapid developments in Chinese aluminium industry, technology in carbon anode production has quickly improved. Sunstone construction project reflected the characteristics and development of Chinese technology in carbon anode production. The main characteristics are shown as following: Pot – type calcining furnaces for petroleum coke are widely used in China with a steady quality, low investment and long service life; Weigh batchers used in blending procedure; mixer used in kneading procedure and vibratory compaction make the green anode with composition of high precision, good effect in kneading and high density; anode quality is closed to international level. The main technique developments are shown as following Emphasis on quality of raw materials; Development of large size in pot – type calcining furnaces; Emphasis on environmental protection and utilization of waste heat; More attentions on homogenization of anode quantity.

Electrode Technology Symposium (formerly Carbon Technology): Cathodes Raw Materials and Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Carlos Zangiacomi, Phelps Dodge International Corporation; John Johnson, RUSAL Engineering and Technological Center LLC

Tuesday PM — Room: 299
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Alexander Proshkin, Rusal; Claude Gauthier, Alcoa Inc

2:00 PM
ISO Standards for Testing of Cathode Materials: *Harald Oye*[1]; [1]Norwegian University of Science and Technology

ISO/TC 226 (Materials for he Production of Primary Aluminium) recently published a CD with 109 ISO Standards and 2 ISO Technical Specifications. covering the materials: Alumina, Pitch, Coke, Anodes, Cathodes and Ramming Paste. All standards are easily accessed from the CD. In addition to the test procedures for common room temperature properties, methods which characterizes the cathode materials at operational conditions has been developed. Examples are: Sodium expansion of cathodes with and without external pressure, rammability of paste and expansion and shrinkage of ramming paste. Additional studies of cathode property changes as function of temperature and time enables room temperature ISO standards to be extrapolated to operational conditions. The standards are not only useful for material evaluation, but also important for development of more reliable mechanical-thermal-electrical models.

2:25 PM
Test and Analysis of Nitride Bonded SiC Sidelining Materials: Typical Properties Analyzed 1997-2007: *Egil Skybakmoen*[1]; Jannicke Kvello[1]; Ove Darell[1]; Henrik Gudbrandsen[1]; [1]SINTEF Materials and Chemistry

During the last ten years, SINTEF has tested a large number of different commercial nitride bonded SiC sidelining materials produced world-wide. The following properties will be summarized in the paper: Apparent porosity, density, mineral phase analyses (α-Si_3N_4, ß-Si_3N_4, Si_2ON_2, Si, and SiC), chemical analyses (level of Al, Fe, Ca and Ti), LECO analyses (total oxygen and total nitrogen), bending strength, hot modulus of rupture (HMOR), cold crushing strength, thermal expansion, thermal conductivity, oxidation resistance, and chemical resistance. Some comparisons between blocks with good and bad properties will be given, as well as some examples of variation in properties within the same block.

2:50 PM
High Swelling Cold Ramming Paste for Aluminum Reduction Cell: *Galina Vergazova*[1]; [1]Rusal, Engineering and Technology Center Ltd.

Life and energy efficiency of a reduction cell largely depend on the sealing efficiency of the ramming paste that determines the extent and rate of electrolytic penetration into the cell bottom. The problem of the ramming paste quality and its densification in the joints is especially acute for more and more widely used graphitic and graphitized cathode blocks with their very low sodium swelling index and heat expansion factor. High swelling capacity of ramming pastes can be an avenue of attack on the problem. The work presents a study to control the swelling capacity and caking with ramming paste blocks based on typical electrically and gas-calcined anthracite and pitch binder by selection of size distribution and liquid and solid additives.

3:15 PM
Wear Mechanism Study of Silicon Nitride Bonded Silicon Carbide Refractory Materials: *Ron Etzion*[1]; James Metson[1]; [1]University of Auckland, Light Metals Research Centre

Unused Silicon Nitride Bonded Silicon Carbide (SNBSC) bricks from different manufacturers were studied using x-ray powder diffraction (XRD), SEM-EDS, XPS and Solid state NMR. Powder XRD analysis reveals that the ratio of α to ß Si_3N_4 varies significantly in different samples and in different zones of the brick. In samples which contain higher ß Si_3N_4 its concentration decreases from the core to the exterior. The high ß Si_3N_4 content in the core of some bricks could be attributed to poor temperature control and the exothermic nature of the nitridation reaction. This would account for the higher internal temperatures conducive to ß Si_3N_4 formation. Core samples with high ß Si_3N_4 content show higher corrosion rates compared to samples with high α Si_3N_4 content. The corrosion test reveals that the degradation mechanism consist of a combination of bath penetration into the sample followed by gas attack.

3:40 PM Break

3:50 PM
Chemical Resistance of Sidelining Refractory Based on Si_3N_4 Bonded SiC: *Richard Laucouret*[1]; Didier Lombard[1]; Abdellatif El Bekkali[2]; Catherine Bessada[2]; Jacques Poirier[2]; Veronique Laurent[1]; [1]Alcan; [2]Centre de Recherche sur les Matériaux à Haute Température

The state of art for the sidewall lining in modern electrolysis cells is Si_3N_4 bonded SiC refractories. The chemical resistance of sidewall materials is one the key factors of the pot lifetime. ALCAN intends to improve its electrolysis technology by identifying and selecting the most chemical resistant materials to the oxidation and chemical dissolution by the cryolithe. The present study is aimed at improving our knowledge of corrosion mechanism of Si_3N_4 bonded SiC materials and determining the key parameters of materials structure which impact on the behaviour of these refractories in operation conditions. The chemical resistance in cryolithe of SiC and Si_3N_4 powders was investigated by XRD, the behaviour of some Si_3N_4 bonded SiC samples was studied in two oxidizing atmospheres: dry and wet air and the influence of the permeability of materials on their oxidation was established.

4:15 PM
Sodium Vapour Degradation of Refractories Used in Aluminium Cells: *Asbjørn Solheim*[1]; Christian Schoning[2]; [1]SINTEF; [2]SINTEF Materials and Chemistry

The bottom lining in aluminium cells can be degraded by several mechanisms. In the present work, reaction with sodium vapour that diffuses through the cathode was studied ("dry attack"). The reaction paths depend on the oxygen level. At reducing conditions, metallic Si is formed, whereas the presence of oxygen gives Na_2O. Based on available thermodynamic data for the compounds present in the system SiO_2-Al_2O_3-Na_2O, each alkemade triangle in the phase diagram could be supplied with numbers for the equilibrium pressure of sodium (or sodium and oxygen), as well as lines showing the change in composition during attack. The calculations show that chamotte bricks normally end up as a mixture of nepheline and albite. This is in accordance with practical observations. The reducing nature of sodium was verified through laboratory experiments, and autopsies of old linings have revealed the presence of Si-metal particles due to the reduction of SiO_2-containing compounds.

4:40 PM
The Effect of Current Density on Cathode Expansion during Start-Up: *Arne Ratvik*[1]; Anne Støre[1]; Asbjørn Solheim[1]; Trygve Foosnæs[2]; [1]SINTEF; [2]Norwegian University of Science and Technology

During start-up of aluminum cells, sodium penetration causes expansion in the carbon cathode, which may influence the lifetime of the cathode lining. Traditionally, the sodium expansion has been measured up to cathode current densities up to 0.75 A/cm². However, it is well known that the current distribution in the cathode is non-uniform, and high local current densities may be experienced close to the sideledge, which commonly is associated with the W wear pattern. Hence, the Na expansion may cause both local stresses in the cathode blocks, as well as in the total cell lining. The aim of this study is to determine the sodium expansion in a wider range of current densities. Typically, it is found that the sodium expansion starts to increase again above 0.7 A/cm², after the plateau reached at 0.2 A/cm². Apparently, this second increase continues outside the range of 1.5 A/cm² applied in this work.

5:05 PM
Change of Electrical Resistivity of Graphitized Cathode Block during and after Electrolysis in Alumina Molten Salt: Hiroshi Imagawa[1]; *Yoshinori Sato*[1]; Noboru Akuzawa[2]; Manabu Hagiwara[2]; [1]SEC CARBON, Limited; [2]Tokyo National College of Technology

It is considered the intercalation between graphitized carbon and sodium in molten salt is one of causes of erosion of graphitized cathode used in aluminium reduction cell. The objective of research is to obtain knowledge about deterioration of graphitized cathode by measuring changes of electrical resistivity (ER) during/after electrolysis. As a result of research, it was found when graphitized sample is used as cathode, ER of sample rapidly decreases from start of electrolysis and reaches the lowest after 30 to 50 minutes, and then increases gradually. Upon interruption of electrolysis, lower ER suddenly increases and return to value of pre electrolysis after reaching the plateau area. The behavior of ER change differs by material and heat treatment temperature of sample. Further, the change of ER obtained by repetitive cycle of ER measurement at electrolysis and its interruption and analysis results of samples after the electrolysis is reported in this paper.

Emerging Interconnect and Packaging Technologies: Pb-Free Solder: Tin Whisker Formation and Mechanical Behavior

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Carol Handwerker, Purdue University; Srinivas Chada, Medtronic; Fay Hua, Intel Corporation; Kejun Zeng, Texas Instruments, Inc.

Tuesday PM — Room: 275
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Thomas Bieler, Michigan State University; Indranath Dutta, US Naval Postgraduate School

2:00 PM Invited
Tin Whisker Formation: Relationship between Intermetallic Formation, Stress and Whisker Nucleation: *Eric Chason*[1]; K.S. Kumar[1]; Nitin Jadhav[1]; Lucine Reinbold[1]; [1]Brown University

Whiskers in pure tin platings limit the reliability of lead-free electronics. To understand the fundamental mechanisms controlling whisker formation, we have made simultaneous measurements of the intermetallic (IMC) growth kinetics, stress evolution and whisker density. We find that the IMC initially grows rapidly and then slows down as the growing layer blocks diffusion of copper into the tin. The corresponding stress saturates after a small amount of IMC is formed and the whiskers start to grow only after the stress saturates. We interpret these results in terms of a model in which the volumetric strain around the growing IMC particles leads to dislocation and diffusional creep processes. Cross-sectional TEM measurements show that plastic relaxation around the precipitating IMC leads to the formation of subgrain boundaries. The presence of the surface oxide is thought to play a critical role in the stress evolution by preventing relaxation at the surface.

2:30 PM Invited
Sn-Whiskers: What Do We Know: *John Osenbach*[1]; [1]LSI Corporation

Although a number of significant improvements in Sn-plating bath chemistry and process have been developed to reduce the probability of Sn-whisker growth on electronic components with a pure Sn surface finish, Sn-whiskers remain a concern for the electronics industry. This talk will focus on what is known and unknown about Sn-whisker nucleation and growth, the effectiveness of mitigation strategies, and the current state of understanding on how to predict when and under what conditionswhisker growth induced field failures may occur.

3:00 PM
Thermo-Electromigration Induced Sn Whisker Growth in Pb-Free Flip Chip Solder Joints: *Fan-Yi Ouyang*[1]; K.N. Tu[1]; [1]University of California, Los Angeles

In-situ Sn whisker growth in flip chip solder joints of Pb-free 96.5Sn-3Ag-0.5Cu has been studied at temperature of 150°C. Voids were found to form on the cold side in the neighboring un-powered bump, which means thermomigration drives Sn atoms to the hot end (silicon side). Also, electromigration pushes Sn atoms to the anode side. This combined effect of thermomigration and electromigration, which we defined as thermo-electromigration, pushed Sn atoms to move in the same direction in the bump with upward electron flow from the substrate to the silicon chip, thus produced a concentration of compressive stress near the exit where the current leaves the solder bump. The stress results in the accelerated growth of Sn whisker in the current crowding region.

3:15 PM Break

3:30 PM
Prevention of Sn Whisker Formation by Surface Treatment of Sn Plating Part II: *Keun-Soo Kim*[1]; Sun-Sik Kim[1]; Seong-Jun Kim[1]; Katsuaki Suganuma[1]; Masanobu Tsujimoto[2]; Isamu Yanada[2]; [1]Osaka University, Institute of Scientific and Industrial Research; [2]C. Uyemura and Company, Ltd.

Establishment of lead-free plating technology and whisker countermeasures is one of the critical problems remaining to be solved for lead-free electronics packaging. Recent researches have revealed the mechanisms of Sn whisker formation and growth. However, more researches are required to find acceptable methods to prevent Sn whisker formation. We propose a new approach to prevent the Sn whisker by surface treatment on pure Sn plating. Ni, Au and Pd layer with the thickness of 0.05μm or 0.2μm was deposited on typical Sn plating by flash-coating process. These samples and pure Sn plating samples were stored in room ambient environment. Comparing with pure Sn plating, Ni/Sn, Au/Sn and Pd/Sn plating is much stable against Sn whisker formation in room ambient environment. Ni/Sn, Au/Sn and Pd/Sn plating samples significantly suppressed the Sn whisker formation under the compressive stress condition.

3:45 PM
Numerical Simulations of Stress Relaxation, Including Whisker Nucleation and Growth, in Mechanically Stressed Sn Films: *Eric Buchovecky*[1]; Allan Bower[1]; Eric Chason[1]; Sharvan Kumar[1]; [1]Brown University

Three-dimensional finite element simulations are used to study the mechanisms of stress relaxation, including the nucleation and growth of whiskers, in a polycrystalline Sn film under compressive loading. Our simulations explicitly model long-range, stress-driven diffusion of Sn through Sn-Sn grain boundaries, as well as anisotropic elasticity and dislocation plasticity within Sn grains. Within a textured, columnar microstructure typical of electroplated Sn films, we examine the role of anomalously oriented grains and non-vertical grain boundaries in producing stress concentrations capable of nucleating a whisker grain (fracturing the native oxide; inducing sliding along vertical grain boundaries). After a whisker nucleates, the rate of whisker growth and the relaxation of stress in the surrounding grains are measured. The results of our study are compared with recent experimental measurements of stress gradients surrounding individual whiskers.

4:00 PM
Effect of Aging on High Strain Rate Deformation and Fracture Behavior of Sn3.8Ag0.7Cu Solder Joints: P. Kumar[1]; *Indranath Dutta*[1]; V. Sarihan[1]; D. Frear[1]; M. Renavikar[1]; [1]US Naval Postgraduate School

With the proliferation of mobile electronic devices, solder joints are frequently subjected to loading under high strain rates. This paper studies the influence of microstructural coarsening on (i) the flow behavior of Sn-3.8Ag-0.7Cu under compression over strain rates ranging from 0.1 to 30s-1, and (ii) the fracture behavior of Sn-3.8Ag-0.7Cu joints at nominal strain rates of 1-100s-1. Yield strength and work hardening rate were observed to increase substantially with increasing strain rate, with the strain rate sensitivity at higher temperatures being greater. Low temperature aging (25-50°C) appeared to enhance yield strength slightly, but decreased the work hardening rate. With aging at higher temperatures, both yield strength and work hardening rate decreased dramatically. Associated with the alteration of flow behavior, transitions in fracture behavior and joint toughness were noted. Correlations between joint microstructure and the observed fracture mechanisms will be highlighted. Supported by NSF-DMR-0705734, Freescale and SRC.

4:15 PM
Strain-Rate-Dependant Mechanical Properties for SnAgCu and SnAgCu-X Solder Alloy: *Luhua Xu*[1]; John H.L. Pang[2]; [1]University of California, Los Angeles; [2]Nanyang Technological University

SnAgCu solders have limited mechanical strength and are within the creep range. Their properties of solders are highly dependent on test temperature and strain rate. The elastic modulus and yield strength of Sn3.0Ag0.5Cu and Sn3.0Ag0.5Cu-X (X=200ppm Ni, Ce) solder alloys were studied at 25, 75, 125°C and a comprehensive range of strain rate from low strain rate (10E-5~10E-2 1/sec) using uni-axial tensile test and extremely high strain rate (104-105 1/sec) by employing Hopkins bar test. The intermediate strain rate test (10E-3~10E1) were conducted by using Nano-indentation continuous stiffness measurement. A statistical method was employed to quantify the strain-rate-dependent mechanical properties. For all the tests conducted, the higher the strain rate the higher the elastic modulus and yield stress of the solder; the higher the temperature the lower the elastic modulus and yield stress. The modulus and yield stress of SnAgCu-X solders are larger than those of the SnAgCu solder.

4:30 PM
Mechanical Properties of Lead-Free Solder Joints – The Size Effect: Peter Zimprich[1]; Usman Saeed[1]; Brigitte Weiss[1]; *Herbert Ipser*[1]; [1]University of Wien

It has been known for some time that various mechanical properties of solder joints depend critically on their size, especially on the gap to thickness ratio. This can have serious effects on brittleness and ductility of these joints and, as a consequence, on their long time stability. Thus it was the aim of this study to investigate the influence of decreasing gap size on tensile, shear, and stress relaxation behavior of solder joints, and to find possible variations with changes in the solder and/or substrate material. The model solder joints were prepared from Sn-3.5Ag and Sn-10.0In-3.2Ag solders using both Cu and Ni substrates. They were of rectangular form with gap sizes varying from 850 down to 25 µm, and they were obtained by a reflow soldering procedure in order to achieve near-industrial process conditions. The microstructure and the complex fracture and crack propagation modes were characterized by scanning electron microscopy.

4:45 PM
Measurement of Impact Toughness of Eutectic SnPb and SnAgCu Solder Joints in Ball Grid Array by a Micro-Impact Tester: Yuhuan Xu[1]; Shengquan Ou[1]; King-Ning Tu[1]; *Kejun Zeng*[2]; Rajiv Dunne[2]; [1]University of California, Los Angeles; [2]Texas Instruments Inc.

The most frequent failure of wireless, handheld, and portable electronic products is an accidental drop to the ground. The impact may cause interfacial fracture of BGA solder joints. Existing metrology, such as ball shear and ball pull tests, cannot characterize the impact induced high speed fracture failure. In this study, a micro-impact tester was utilized to measure the impact toughness and to characterize the impact reliability of both SnPb and SnAgCu solder joints. The annealing effect at 150°C on the impact toughness was investigated. The impact toughness of SnAgCu solder joints with the plating of electroless Ni/immersion Au (ENIG) became worse after annealing, decreased from 10 or 11 mJ to 7 mJ because of a ductile-to-brittle transition. On the other hand, an improvement of the impact toughness of eutectic SnPb solder joints with ENIG was found after annealing, increased from 6 or 10 mJ to 15 mJ.

5:00 PM
Microstructural Modification of Thick Copper Films to Optimize Chemical-Mechanical Planarization of Through-Wafer Interconnects: *Patrick Andersen*[1]; Mariela Bentancur[1]; Megan Frary[1]; [1]Boise State University

Large through-wafer interconnects may be required for high current applications or novel devices like backside connected solar cells. The electroplated copper film required to fill large vias is typically much thicker than conventional films and has a much different microstructure. Previous work has shown films with smaller, more equiaxed grains tend to have lower removal rates and different topography after chemical mechanical planarization (CMP). Here we alter the plating parameters (bath chemistry and current density) and thermal treatments in order to create different microstructures and measure their effects on CMP outputs. Electron backscatter diffraction is used to quantify microstructural parameters such as grain size and crystallographic texture. We find that annealing thick copper films after complete room-temperature relaxation has a significant impact on CMP outputs such as removal rate and surface roughness. The results of this work could be applied to optimize CMP of thick copper films and through-wafer interconnects.

Emerging Methods to Understand Mechanical Behavior: Subscale Methods: Tension and Compression

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Brad Boyce, Sandia National Laboratories; Mark Bourke, Los Alamos National Laboratory; Xiaodong Li, University of South Carolina; Erica Lilleodden, Forschungszentrum

Tuesday PM — Room: 285
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Erica Lilleodden, Forschungszentrum; Nicholas Randall, CSM Instruments

2:00 PM Invited
Size Effects in Metal Deformation and the Role of Stress Concentrations: *Cynthia Volkert*[1]; Daniel Gianola[2]; Reiner Moenig[2]; [1]Institute of Materials Physics, University of Goettingen and Institute of Materials Research II, Forschungszentrum Karlsruhe; [2]Institute of Materials Research II, Forschungszentrum Karlsruhe

A wide range of studies on the strength of polycrystalline and single crystalline metals support the idea that "smaller is stronger". In this presentation, an explanation for this trend will be proposed that is based on requiring critical stress concentrations in a critical sized volume for dislocation nucleation. This will be discussed in detail for micro-compression specimens, where the stress concentrations are attributed to sticking between the sample and the punch. Possible extensions to the case of bulk and thin film polycrystals will also be presented and used to provide a justification for Hall-Petch behavior. Such a picture calls into question the existence of size effects in homogeneously loaded, micrometer-size specimens. Tensile testing of single crystal metal nanowires by in-situ manipulation in a dual beam SEM/FIB is proposed as a method to distinguish between size effects from homogeneous and inhomogeneous stress states.

2:30 PM
Microtensile Testing of Nanocrystalline Thin Films for MEMS: *Timothy Rupert*[1]; John Sharon[1]; Daniel Gianola[2]; Kevin Hemker[1]; [1]Johns Hopkins University; [2]Forschungszentrum Karlsruhe, Institute for Materials Research II

Microtensile testing techniques have been employed to characterize the scale-specific mechanical behavior of materials for MEMS through the study of nanocrystalline thin films. The challenges and opportunities associated with these techniques will be reviewed and case studies involving micro-scale specimens presented. MEMS structures are often deposited far from microstructural equilibrium and the mechanical properties of these materials will be shown to be strongly dependent on processing, structure, and temperature. The mechanical behavior of nanocrystalline thin films has been shown to be dependent on microstructural stability. Of particular interest is the extended plasticity that occurs as a result of discontinuous grain growth, where grain boundary pinning and stress-assisted grain boundary migration appear to be important. Experiments to probe the stress dependence of this mechanism will be highlighted and the fact that the mechanical behavior appears to not only be different than that of microcrystalline metals, but also dynamic, will be discussed.

2:50 PM
Mini-Tensile Experiments of Clock-Rolled Zirconium Plate: *George Kaschner*[1]; Manuel Lovato[1]; Michael Stout[1]; Gwénaëlle Proust[1]; Irene Beyerlein[1]; Carlos Tomé[1]; [1]Los Alamos National Laboratory

We present our efforts to measure the tensile strength of clock-rolled pure zirconium in the through-thickness direction of the plate. Such measurements are relevant to benchmarking our constitutive models of hardening and texture evolution. We have designed a fixture and sample to perform tensile tests on our 9mm thick plate. The sample is a double-legged mini-tensile sample: 8mm x 8mm x 1mm overall; each leg has a gage section of 1mm x 1mm x 3mm. In contrast, our standard "macro" tensile sample is a flat dogbone with a gage section of 3mm x 1.5mm x 25mm. We validate our design by comparing the results of meachanical tests performed on samples of both geometries. Although the hardening response is nearly identical, the flow stress of the miniature samples is offset by +25 MPa at the onset of plastic yield. We present our efforts to resolve the origin of this offset.

3:10 PM
Tensile Testing with the Double Ligament Specimen: *David Alexander*[1]; Cheng Liu[1]; [1]Los Alamos National Laboratory

The double-ligament (DL) tensile specimen has been used to measure the tensile properties of 6061-T651 aluminum plate. These results have been compared to conventional tensile tests. The DL specimen can be used to measure through-thickness tensile properties in plates as thin as 0.25 in. The strain fields in the DL specimen have been measured during the test by using full-field digital image correlation methods for strain mapping. The effects of sample geometry on the strain distribution in the DL specimen, and the resultant effects on the stress-strain data from the DL test are discussed.

3:30 PM
Room and Elevated Temperature Validation of a Novel Sub-Scale Electrothermomechanical Tester: *Benjamin Peterson*[1]; Daniel Huber[1]; Peter Collins[1]; Hamish Fraser[1]; [1]Ohio State University

The Electrothermomechanical Tester (ETMT) is a fully instrumented load frame with software to control direct resistive heating of subscale coupons during thermomechanical excursions and mechanical testing. This talk will focus on the determination of the mechanical properties of alpha/beta and beta processed Ti64 that were heat-treated to produce an extended range of properties. The tests were conducted at several temperatures, including room temperature, and the results compared to identical conventional tensile testing. A size scale exists that is dependent upon the microstructure, and will be discussed. Digital image correlation is employed to measure strain. This has been used to characterize the local and macro strain distribution due to inherent temperature variations along the length of the samples. The statistical variation between samples is also discussed. Once validated, this method offers an attractive alternative to conventional testing for the population of databases relating composition, microstructure, and properties.

3:50 PM
Tension-Tension Cyclic Testing of Sub-Micrometer Copper Freestanding Thin Films: *Ming-tzer Lin*[1]; Kai-Shiang Shiu[1]; Chi-Jia Tong[1]; [1]National Chung Hsing University

A specially designed apparatus to carry out a series of tension-tension cyclic testing of submicron freestanding copper films attached on electroplated frame integrates pin holes, spring and load sensor beam is demonstrated. The sample fabrication involves three steps of lithography and two steps of electroplating to hold a dog bone freestanding copper thin film. The gage section of films is 600x100μm with a thickness of 300~900nm. The remaining thin film outside the gage section maintained good adhesion to the frame that would subsequently be gripped for tensile testing. It was then loaded by performing monotonic and tension-tension fatigue experiments at 10Hz up to 10^7cycles. Loading was applied by piezoelectric actuator and loads were measured by capacitor load cell. All samples tested here passed 10^3 cycles under constant stress amplitude below yielding and a trend of decreasing cycles to failure with increasing loading amplitude and increasing mean stresses has been noted.

4:10 PM Break

4:25 PM Invited
Mechanical Behavior/Microstructure Relationship in Cu/Nb Multilayers Tested via Micropillar Compression: *Nathan Mara*[1]; Dhriti Bhattacharyya[1]; Pat Dickerson[1]; Richard Hoagland[1]; Amit Misra[1]; [1]Los Alamos National Laboratory

Mechanical testing of nanoscale multilayers has been largely limited to nanoindentation, with few studies carried out to examine bulk mechanical behavior, such as tensile testing of freestanding samples or micropillar compression testing. In this work, the mechanical behavior of Cu/Nb multilayers are evaluated using three test methods: micropillar compression, nanoindentation, and tensile testing of freestanding samples. Through the use of Focused Ion Beam (FIB) milling, post-deformed microstructures are examined via Transmission Electron Microscopy (TEM). Individual layer thicknesses tested range from 100 nm to 5 nm, with flow stresses ranging from ~1.1 GPa to nearly 3 GPa, respectively. Remarkable deformability is demonstrated in these materials during micropillar testing, with 40 nm Cu/Nb exhibiting 30% strain to failure and 5 nm Cu/Nb exhibiting strains in excess of 20%. The unique behavior of these materials will be discussed in terms of interfacial effects on dislocation motion.

4:55 PM
Characterization of Damage of Ferritic ODS Alloys with Advanced Micro-Sample Methods: *Wolfgang Hoffelner*[1]; Manuel Pouchon[1]; Jiachao Chen[1]; Johann Michler[2]; Maria Samaras[1]; [1]Paul Scherrer Institute; [2]Swiss Federal Laboratories for Materials Testing and Research (EMPA)

Oxide dispersion strengthened (ODS) steels are candidate materials for advanced electric energy and heat generationg plants (nuclear, fossil). Understanding the degradation of mechanical properties of these alloys as a result of service exposure is very important. For advanced nuclear applications combination of temperature, irradiation and stress are important damage conditions which are preferentially studied with subsized samples like nano-indentation, micro-pillar or thin strip testing due to size constraints given by irradiation. Irradiation hardening, thermally and/or irradiation induced phase reactions and creep (irradiation induced, thermal) deformation were investigated with these methods. The results were linked with with transmission electron microscopic investigations. Ferritic ODS steels with 16 to 20% chronium were investigated. The materials were studied in qualities differing in grain sizes and in sizes of the dispersoids. Irradiation was performed in an accelerator using He ions and protons. The interpretation of the results with multiscale modeling will be discussed.

5:15 PM
A Micro-Compression Study of Shape-Memory Deformation in U-6wt%Nb: *Amy Clarke*[1]; Robert Field[1]; Patricia Dickerson[1]; Rodney McCabe[1]; John Swadener[1]; Robert Hackenberg[1]; Donald Brown[1]; Dan Thoma[1]; [1]Los Alamos National Laboratory

Quenching U-6wt%Nb results in a martensitic phase transformation from the high temperature, bcc γ phase to a monoclinic α'' structure. This material has been shown to display the shape-memory effect (SME), where deformation proceeds by twinning and twin rearrangement via boundary migration within the SME regime. A theoretical single crystal analysis, which incorporates the Bain strain and the parent γ phase orientation relationship with the product martensite, successfully predicts polycrystalline deformation structures and texture evolution within the SME regime. In the current study, micro-compression samples from single γ grains of a polycrystalline sample have been fabricated using focused ion beam milling and tested in a nano-indentation instrument to directly investigate the single crystal behavior and determine the influence of orientation on stress-strain behavior. Microstructure characterization was performed using orientation imaging microscopy (OIM) and transmission electron microscopy (TEM).

5:35 PM
Size Effects during Compression of Passivated Micropillars: A Comparison between Dislocation Dynamics and Experiments: *Lucia Nicola*[1]; Erik Van der Giessen[2]; Alan Needleman[3]; [1]Delft University of Technology; [2]University of Groningen; [3]Brown University

Several experiments have shown a size dependent flow strength of micropillars under compression. The size effect is even more pronounced when the pillars are passivated. The aim of this study is to attain a better understanding of the size effect in the pillars by modeling their plastic deformation using two dimensional discrete dislocation plasticity and comparing the results to experimental findings. To capture the size effect, we rely on the length scales intrinsic in the model, i.e. the dislocation Burgers vector and the average spacing between dislocation sources. Additional length scales that may appear during evolution of the dislocation structure, like the length of dislocation pile-ups, will also play a role. By changing not only the pillar diameter but also its height (and therefore volume) some insight can be gained in issues like nucleation controlled hardening and dislocation starvation.

5:55 PM
Fabrication and Mechanical Properties of Miron-Sized Nip Amorphous and Composite Pillars: Xiao Qiang Zhang[1]; *Yi Li*[2]; [1]Singapore-MIT Alliance; [2]National University of Singapore

The array of micro-sized pillars of NiP amorphous alloy and its composite are fabricated by electro-plating into the UV-lithography defined photoresist mask. This method offers a batch of pillar samples with various shapes for the investigation of the mechanical behavior of amorphous and its composites in micro-size under compressive load. The mechanical properties of the amorphous alloy were studied by micron-compression conducted in a nanoindenter. The effects of samples' geometry and microstructure on the compressive mechanical properties are reported.

Enhancing Materials Durability via Surface Engineering: Novel Surface Durability Approaches

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: High Temperature Alloys Committee
Program Organizers: David Mourer, GE Aircraft Engines; Andrew Rosenberger, US Air Force; Michael Shepard, Air Force Research Laboratory/MLLMN; Bruce Pint, Oak Ridge National Laboratory; Brian Gleeson, University of Pittsburgh

Tuesday PM — Room: 388
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Brian Gleeson, University of Pittsburgh; Bruce Pint, Oak Ridge National Laboratory

2:00 PM Introductory Comments

2:05 PM Invited
The Use of Copper to Prevent High Temperature Surface Attack by Carbon: *David Young*[1]; Jianqiang Zhang[1]; [1]University of New South Wales

Carbon-rich reducing gases can attack heat resisting alloys by internal carburisation and/or metal dusting. Both processes involve alloy supersaturation by carbon, followed by precipitation of either carbides or graphite at or near the surface. The addition of copper to pure nickel largely suppresses dusting at 680°C. This benefit is also achieved for 304 and 310 stainless and Alloy 800, but is limited by copper solubility in these alloys. The basis of the copper effect is discussed in terms of nucleation and growth of the precipitating species, together with the accompanying permeation of carbon through the alloy matrix. Carburisation rates measured for model Ni-Cu-Cr alloys are used to assess the effect of copper on carbon permeability in austenite.

2:35 PM
Diffusion Alloying of Thin Stainless Steel Foils by Simultaneous Addition of Al and Reactive Elements: *Daniela Pilone*[1]; Ferdinando Felli[1]; Umberto Bernabai[1]; [1]Sapienza Università di Roma

Automotive catalytic converter lifetime is linked to the amount of aluminum present in the base alloy that promotes the formation of a protective alumina layer. The process involving cold rolling and annealing allows to produce ferritic stainless steel containing more than 7 wt.% of Al. Unfortunately many α-Al_2O_3 scales tend to spall particularly during thermal cycling with consequent revelation of the bare alloy. Small additions to the ferritic stainless steel of oxygen reactive elements are known to improve the adhesion of a-Al_2O_3 scales. In this work reactive elements were added to the aluminum foil before roll bonding and their behavior during the diffusion bonding was evaluated. This novel process, that appears really promising, allows to overcome problems related to steel alloying with reactive elements and reduces the quantity of expensive reactive elements that needs to be added per square meter of catalyst carrying foil.

3:00 PM
The Performance of Al-Rich Oxidation Resistant Coatings for Fe-Base Alloys: *Bruce Pint*[1]; Ying Zhang[2]; Sebastien Dryepondt[3]; [1]Oak Ridge National Laboratory; [2]Tennessee Technological University; [3]University of California at Santa Barbara

Oxidation resistant Al-rich coatings are being investigated for applications such as ultra-supercritical fossil-fuel plants. The presence of water vapor accelerates the environmental degradation of chromia-forming ferritic and austenitic steels but has little effect on coatings able to form an Al-rich surface oxide. Long-term (=10,000h) diffusion and oxidation experiments are being conducted to determine the rate of Al loss from the coating by oxidation and interdiffusion. Based on these data, a coating lifetime model is being developed. Creep experiments were conducted to assess the effect of the coating on substrate mechanical properties.

3:25 PM Break

3:35 PM
Novel Surface Modification of Steel Using High-Density Infrared Heating: *Ryan Haase*[1]; Brian Gleeson[1]; [1]Iowa State University

High-density infrared heating is a surface heating technique capable of producing wear-resistant coatings over a considerably larger processing area than currently available techniques (e.g. lasers). The focus of this study was to establish plasma arc-lamp processing parameters for the production of carbon-enriched, wear-resistant coatings on a 1018 steel substrate. The phase transformations involved were found to be in accordance with what occur in a fast-cooled hypoeutectic Fe-C system. The resulting structures contained a significant amount of Fe_3C near the surface present as discrete plates and with the ledeburite (i.e., eutectic microconstituent). A large fraction of carbide and the fine scale of the structures resulted in high hardnesses, reaching 750 HV 0.1 and 980 HV 0.1 for the graphite-only and Fe-Mo-C surface-modified regions, respectively. The high hardness and carbide fraction resulted in a significant improvement in two-body sliding wear-resistance over a standard carburized-and-hardened microstructure.

4:00 PM
Interdiffusion Behavior of "Simple" Pt-Enriched γ+γ' Coatings on Ni-Based Superalloys: *Justin Stacy*[1]; Ying Zhang[1]; Bruce Pint[2]; Allen Haynes[2]; Brian Hazel[3]; Ben Nagaraj[3]; Lirong Lui[1]; [1]Tennessee Technological University; [2]Oak Ridge National Laboratory; [3]General Electric Aircraft Engines

"Simple" Pt-enriched γ+γ' coatings (16-19 at.% Al, ~18 at.% Pt) were synthesized on René 142 and René N5 Ni-based superalloys by electroplating ~7 μm Pt followed by an anneal treatment in vacuum at 1175°C. The interdiffusion experiments were carried out in the temperature range of 900-1100ºC with different periods of time for investigation of coating compositional and microstructural evolutions. After 1000h at 1000°C, the 30-µm coating diffused into the substrate to a depth of ~65 µm. At 1050°C, the coating became ~120 µm after 1000h. The changes in the compositional profiles after the diffusion experiments were determined by electron probe microanalysis, and a diffusion model was used to predict the Pt content at the coating surface.

4:25 PM
Cold Roll Bonding and Annealing Process to Produce an Intermetallic Layer on Ti Substrate: *Daniela Pilone*[1]; Ferdinando Felli[1]; [1]Sapienza Università di Roma

Titanium is characterized by poor resistance against oxidation at high temperature. Surface modification by formation of titanium aluminide coatings would be effective to improve oxidation resistance. In this study, a 100 µm aluminum foil was initially bonded with a 1 mm Ti sheet by means of cold roll bonding. The aluminum clad Ti foil was heat treated over the temperature range 600-1000°C in order to form an outer $TiAl_3$ layer protecting the substrate from high temperature oxidation. Al and Ti concentration profiles, as well as microhardness profiles, were determined in order to investigate the relationship between heat treatment conditions and formation of intermetallic compounds. The morphology and the structure of $TiAl_3$ layer formed at different temperatures were studied by means of SEM/EDS and X-ray diffraction.

4:50 PM Break

National Academies Corrosion Education Study Community Town Hall Meeting

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee
Program Organizer: Michael Moloney, National Academies

Tuesday PM March 11, 2008 — Room: 388 — Location: Ernest Morial Convention Center

Session Chair: Michael Moloney, National Academies

5:00 PM
The National Materials Advisory Board has convened a committee to assess the level and effectiveness of existing engineering curricula in corrosion science and technology, including corrosion prevention and control, and recommend actions that could enhance the corrosion-based skill and knowledge base of graduating and practicing engineers. The committee conducting this study, chaired by Dr. Wesley Harris from MIT, is soliciting community input in a number of fora. This town meeting will provide TMS members an opportunity to provide comments directly to committee members and NRC staff. More information on the study can be found at <www.nationalacademies.org/corrosioneducation> and comments can also be emailed to the committee at corrosion@nas.edu.

General Abstracts: Materials Processing and Manufacturing Division: Films, Coatings, and Surface Treatments

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: Global Innovations Committee, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Phase Transformations Committee, TMS: Powder Materials Committee, TMS: Process Technology and Modeling Committee, TMS: Shaping and Forming Committee, TMS: Solidification Committee, TMS: Surface Engineering Committee
Program Organizers: Ralph Napolitano, Iowa State University; Neville Moody, Sandia National Laboratories

Tuesday PM March 11, 2008 — Room: 282 — Location: Ernest Morial Convention Center

Session Chairs: Jun Qu, Oak Ridge National Laboratory; Arthur Heuer, Case Western Reserve University

2:00 PM
Advanced Polymer Coatings Produced by High Velocity Thermal Spray: *Bryony James*[1]; Margaret Hyland[1]; Krishal Patel[1]; [1]University of Auckland

Coatings of Poly-ether-ether-ketone (PEEK) have been deposited on stainless steel substrates using high velocity air fuel (HVAF) thermal spray. One essential parameter that dictates the performance of any coating is the adhesion of the coating to the substrate. In this case, of PEEK coatings on metal, adhesion occurs between an organic molecule and the surface oxide/hydroxide layer of the metals and as such can be altered by surface pretreatments of the metal. Surfaces of stainless steel were modified by degreasing, using proprietary etchants, and bead blasting with a variety of media. These surface pretreatments altered both chemistry and morphology of the metal as shown by analysis using X-Ray Photoelectron Spectroscopy (XPS) and Atomic Force Microscopy (AFM). Single splat studies of PEEK wetting on the substrates were used to determine optimum conditions to promote wetting and good splat morphology, and these conditions were subsequently used to produce well adhered PEEK coatings.

2:20 PM
Optical Property of Thermal Barrier Coating at High Temperature: *Geunsik Lim*[1]; Aravinda Kar[1]; [1]University of Central Florida

A radiative component in thermal barrier coatings and other ceramic oxides becomes significant at high temperature. Low radiative absorption and high scattering properties of thermal barrier coating play in reducing the temperature gradient. The dielectric constant, refractive index, and extinction coefficient are determined for improvements in turbine blade performance at high temperature. Optical properties of zirconia containing 7wt% Y_2O_3 will be discussed both for the conductive and the radiative components in the temperature range 1000-1873 K. The effect of various influencing parameters, i.e., radiation-conduction parameter, surface emissivity, single scattering albedo and optical thickness has been illustrated.

2:40 PM
Anodizing Steel for Corrosion Protection: T. Burleigh[1]; *Samuel Gabay*[1]; Taylor Dotson[1]; Keenan Dotson[1]; Trista Sloan[2]; Shannon Ferrell[1]; [1]New Mexico Institute of Mining and Technology; [2]Vibrant NDT

A novel process has been developed to protect steel from corrosion by anodization. Steel was anodized in a simple two-electrode cell in a concentrated NaOH or KOH solution. The steel grew a thick oxide surface film and was more resistant to corrosion than bare steel. Different ranges of anodizing voltage, time and temperature were found that produced varying thicknesses, adherency, and color. Analysis of the oxides revealed that they were primarily magnetite. A follow-up heat treatment increased the corrosion protection beyond either anodizing or heat-treating alone. Anodizing steel could help address the economic problem of corrosion of steel while providing an alternative to costly or toxic coatings. This paper describes what steps researchers have made in anodizing steel, what future improvements are needed, and how anodizing steel can benefit different industries.

3:00 PM
A Model of Thermo-Induced Processes during Particle Cooling in the Component-Coating System in Plasma Spraying Processes: Niu Liping[1]; Zhang Ting-an[1]; *Dou Zhihe*[1]; [1]Northeastern University

The temperature distributions on coating are analyzed in the component-coating system in plasma spraying processes. This process can be considered a thermo-mechanical treatment and with respect to other treatments, such as shot-peening in which the target is hit by metal or ceramic spheres, the biggest difference is the thermo effects. Temperature change is a source of stresses in target and the purpose of this analysis is to evaluate the stress by some numerical methods. A model of thermo-induced processes is obtained by calculation-experimentation during the particle cooling between elastic limit and temperature of the particle material.

3:20 PM
Forming a Wear-Resistant Nanocomposite Surface Using Friction Stir Processing: *Jun Qu*[1]; Hanbing Xu[1]; Zhili Feng[1]; D. Alan Frederick[1]; Brian C. Jolly[1]; Rick Battiste[1]; Peter Blau[1]; Stan David[1]; [1]Oak Ridge National Laboratory

Aluminum alloys would have much wider usage in bearing applications if their wear-resistance could be significantly improved. In this study, friction stir processing was used to stir and mix nano-sized $Al2O3$ particles into an aluminum surface to form a nanocomposite layer to improve the hardness, strength, and wear-resistance without sacrificing the bulk ductility and conductivity. Compared with a non-processed aluminum surface, the nanocomposite surface demonstrated increased hardness (by 3X) and yield strength (by 8X), and reduced friction coefficient (by 55%) and wear rate (by 200X). Major challenges encountered in process development include the low densities of nano-powders and the high wear of conventional FSP tools. Solutions to these challenges are proposed. Unlike most other surface engineering techniques, this process can form very thick layers, up to centimeters in thickness, with delamination avoided because of the inherent material continuity.

3:40 PM Break

3:50 PM
Fatigue of Surface-Treated Austenitic Fe-Base and Ni-Base Alloys: *Arthur Heuer*[1]; Gary Michal[1]; Frank Ernst[1]; Hal Kahn[1]; Yindong Ge[1]; [1]Case Western Reserve University

A low-temperature gas-phase carburization process has been developed that allows surface concentrations up to 15 at% carbon in austenitic stainless steels and Ni-base alloys without carbide formation, and with no decrease in ductility. Fatigue lifetimes under fully reversed loading ($R = -1$) are significantly enhanced, due to the extremely high strength and tough surface region, which contains residual compressive stresses of ~2 GPa generated by the carburization process. The increase in fatigue performance is associated with the absence of surface fatigue crack nucleation. Virtually all fatigue cracks nucleate in the interior of the sample. Several stainless steels have been investigated and will be discussed, including the 316L, 304, AL-6XN, and A286 grades, in addition to Ni-base IN718. The microstructural features responsible for crack initiation are described and the role of surface modification on fatigue life is discussed.

4:10 PM
Optimization of Gas Carburizing Process in Batch Furnaces with Endothermic Carburizing Atmosphere: *Olga Karabelchtchikova*[1]; Md. Maniruzzaman[1]; Richard Sisson[1]; [1]Worcester Polytechnic Institute

The paper addresses the current industrial issues with the gas carburizing process control and carburizing time/cost optimization. The optimization strategy is based on 1) understanding the effect of process parameters on the mass transfer coefficient and carbon diffusivity in austenite; 2) functional correlation of the observed variations in the process parameters on the kinetics of carburizing; and 3) developing a robust optimization technique to achieve the desired case depth with minimum cost and processing time. The index of performance for the process optimization involves both the surface carbon concentration and the case depth. While the first parameter depends on accurate control of the atmosphere and the carbon potential in the furnace, the case depth is primarily influenced by the temperature in the furnace and the duration of the carburizing process. Application of this optimization technique will result in significant energy reduction by shortening cycle time and thereby enhanced furnace capacity.

4:30 PM
Enhanced Corrosion Resistance of Austenitic Stainless Steels Due to Low-Temperature Carburization: *Arthur Heuer*[1]; Gary Michal[1]; Frank Ernst[1]; Hal Kahn[1]; Farrel Martin[2]; Ted Lemieux[3]; Theresa Newbauer[3]; Bob Bayles[3]; Paul Natishan[3]; [1]Case Western Reserve University; [2]SAIC/Naval Research Laboratory Operations; [3]U. S. Naval Research Laboratory

A low-temperature (~460°C) gas-phase carburization process has been developed that allows surface carbon concentration up to 15 at% in austenitic stainless steels without carbide formation. The carburization occurs under paraequilibrium conditions. The corrosion resistance of the steels after carburization is significantly enhanced – for example, pitting corrosion of 316L stainless steel is essentially suppressed, breakdown potential is increased by 850 mV, and crevice corrosion resistance can be greater than some Ni-base alloys such as Inconel 625. The carburized samples re-passivate ~150 mV below the breakdown potential, and the increased corrosion resistance is also evident in slow-strain-rate experiments performed in saltwater, particularly when the samples contain a crevice former. Possible mechanisms involved in the improved corrosion resistance are discussed, as are results for other carburized alloys.

4:50 PM
Experimental and Numerical Study of Flame Load Heat Transfer in an Experimental Furnace: *Ashwini Kumar*[1]; Raj Venuturumilli[1]; Laszlo Kiss[2]; Geza Walter[2]; [1]Fluent Inc.; [2]University of Quebec

Flame impingement on solid objects is a routinely encountered phenomenon in metallurgical operations. This was studied in an experimental furnace in order to obtain heat transfer data. In this paper, the same is simulated using Computational Fluid Dynamics (CFD) approach and the resulting temperature, velocity and chemical composition distributions were compared to the experimental data. This comparative analysis demonstrates the potential of mathematical modeling not only in complementing the experiments but also in providing a cost-effective framework for obtaining the data after proper validation with the experimental data. Examples with different burner and flame types used in the industry are given.

5:10 PM
Evaluation of Braze-Bonding Mechanism of Ternary Boride Based Cermet on to Ferrous Alloy Substrates for Improved Performance: *Barath Palanisamy*[1]; Anish Upadhyaya[1]; K. Anand[2]; [1]Indian Institute of Technology; [2]GE India Technology Centre

Materials possessing good wear resistance also possess high hardness and strength. Boride based cermets (C50, V30) are a new class of materials and aim to improve wear resistance of ferrous alloy substrates. The current work focuses on studying a unique surface modification process, Sinter-bonding. The process of sintering of the cermet, the substrate and the bonding between them occur simultaneously thereby rendering it economic. A range of ferrous alloy substrates were used with significant change in bonding mechanism owing to the change in composition of the base material. Bonding was carried out at 1250°C and it was observed that the atmosphere used affects the surface appearance and densification of the bonded cermet. The study was performed using SEM, EDS, XRD characterization tools and the mechanical properties were evaluated using micro-hardness, transverse rupture strength and tribological tests and it is observed that the material is competitive with other prevalent hard coatings.

General Abstracts: Structural Materials Division: Novel Issues in Materials Processing

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Alloy Phases Committee, TMS: Biomaterials Committee, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Composite Materials Committee, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee, TMS: Product Metallurgy and Applications Committee, TMS: Refractory Metals Committee, TMS: Superconducting and Magnetic Materials Committee, TMS: Titanium Committee
Program Organizer: Ellen Cerreta, Los Alamos National Laboratory

Tuesday PM — Room: 387
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Pietro Navarra, Hatch

2:00 PM
Bonding and Adhesion at the Fe/TiC Interface: Theoretical Strength and Misfit Dislocations: *Oleg Kontsevoi*[1]; Yuri Gornostyrev[2]; Arthur Freeman[1]; Gregory Olson[1]; [1]Northwestern University; [2]Institute of Metal Physics

The dispersed inclusions of TiC were shown to provide superior fracture toughness and increased strength for steels. To obtain a fundamental understanding of their effect, we calculated the work of adhesion for two kinds of Fe/TiC interfaces with the first-principles FLAPW method. For the coherent <100>{001}Fe//<110>{001}TiC interface, a theoretical strength of 3.89 J/m^2 was obtained. Within the framework of the Peierls-Nabarro model with ab-initio generalized stacking fault energetics, we estimated that the misfit dislocations, which may form due to 6.4% Fe-TiC lattice mismatch, would decrease the interface strength by 0.8 J/m^2. The second, <110>{110}Fe//<010>{100}TiC semicoherent interface contains one primary misfit dislocation per unit cell. Despite that, it shows a surprisingly high strength of 3.24 J/m^2, and the dislocation energy is estimated at 0.6 J/m^2. We discuss the electronic origins of strong interfacial bonding based on electronic structure and charge densities analyses. Supported by the ONR through Dynamic Microstructure Design Consortium.

2:20 PM
Direct Production of Super-Grade Materials in Nano-Particle Based Fuels: Liviu Popa-Simil[1]; *Claudiu Muntele*[2]; [1]LAVM LLC.; [2]AAMU

Nuclear transmutation reactions are based on the absorption of a smaller particle as neutron, proton, deuteron, alpha, etc. The resulting compound nucleus gets out of its initial lattice mainly by taking the recoil, also with help from its sudden change in chemical properties. The recoil implantation is used in correlation with thin and ultra thin materials mainly for producing radiopharmaceuticals and ultra-thin layer radioactive tracers. In nuclear reactors, the use of nano-particulate pellets could facilitate the recoil implantation for breeding, transmutation and partitioning purposes. Using enriched 238U or 232Th leads to 239Pu and 233U production while using other actinides as 240Pu, 241Am etc. leads to actinide burning. When such a lattice is immersed into a radiation resistant fluid (water, methanol, etc.), the recoiled product is transferred into the flowing fluid and removed from the hot area using a concentrator/purifier, preventing the occurrence of secondary transmutation reactions.

2:40 PM
Simulation Study on Dynamics of Femtosecond Laser Ablation of the CMSX-4 Ni-Based Superalloy: *Mousumi Das*[1]; Katsuyo Thornton[1]; [1]University of Michigan

We have studied the dynamics of femtosecond-pulsed-laser ablation of the CMSX-4 Ni-based superalloy by hydrodynamics simulations. Simulation results qualitatively describe time-resolved pump-probe shadowgraphic images obtained experimentally. In the experiment, a jet-like feature formed by ablated material propagated perpendicularly to the sample surface within an envelope of the air-shock that expanded in both perpendicular and parallel directions. The simulation was performed using two- and three-dimensional hydrodynamics code (FLASH), with initial condition provided by one-dimensional radiation-hydrodynamics (HYADES) simulation. The simulated density profile matches well with pump-probe shadowgraphs, providing insights into the origin of the contrast obtained by shadowgraphic imaging.

3:00 PM Break

3:30 PM
Rapid Manufacturing of Ti64 Components Using Net Shape Hot Isostatic Pressing: *Xinhua Wu*[1]; Junfa Mei[1]; Wayne Voice[2]; Martin Bache[3]; [1]University of Birmingham; [2]Rolls Royce; [3]University of Wales

Manufacturing large Ti64 components using Net Shape HIPping is introduced. Recent work on Net Shape HIPing has shown that the shape control, after one iteration is comparable with that found in investment casting. The mechanical properties are generally similar to those found in thermomechanically processed samples although the fatigue properties of Net Shape HIPed samples are slightly lower but the fracture toughness of the as-HIPed samples much higher than that of thermomechanically processed samples. The factors which control the properties of as-HIPed samples will be reviewed and methods to increase the cost-effectiveness of the process will be discussed.

3:50 PM
Synthesis of Bulk Mo-Ta Multilayers with Accumulative Roll Bonding: *Rainer Hebert*[1]; Girija Marathe[1]; [1]University of Connecticut

Metallic multilayers with a nanoscale layer thickness reveal extraordinary strength levels. While thin-film techniques are limited in synthesizing bulk samples for structural applications, accumulative rolling and folding can provide bulk samples with individual layer thicknesses of less than 100 nm. Rolling and folding experiments with multilayers of tempered Mo and Ta foils lead to a codeformation behavior and layer thicknesses of approximately 200 nm at a strain of -10. The codeformation behavior is rationalized based on microhardness measurements of the multilayers and on micro- and nanoindentation measurements of the individual layers, complemented by cross-sectional SEM and TEM analysis. The rule of mixture is examined for the hardness of the layers and the multilayer throughout the deformation range. The codeformation behavior is furthermore rationalized based on the ratio of the hardness of the Mo and Ta layers. The support of the NSF (CMMI-0700377) is gratefully acknowledged.

4:10 PM
Synthesis and Microstructure of Si_3N_4-TiN Nanocomposites: Mei Yang[1]; Mingli Lv[1]; *Hongmin Zhu*[1]; [1]Beijing University of Science and Technology

Si_3N_4-TiN nanocomposite was prepared by in situ coating in liquid ammonia using $TiCl_4$ and Si_3N_4 as starting materials. The reduction reaction of $TiCl_4$ by sodium in liquid ammonia was performed and resulted TiN nano-powders which nuclei and grew on the surface of Si_3N_4. The composite powder was consolidated though Spark Plasma Sintering (SPS) at 1600°C and dense compact (>98% of theoretical) with a mean grain size of 100~300 nanometers was obtained. The electronic conductivity value ($3.1\times10^2 O^{-1}\bullet cm^{-1}$) enough for electrical discharge machining (EDM) was reached by compositing 20vol% TiN to Si_3N_4. The relationship of microstructure and conductivity was discussed.

Hael Mughrabi Honorary Symposium: Plasticity, Failure and Fatigue in Structural Materials - from Macro to Nano: Cyclic Deformation and Fatigue of Metals I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: K. Jimmy Hsia, University of Illinois; Mathias Göken, Universitaet Erlangen-Nuernberg; Tresa Pollock, University of Michigan - Ann Arbor; Pedro Dolabella Portella, Federal Institute for Materials Research and Testing; Neville Moody, Sandia National Laboratories

Tuesday PM Room: 386
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Pedro Portella, Federal Institute for Materials Research and Testing (BAM); Petr Lukas, Czech Academy of Sciences

2:00 PM Keynote
Fatigue Behavior of Nano-Twinned High Purity Copper Films: Julia Weertman[1]; *Andrea Hodge*[2]; Troy Barbee[2]; Mark Seniw[1]; Anthony Escuadro[1]; [1]Northwestern University; [2]Lawrence Livermore National Laboratory

The response of high purity, nano-twinned ultra-fine grained Cu thin films to tension-tension fatigue is examined. The 5-9s purity copper samples studied are from sputter deposited free standing 175 micrometer thick films fabricated in a manner eliminating damage occurring during removal from the (100) single crystal silicon substrates or as a result of specimen preparation. Thus, the properties reported are characteristic of the nano/micro structure of these thick copper films. The films' fatigue endurance is determined and compared to coarse-grain as well as UFG Cu produced via cold-rolling. The films are also characterized by changes in surface features and internal microstructure that develop during cyclic loading. This work was partially performed under the auspices of the U.S. Department of Energy by the University of California, LLNL under Contract No. W-7405-ENG-48 amd at Northwestern Univeristy by US DOE grant DE-FG02-02ER46002.

2:30 PM Invited
Cyclic Deformation Behavior and Fatigue Life of Ultrafine-Grained Metals: *Heinz Werner Höppel*[1]; [1]University Erlangen-Nürnberg, Institute General Materials Properties WWI

Sufficient or improved fatigue properties are one of the key-features for a successful use of bulk nanostructured materials for potential technological applications. Consequently, a detailed understanding of the cyclic deformation behaviour and fatigue life plays a very significant role. Due to the significantly smaller grain size in UFG materials, the grain size becomes the dominating microstructural length scale and hence strongly affects the (dominating) cyclic deformation mechanisms. Hence, the cyclic mechanical properties of UFG materials cannot simply be explained by the well known Hall-Petch relationship. Other key features, like microstructural stability, strain rate dependence of the deformation behaviour, effect of the grain size and potential changes in the mechanisms of localization of plastic deformation compared to conventionally grained materials have to be considered. The addressed aspects on the deformation mechanisms as well as on the fatigue life will be reviewed in the talk.

2:50 PM Invited
On the Influence of Biaxiality on Fatigue Deformation and Microstructure: *Horst Biermann*[1]; Sebastian Henkel[1]; Juliane Fischer[1]; Tamas Ungar[2]; [1]Technical University Bergakademie Freiberg; [2]Eötvös University, Institut for General Physics

The LCF behaviour of the metastable austenitic stainless steel AISI 304 was investigated in monoaxial and biaxial strain-controlled fatigue tests. Constant amplitude, incremental step and load enhancement tests, respectively, where performed on monoaxial, hourglass-shaped specimens and on planar biaxial cruciform samples. In biaxial strain control, different strain paths, i.e. different strain axis ratios and phase angles were studied. The microstructure evolution depending on the different strain paths was investigated by scanning and transmission electron microscopy and x-ray line profile analysis. The percentage of deformation-induced martensitic phase was measured using a magneto-inductive ferrite-sensor and gives a correlation to the applied total strain amplitude. In addition, crack initiation and crack growth where studied using surface replicas. Based on the experimental results, the influence of the biaxiality is discussed. The von Mises equivalent strain can be applied for proportional load cases. The biaxial IST gives a better understanding of real multiaxial load cases.

3:10 PM
Biaxial Cyclic Stress-Strain Response of Ultrafine Grain Nickel: Ntirelang Batane[1]; *David Morrison*[1]; John Moosbrugger[1]; [1]Clarkson University

The biaxial cyclic stress-strain response of ultrafine grain (UFG) nickel produced by an electrodeposition process was studied by performing proportional and 90º out-of-phase nonproportional axial-torsional fatigue experiments at room temperature. For comparison purposes, identical experiments were also accomplished on conventional grain (CG) nickel. The grain sizes of the UFG and CG metals were on the order of 100 nm and 50 μm respectively. Axial-torsional tests were performed at constant effective plastic strain amplitudes of $1.0x10^{-4}$ and $1.0x10^{-3}$. In addition, multiple step tests were accomplished at selected effective plastic strain amplitudes between $1.0x10^{-4}$ and $1.0x10^{-3}$. The effects of grain size, loading path, and strain amplitude on cyclic stress-strain response will be discussed in terms of cyclic hardening behavior, cyclic stress-strain curves, and the evolution of dislocation structures.

3:25 PM
Damage Evolution during Low-Cycle Fatigue Testing of Ultra-Fine Grained Interstitial-Free Steel: *Thomas Niendorf*[1]; Hans Maier[1]; Ibrahim Karaman[2]; [1]University of Paderborn; [2]Texas A&M University

In the present study damage evolution in bcc UFG IF steel under cyclic loading is investigated. We focus on microstructural aspects such as fractions of high-angle and low-angle grain boundaries. ECAE processing following route A leads to high fractions of LAGBs and processing along routes C' and E leads to high fractions of HAGBs. We link the different kinds of boundaries with crack nucleation on the specimens' surfaces. The experiments used for this investigation included LCF tests at room temperature and EBSD measurements in a SEM. EBSD measurements conducted on the same surface area before and after fatigue testing showed that LAGBs are not stable under cyclic loading, as indicated by a rearrangement of boundaries in areas that are dominated by LAGBs and a high density of surface extrusions in these areas. The rearrangement of the LAGBs causes the observed softening and localized damage of route A UFG materials.

3:40 PM Break

3:50 PM Invited
Mechanisms of Cyclic Plastic Deformation in Ultrafine-Grained Copper Produced by Severe Plastic Deformation: *Petr Lukas*[1]; L. Kunz[1]; M. Svoboda[1]; [1]Czech Academy of Sciences

Fatigue properties and microstructure of UFG coppers of different purities produced by ECAP technique were studied in a broad region of stress amplitudes. While the fatigue strength of low purity UFG copper is by a factor of about 2 higher than that of conventional-grain-size copper in the broad region of fatigue lives from $6x10^3$ to $2x10^{10}$ cycles, the high purity UFG copper is superior to CG UFG copper only at high stress amplitudes. For both low purity and high purity UFG copper the grain structure is stable and undergoes only very marginal changes during cycling. Two mechanisms of cyclic plastic deformation are taken into consideration: (i) bulk mechanism consisting of irreversible movement of dislocations within the grains and (ii) surface mechanism operating in the surface layer consisting of GB sliding along the trace of the last ECAP pass. The later mechanism depends strongly on purity and on temperature.

4:10 PM Invited
Structural and Functional Fatigue of NiTi Shape Memory Alloys: *Gunther Eggeler*[1]; [1]Ruhr-University Bochum

Cyclic loading characterizes present and potential future applications of NiTi shape memory alloys which exploit mechanical (pseudo elasticity) or thermal shape memory (one and two way effect). It is associated with structural and functional fatigue, which both limit the service life of shape memory components. Structural fatigue describes microstructural damage accumulation that eventually leads to failure. Functional fatigue indicates that the intensity of shape memory effects like the working displacement in a one way effect (1WE) actuator or the dissipated energy in a loading-unloading cycle of a pseudo elastic (PE) damping application decreases during cycling. This is due to a gradual change in microstructure. In both cases it is important to know how fatigue cycling affects microstructure and shape memory properties. The present paper provides an overview and highlights some new microstructural results from shape memory fatigue research within the collaborative research center SFB 459 (shape memory technology).

4:30 PM Invited
Advances in the Analysis of Short Fatigue Crack Growth: *Arthur McEvily*[1]; [1]University of Connecticut

Although fatigue of metals has been a subject of importance since the early nineteenth century, interest in the details of the fatigue crack propagation aspect of fatigue failure did not develop until after the crashes of Comet aircraft in 1953. Prior to that time the approach to fatigue design had been largely based solely upon the work of Wöhler and the use of S/N curves. Since the 1950's advances in the fracture mechanics, computers, testing equipment and techniques, and in transmission and scanning electron microscopy have all contributed to a tremendous growth in the understanding of the fatigue crack growth process and of its role in the overall fatigue process. The object of this presentation will be to review some of the advances that have made to provide the basis for the quantitative analysis of fatigue crack propagation.

4:50 PM
Fatigue Behavior of Homogeneous-Microstructure and Mixed-Microstructure Steels: *Donato Firrao*[1]; Paolo Matteis[1]; Giovanni Mortarino[1]; Pasquale Russo Spena[1]; Maurizio Chiarbonello[1]; Giuseppe Silva[2]; Barbara Rivolta[2]; Riccardo Gerosa[2]; Andrea Ghidini[3]; [1]Politecnico di Torino; [2]Politecnico di Milano; [3]Lucchini Sidermeccanica

Molds for plastic automotive components such as bumpers and dashboards are usually machined from large pre-hardened steel blocks. Due to the dimensions, the heat treatment of these blocks produces mixed microstructures, continuously varying with the distance from the quenched surface. The fatigue behavior of these mixed microstructures is not well known, and peculiar results stemmed from previous tests. Such a theme has been examined through experimental tests on two different plastic mold steels, the traditionally used ISO 1.2738 steel and a quenched and tempered microalloyed steel recently proposed for the purpose. The threshold and fatigue crack growth behavior has been investigated. Moreover, in order to clarify the correlations between the microstructural features and the fatigue behavior, fatigue crack growth tests have been performed on re-heat-treated samples with homogenous microstructures (pearlite, or tempered martensite, or bainite), and have been compared with the results obtained while testing mixed microstructure samples.

5:05 PM
A Comparative Study on Fatigue Behavior of Shot Peened and Surface Severe Plastically Deformed Nickel Alloys: J. W. Tian[1]; J. C. Villegas[2]; K. Dai[3]; A. L. Ortiz[4]; R. Ren[5]; M. Manjarres[6]; *Leon Shaw*[6]; Peter K. Liaw[1]; D. L. Klarstrom[7]; [1]University of Tennessee; [2]Intel Corporation; [3]Quality Engineering and Software Technology; [4]Universidad de Extremadura; [5]Dalian Jiaotong University; [6]University of Connecticut; [7]Haynes International

A surface severe plastic deformation (S2PD) method has been applied to bulk specimens of a nickel-base C-2000 alloy. The fatigue behavior of the S2PD-processed samples has been characterized and compared with that of the shot-peened samples. The microstructure of the near-surface layer, macroscopic residual stresses, and cross-sectional hardness profiles have been quantified using a variety of instruments. Both shot peening (SP) and S2PD improve the fatigue resistance of the C-2000 alloy with S2PD showing better enhancement. The different improvements in the fatigue resistance resulting from SP and S2PD are related to differences in the nano-grains at the surface region, the residual compressive stresses, and the work-hardened surface layer. Finite element modeling is performed to provide quantitative description of these differences and insights into the mechanism responsible for the observed improvements. This study demonstrates that S2PD can provide better fatigue resistance than SP.

5:20 PM
Microtexturing Effects on the Fatigue Lifetime Variability of an Alpha + Beta Ti Alloy: *Christopher Szczepanski*[1]; Sushant Jha[2]; James Larsen[3]; J. Wayne Jones[1]; [1]University of Michigan; [2]Universal Technology Corporation; [3]US Air Force

Ultrasonic fatigue was used to characterize the fatigue behavior of Ti-6%Al-2%Sn-4%Zr-6%Mo in the very high cycle fatigue regime. Observed lifetimes ranged from 10^6 – 10^9 cycles. The initiation mechanism was crystallographic and the facets at the site of crack initiation resulted from fracture of the equiaxed alpha grains along the basal plane. Additionally, the initiation sites were located within microtextured regions where a majority of the alpha phase material was oriented for basal <a> or prismatic <a> type slip modes. OIM analysis indicated that these microtextured regions are generally larger than 1mm in size. The presence of these microtextured regions is believed to promote crack initiation and small crack growth. A computational model has been developed for probabilistic analysis of the size and spatial distributions of these microtextured regions. These results will be presented to highlight the relationship of microtexturing to the variability in fatigue crack initiation and associated lifetimes.

5:35 PM
Crystal Plasticity and Failure at Metal/Ceramic Interfaces: From Nano to Macro: *Siegfried Schmauder*[1]; A. Siddiq[1]; [1]University of Stuttgart/Institute for Material Testing, Materials Technology and Strength Theory

Deformation and fracture at metal/ceramic interfaces are related to local processes at the crack tip. Internal interfaces play a prominent role in metal/matrix composites between ceramic (such as Al_2O_3.) particles and a metallic matrix, e.g. Al. Despite their widespread use, a basic understanding of these interfaces is still underway. The deformation behaviour of niobium single crystals is simulated using crystal plasticity theory. Good agreement between experiment and simulation results was found. The second part provides results on effects of the different niobium single crystalline material orientations on crack initiation energies. Crack propagation analyses of niobium/alumina bicrystal interface fracture are performed using a cohesive modelling approach for three different orientations of single crystalline niobium. Parametric studies are presented. The results show that cohesive strength has a stronger effect on the macroscopic fracture energy as compared to the work of adhesion. In the last part, a correlation among the macroscopic fracture energy, cohesive strength, work of adhesion and the yield stress of niobiu single crystalline material will be derived.

Hume-Rothery Symposium - Nanoscale Phases: Session IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Sinn-wen Chen, National Tsing Hua University; David Cockayne, University of Oxford; Seiji Isoda, Kyoto University; Robert Nemanich, Arizona State University; K.-N. Tu, University of California, Los Angeles

Tuesday PM Room: 276
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Sinn-wen Chen, National Tsing Hua University; Seiji Isoda, Kyoto University

2:00 PM Invited
Grain Size Refinements in Al-Mg and Cu-Mg Alloys by Hydrogen Heat Treatment: *Atsunori Kamegawa*[1]; Masuo Okada[1]; [1]Tohoku University

We have investigated the hydrogen heat treatment of the so-called hydrogenation disproportionation desorption recombination (HDDR) process applied to alloys that contain some amounts of elements with a strong affinity for hydrogen, for example Mg, even though the major constituent of these alloys are mostly elements with a weak affinity for hydrogen. Upon the hydrogenation of the Al-Mg alloys, a disproportionation reaction occurred in the formation of MgH_2 embedded in the Al matrix phase. In the subsequent hydrogen-desorption of the alloys, MgH_2 was decomposed and Mg was resolved into the Al matrix phase, thereby resulting in the original solid solution alloys, and the Al grain size of about 10nm was observed. This indicates that HDDR phenomena occurred in the Al-Mg alloys. In addition, it was found that the Cu-Mg alloys also exhibit HDDR phenomena and that the grain size was refined.

2:25 PM Invited
Formation of Hexagonal Close Packing at Grain Boundaries in Gold: *Douglas Medlin*[1]; J. Hamilton[1]; O. Uche[1]; G. Lucadamo[1]; [1]Sandia National Laboratories

Grain boundaries in metals that possess low stacking fault energies can reconstruct into three-dimensional configurations by the emission of stacking faults. An important question is how the arrangement of these faults, and hence the phase of the interfacial layer, depends on the orientational parameters of the interface. Here, we present electron microscopic observations and modeling of two boundary misorientations in gold that both reconstruct to form a nanometer-scale layer of hexagonal-close-packed (HCP) material. In both cases, the HCP layer and its relationship to the grain misorientation is directly explained and predicted by the arrangement of Shockley partial dislocations at the interface. A comparison of the two boundary structures, one of which has partials paired as full lattice dislocations and other which does not, provides insight concerning the formation of other stacking arrangements, such as 9R, that have been observed at other grain misorientations in low SFE FCC metals.

2:50 PM Invited
Application of Microscopy to Nanoscale Organic Materials: *Seiji Isoda*[1]; Hiroki Kurata[1]; [1]Kyoto University

There is an awareness among scientists and technologists that organic materials offer considerable potential for application in many diverse areas. Recent advances in organic materials are found in electrically conducting materials, superconductors, non-linear optical materials, organic electroluminescence devises, organic solar cells, organic field-effect transistors (FETs) and molecular electronic devises. In these innovations, novel system performance could be recognized through nanostructuring as well as molecular property itself. As the dimensions of the structures approach the nanometer level, it becomes increasingly important to characterize materials properties at nanometer scale. Much of information on such nanoscale phases is being obtained by electron microscopy and this method is also an area of rapid progress. In the presentation, structural aspects of nanoscale phases in organic FETs will be discussed in relation with their properties, in which the phases are analyzed using various recent techniques in electron microscopy.

3:15 PM Invited
Bioactivity of Nanostructured and Plasma-Treated Biomaterials: *Paul Chu*[1]; [1]City University of Hong Kong

The biological properties of biomaterials are influenced by their micro- and nano-structures and can be improved by surface treatment. Nano-TiO_2 and ZrO_2 coatings have been produced using plasma spraying and plasma immersion ion implantation. The structure is evaluated by TEM, SEM, Raman and XRD and their bioactivity and biocompatibility are assessed using simulated body fluid soaking test and cell culturing. The nanostructured surfaces treated with the proper plasma processes exhibit excellent bioactivity and biocompatibility. The bioactivity of the films depends on a nanostructured surface composed of enough small particles, as reflected by the ability of the nano films to induce bone-like apatite formation on their surfaces after immersion in a simulated body fluid for a certain period of time. The plasma treatment is believed to provide the nanostructured surfaces have a high density of negative charges that influence the adsorption of molecules and ions from the simulated body fluids.

3:40 PM Break

4:00 PM Invited
An EDX-Based Oxidation Test for Evaluating Cu Out-Diffusion in Pre-Plated Cu-Alloy Leadframes: Lilin Liu[1]; Ran Fu[2]; Deming Liu[2]; *Tongyi Zhang*[1]; [1]Hong Kong University of Science and Technology; [2]Assembly Automation Ltd.

An EDX-based oxidation test is developed to study the Cu out-diffusion and oxidation in pre-plated Cu-alloy leadframes. A model is established to determine the diffusion coefficients of copper in nickel barrier platings and copper in an in-situ formed Sn-based intermetallic compound(IMC) nano-barrier-layer. The calculated diffusion coefficient is $(1.56\times10^{-10}cm^2/s)exp(-0.736eV/kT)$ for copper in the IMC nano-layer at the temperature 150°C~400°C, and $(1.59\times10^{-9}cm^2/s)exp(-0.602eV/kT)$ for copper in the nickel plating at the temperature 260°C~300°C, indicating that the in-situ formed Sn-based IMC nanolayer is a more effective diffusion barrier than the Ni layer. Discussion on the EDX-based oxidation test is given in details, thereby results in that the developed EDX-based oxidation test proves to be a simple and effective way for quantitative analysis of diffusion and oxidation at the nanometer scale.

4:25 PM Invited
Metal Phase Formation and Interaction in Electronic Packaging: *C Robert Kao*[1]; [1]National Taiwan University

An intriguing materials interaction problem arising from the metallurgical bonding in advanced electronic packages will be discussed. We report the strong cross-interaction between Sn-based solders and two common substrate materials, Cu and Ni. The interaction of Cu and Ni causes many problems. Nevertheless, this cross-interaction may also offer us opportunity to design a longer lasting UBM. The effect of Ni on the solders/Cu reactions is to be reviewed first. The effect of Cu on the solders/Ni reactions is then introduced. A slight variation in Cu concentration can change the reaction product from one phase to another. The knowledge gained from the Cu and Ni effects is applied to explain the recently discovered intermetallic massive spalling. It is pointed out that the massive spalling is caused by the shifting of the equilibrium phase as more and more Cu is extracted out of the solder by the growing intermetallic.

4:50 PM Invited
Size Effects in the Electronic Solder Joints: *Sinn-wen Chen*[1]; Yu-chih Huang[1]; Kuan-hsien Wu[1]; [1]National Tsing Hua University

With the emerging of flip chip technology and the trend of higher integration density in electronic products, sizes of electronic solder joints are shrinking. Solder joints in the electronic products are of various sizes, and more importantly there are solder joints which are very small. Melting temperature ranges of solders and interfacial reactions are the primary issues in the electronic soldering. Different interfacial reactions have been observed in the electronic solder joints of different sizes. Smaller solder volumes result in less supply of atoms, and the time frames of reaction sequences change significantly. With even smaller sizes of electronic solder joints in the future, the melting and interfacial reaction will be affected as well. Solders of nano-size are of lower melting points, and then the reflowing temperatures should be lower. Size effects in the electronic solder joints are certainly an important and challenging issue needs to be addressed.

5:15 PM Concluding Comments

Magnesium Technology 2008: Casting

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Mihriban Pekguleryuz, McGill University; Neale Neelameggham, US Magnesium LLC; Randy Beals, Chrysler LLC; Eric Nyberg, Pacific Northwest National Laboratory

Tuesday PM — Room: 292
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Henry Hu, University of Windsor

2:00 PM
Castability of Magnesium Alloys: Shezad Khan[1]; *Norbert Hort*[1]; Ingo Steinbach[2]; Siegfried Schmauder[3]; [1]GKSS Research Center; [2]Access e.V.; [3]Universität Stuttgart

Research and development of magnesium alloys depends largely on the metallurgist's understanding and ability to control the microstructure of the as-cast part. This work comprises the determination of experimental input parameters to run a successful and vast informative simulation using state of the art software. Various thermodynamic analyses have been used to study the thermo physical properties of binary magnesium-aluminum alloys, and the resultant microstructures have been simulated and then compared with the experimental output. The calculated heat distributions have been used to simulate the resulting microstructure using the phase-field method. This paper presents an overview of a range of ideas that have been undertaken to improve our understanding on the gravity die-casting behavior and solidification characteristics of Mg-Al alloys. It follows the solidification process of binary alloys Mg-Al, beginning with the nucleation and grain refinement. The simulated and the experimental results are compared and used for validation.

2:20 PM
Characterization of AM60B Magnesium Castings Using a Counter Gravity (Hitchiner) Lost Foam Casting Process: *Kenneth Currie*[1]; Qingyou Han[2]; [1]Tennessee Technological University; [2]Purdue University

Utilizing a Taguchi Design of Experiments (DOE), the results reported in this paper help to identify preferred parameters when using a Counter Gravity (Hitchiner) process for lost foam casting of magnesium alloys. Contrary to most other casting studies, a complex "widget" pattern was used as the test article with foam type, temperature, coating type, and pressure as the key parameters. Cast patterns were analyzed by volumetric and gravimetric standards as well as hardness and tensile strength. Statistical results from the DOE will be reported along with other general conclusions.

2:40 PM
Grain Refinement and Grain Morphology Evolution in Magnesium Castings Investigated by Phase-Field Simulations: *Janin Eiken*[1]; Ingo Steinbach[1]; Gerald Klaus[2]; A. Bührig-Polaczek[2]; [1]RWTH-Aachen; [2]RWTH Foundry Institute

The role of the composition of a quiternary Mg-Al-Zn-Ca-Mn alloy on the nucleation and growth morphology of equiaxed grains is investigated by phase-field simulations. A model for heterogeneous nucleation on particles with a prescribed size distribution is used to investigate the interplay between growth, solute redistribution and latent heat release in the solidifying mush. The morphology evolution of the individual grains is characterized by their volume to surface ratio and studied in its dependence on the alloy composition. The results are compared to experimental results from laboratory castings.

3:00 PM
Mould Thermal Analysis in Direct-Chill Casting of Magnesium Alloys: *Dimitry Sediako*[1]; William Macdonald[2]; Stephen Hibbins[2]; [1]National Research Council of Canada; [2]Timminco Metals

This study was performed to evaluate the major thermal parameters in the areas of primary and secondary cooling in magnesium DC casting process. The analysis included comprehensive experimental study of the major thermal parameters of the process, as well as 3D computer simulation of the casting process and its verification based on the experimental data. Three segments of primary cooling were clearly identified along the mould, length of these segments depends greatly on mould cooling arrangement and casting speed. The results included evaluation of thermal conditions of the process that might cause casting defects, such as surface folding and radial cracking. Neutron diffraction studies were performed to evaluate crystallographic texture of the as-cast metal depending on the cooling rates for the specimens taken from the centre and surface of an as-cast bloom. The outcome of the study has been discussed from the point of view of further "optimal" mould development.

3:20 PM Break

3:40 PM
Strengthening of Cast Mg Alloys by Friction Stir Processing: *Taiki Morishige*[1]; Masato Tsujikawa[1]; Sachio Oki[2]; Tomotake Hirata[3]; Kenji Higashi[1]; [1]Osaka Prefecture University; [2]Kinki University; [3]Technology Research Institute of Osaka Prefecture

Cast magnesium alloys were processed by friction stir processing (FSP) to acquire fine grain and high strength. FSP is a novel grain refinement method for light metal alloys. By using FSP, the microstructure was refined by dynamic recrystallization (DRX) and second phase particles could be finely dispersed. Moreover, for casting products, FSP is effective process to eliminate cast defects like micro porosities. Thick plate of commercial Mg alloy for die-casting AZ91D or Mg-Y-Zn alloys of two different chemical compositions have been prepared for FSP. Changes in the FSP-ed microstructures that affect mechanical properties were not only grain size refinement, but also the distributions of second phase particles. As-cast Mg-2at.%Y-2at.%Zn alloy, differs from Mg-2at.%Y-1at.%Zn, has icosahedral quasi-crystalline phase in grain boundaries. It has higher elevated temperature stability than inter-metallic compound of Al12Mg17. The distribution of fine second phase particles and mechanical properties were discussed.

4:00 PM
Further Improvements in HPDC Mg Alloys for Powertrain Applications: Mark Gibson[1]; Mark Easton[2]; Vinay Tyagi[1]; Morris Murray[3]; *Gordon Dunlop*[3]; [1]CSIRO; [2]Monash University; [3]Advanced Magnesium Technologies

Rare earth containing magnesium alloys are the most promising alloys for powertrain applications. AM-HP2 has the best creep properties of currently proposed high pressure die casting alloys for elevated temperature applications. One of the most important issues for all creep resistant Mg alloys has been to obtain excellent creep properties and good castability simultaneously. A new die for assessing multiple aspects of castability has been designed. This die allows assessment of the many aspects of castability that are required for good casting quality including die filling, susceptibility to hot tearing and defects at flow fronts. Using this die, alloys with improved die castability and which retain the already excellent creep properties of AM-HP2, have been identified.

4:20 PM
The Effect of Different Direct-Chill Casting Process on Microstructure, Macrosegregation and Mechanical Properties of ϕ200mm AZ31 Billets Obtained: *Zhiqiang Zhang*[1]; [1]Key Laboratory of Electromagnetic Processing of Materials, Ministry of Education

AZ31 alloy billets of ϕ200mm diameter have been produced by three different process of conventional direct chill (DC) casting, low frequency electromagnetic casting (LFEC) and low frequency electromagnetic vibration casting (LFEVC), respectively. The effect of LFEC and LFEVC on the microstructures, macrosegregation and mechanical properties in AZ31 billets was investigated. In conventional DC casting, the AZ31 billets exhibited coarse grains (about 370μm) and severe segregation of Al and Zn. In the presence of a solo low frequency alternating magnetic field(LFEC) or a low frequency electromagnetic vibration field(LFEVC) applied during DC casting of F200mm AZ31 billets, the grains of the AZ31 billets was effectively refined (about 210μm) and the macrosegregation of Al and Zn in the billets was greatly decreased. Furthermore, there was a significant increase in tensile strength, facture elongation and hardness of the as-cast AZ31 billets obtained by LFEC and LFEVC relative to that cast by conventional DC casting.

4:40 PM
Microstructure Refinement of AZ31B Alloys Processed with an Electromagnetic Vibration Technique: *Kenji Miwa*[1]; Mingjun Li[1]; Takuya Tamura[1]; [1]National Institute of Advanced Industrial Science and Technology (AIST)

Electromagnetic vibrations, which are generated by simultaneous imposition of a static magnetic field and an alternating electric field, are considered to lead to the formation and collapse of cavities even in a molten metal, and then achieve the refinement of solidified structure. In order to refine the microstructure of AZ31B magnesium alloy, the electromagnetic vibration (EMV) process has been applied. Fine equiaxed grains can be obtained when AZ31B alloys are solidified at a frequency interval of ca. 500 up to 2000 Hz. The difference in electrical resistivity between the solid and liquid in mushy zone originates uncoupled motion, which breaks dendrite into fragments, destroys the crystallographic orientations, and yields deformation twins. Effective refinement occurs when the leading solid is driven to move out the operation scope of solute redistribution area where the solute cannot be piled up to generate a constitutionally undercooled region that always destabilizes.

5:00 PM
Carbonate Inoculation and Grain Refinement of Magnesium Alloys: *Young Min Kim*[1]; Chang Dong Yim[1]; Sung Soo Park[1]; Bong Sun You[1]; [1]Korea Institute of Materials Science

While low cost grain refiners have been developed for castings of aluminum alloys, there are no commercial inoculants for magnesium alloys, especially Mg-Al alloys. Although hexachloroethane is a very effective inoculant for grain refinement of Mg-Al alloys, the emission of toxic gases gives rise to detrimental problems in environment and workshop, resulting in the restriction of its use. Therefore many alternative grain refiners being more effective for

Mg-Al alloys than hexachloroethane without any environmental problem have been investigated. In this study, the effect of carbonate inoculation on grain refinement of Mg-Al base alloys was investigated. The results show that $MgCO_3$ and $MnCO_3$ have excellent grain refining efficiency in Mg-Al base alloys, of which average grain size is less than 70µm. This is because inoculation of carbonates causes the formation of heterogeneous nucleant in a melt as well as the agitation of melt by the generation of carbon dioxide gas.

5:20 PM
Hot Tearing in Castings of Mg-Al-Sr Alloys: Sindu Kou[1]; *Guoping Cao*[1]; [1]University of Wisconsin

Ternary Mg-Al-Sr alloys are the base of a few new creep-resistant, light-weight Mg alloys for automobiles. Hot tearing in Mg-Al-Sr alloys was studied, including Mg-(4,6,8)Al-1.5Sr and Mg-(4,6,8)Al-3Sr, by constrained rod casting (CRC) in a steel mold. The hot tearing susceptibility was determined based on the widths and locations of the cracks in the rods. With Mg-(4,6,8)Al-1.5Sr alloys, the hot tearing susceptibility decreased significantly with increasing Al content. With Mg-(4,6,8)Al-3Sr alloys, the trend was similar but not as significant. At the same Al content, the hot tearing susceptibility decreased significantly with increasing Sr content. The onset of hot tearing was detected by instrumented CRC, in which the rising tension in the bottom rod was measured with a load cell and the temperature near the crack site was measured with a thermocouple. The results of instrumented CRC were correlated with those of CRC.

Magnesium Technology 2008: Wrought Alloys III

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Mihriban Pekguleryuz, McGill University; Neale Neelameggham, US Magnesium LLC; Randy Beals, Chrysler LLC; Eric Nyberg, Pacific Northwest National Laboratory

Tuesday PM — Room: 291
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Alan Luo, General Motors Corporation; Tyrone Jones, US Army Research Laboratory; Luke Mackenzie, Novelis

2:00 PM
Anisotropy of the Damping Capacity of Rolled Mg-0.4Zn-0.6Zr (ZK01) Alloy: *Liming Peng*[1]; Lin Du[1]; Li Jin[1]; Qudong Wang[2]; Wenjiang Ding[1]; [1]Shanghai Jiaotong University; [2]Shanghai Jiao Tong University

Anisotropy of the damping capacity of Mg-0.4Zn-0.6Zr (ZK01) and the effect of the texture on it were investigated in this study. The textures were observed by X-ray diffraction (XRD), the results show that there is typical rolling texture ND//[0001] fiber texture in this Mg-0.4Zn-0.6Zr (ZK01) alloy sheet and the damping capacity are different in these specimens which have the included angles relationship, 0°,45° and 90° with the rolling direction (ED). The higher damping capacity can be ascribe to the higher dislocation slip possibility in grains.

2:20 PM
Effect of Twinning on the Mechanical Behavior of Mg-Zn-Y Alloys: *Ju Youn Lee*[1]; Hyun Kyu Lim[1]; Do Hyung Kim[1]; Won Tae Kim[2]; Do Hyang Kim[1]; [1]Yonsei University; [2]Department of Nanoscience, Chongju University

The effect of twinning on the mechanical behavior of Mg-Zn-Y alloys was investigated by controlling process parameters and alloy chemistry. To study the effect of process parameters, Mg-6Zn-1.2Y sheets were prepared by changing rolling speeds. DRXed-grains are developed under a lower rolling speed while twinned-grains are obtained under a higher rolling speed. The uniform elongation increases from 5.3% to 7.3% with increasing rolling speed from 32 to 64 mm/s. To study the effect of alloy chemistry, Mg-xZn-0.6Y (x= 3~7) sheets consisted of a–Mg and quasicrystalline particles were prepared. Strength and elongation-to-failure increase with increasing Zn despite of similar grain size and volume fraction of particles. Interrupted tensile tests showed that larger fraction of twinned-region developed in Mg-7Zn-0.6Y than in Mg-3Zn-0.6Y. EBSD analysis shows that extension twins are developed. It is suggested that twinning leads to reorientation of basal-plane to more favorable orientations, improving the elongation in Mg-Zn-Y alloys.

2:40 PM
Effect of {10-11} Contraction and {10-11}-{10-12} Double Twins on the Subsequent Deformation Behavior of Two Mg Alloys: *Lan Jiang*[1]; John Jonas[1]; Raj Mishra[2]; [1]McGill University; [2]General Motors

Mg alloy tubes are frequently bent prior to hydroforming. As a result, such metals are subjected to reversals of strain path. Therefore, it is useful to know the effects of changing strain path as well as of twinning on the stress-strain response after a change in strain path. In this study, experiments were designed and conducted to introduce contraction and double twins during prestraining; these prestrained samples were subsequently compressed at a strain rate of 0.1/s and at ambient temperature as well as at 200°C. It shows that the initial compressive deformation is slip-dominated, which is then followed by extension twin-dominated deformation. In addition, an annealing treatment (heat at 300°C for one hour) was performed on some prestrained tensile samples. The heat treatment of heavily extended material containing contraction and double twins displays an effective method of refining the grain size and modifying the texture.

3:00 PM
Effect of Pre-Ageing Treatment on Hot Compression Behavior of AZ31 Magnesium Alloy: *Lihong Shang*[1]; Stephen Yue[1]; Elhachmi Essadiqi[2]; Javid Amjad[2]; Verma Ravi[3]; [1]McGill University; [2]CANMET Materials Technology Laboratory; [3]General Motors Research and Development Center

The effect of second phase precipitates on the hot working behavior of cast AZ31 magnesium alloy was investigated. A series of cast AZ31 samples were solution treated at 450°C, quenched, and then aged at 150-350°C for one hour to produce precipitates with different amounts and size distributions. The aged samples were heated to the test temperature of 350°C at a heating rate of 1°C s-1, and compression tested at a true strain rate of 0.01 s-1, to a true strain of 0.6. The results show that the lower the aging temperature, the higher is the compression flow stress. This is explained in terms of the second phase characteristics observed in the aged samples.

3:20 PM
Influence of Cerium on Texture and Ductility of Magnesium Alloy Extrusions: *Raja Mishra*[1]; Anil Gupta[2]; P. Rao[3]; Anil Sachdev[1]; Arun Kumar[4]; Alan Luo[1]; Li Jin[5]; [1]Materials and Processes Laboratory, General Motors Research and Development; [2]National Physical Laboratory; [3]International Advanced Research Centre for Powder Metallurgy and Advanced Materials; [4]General Motors India Science Laboratory; [5]Shanghai Jiaotong University

Ce-containing Mg alloys were extruded as solid rounds at 400°C in a 500 ton vertical extrusion press at 10 mm/sec. Careful EBSD analysis of the recrystallized extrudate was conducted. This paper will discuss the tensile properties and texture evolution of these alloys following extrusion and recrystallization, and also a detailed microstructural evaluation of the polished cross-section close to the fracture surface as well as the fracture morphology.

3:40 PM
Mechanical Properties and Microstructures of Extruded Mg-2.4at%Zn Alloys Containing Ag and Ca: *Chamini Mendis*[1]; Keiichiro Oh-ishi[1]; Yoshiaki Kawamura[2]; Tomoyuki Honma[2]; Shigeharu Kamado[2]; Kazuhiro Hono[1]; [1]National Institute for Materials Science; [2]Nagaoka University of Technology

Mg-Zn based ZK60 (Mg-2.4at%Zn-0.16at%Zr) are used widely as wrought magnesium alloys due to the good combination of strength and ductility. Recently we reported that addition of Ag and Ca in trace quantities (0.1at% each) to Mg-2.4at%Zn alloys has shown significant increments to the precipitation hardening response. Examination of the mechanical properties of extruded Mg-2.4at%Zn(-0.16at%Zr) with trace additives of Ag and Ca has shown to significantly improve tensile properties. Tensile yield stress of 290MPa, UTS of 350MPa and an elongation of 17.% has been realized for as extruded Mg-2.4at%Zn-0.1at%Ag-0.1at%Ca-0.16at%Zr alloy. Following extrusion these alloys may be precipitation hardened by ageing at 160°C. The mechanical properties of these alloys may be further improved with heat treatments subsequent to the extrusion processing. The microstructures observed following processing have been characterized with the use of optical and transmission electron microscopy and show that there is significant refinement of both grain size and precipitates.

4:00 PM
Forming Characteristics of Mg Sheet with Variation of Temperature: *Yong Nam Kwon*[1]; Y-S Lee[1]; J-H Lee[1]; [1]Korea Institute of Materials Science

Mg alloys are the lightest alloys among the industrially applicable engineering alloys. Wrought alloys are superior in terms of mechanical properties and reliability to casting. A lot of efforts has given to establish a optimum forming condition for the industry uses, since wrought Mg alloy has a limited applications due to the poor formability. Most wrought Mg parts are fabricated under the elevated temperature condition of around 250°C where more slip systems are activated with temperature rise. Superplastic forming is another way to get over the low formability of Mg alloys. In the present study, formability of AZ31 sheet was investigated in terms of forming temperature with differing forming speed. For this purpose, both experimental and computational studies were carried out.

4:20 PM
The Influence of Calcium and Rare Earth Metals on the Microstructure and Mechanical Properties of Mg-3Al-1Zn during Extrusion: Torsten Laser[1]; Christian Hartig[1]; *Timo Ebeling*[1]; Marcus Nürnberg[2]; Dietmar Letzig[2]; Rüdiger Bormann[2]; [1]Technical University Hamburg-Harburg; [2]GKSS Research Centre

The role of calcium and rare earth rich intermetallic particles in magnesium alloys during extrusion is studied by mechanical tests and texture measurements. After extrusion a reduction of the final grain size mainly caused by the influence of these intermetallic particles is observed, which becomes more distinct for higher alloying contents and low extrusion temperature. The extruded rods exhibit typical extrusion fibre-textures (c-axes in radial direction) with a high tension-compression asymmetry of the yield stress in the extrusion direction. Differences in mechanical properties are related to changes of the texture development. The influence of the intermetallic particles on the texture evolution are discussed in view of the activation of non basal slip modes at higher temperatures and mechanisms of dynamic recrystallization.

4:40 PM
Microstructures and Mechanical Properties of Magnesium Composite Alloys Dispersed with Carbon Nanotube via Powder Metallurgy Process: *Katsuyoshi Kondoh*[1]; Hiroyuki Fukuda[1]; HIsashi Imai[1]; Bunshi Fugetsu[2]; [1]Osaka University; [2]Hokkaido University

AZ31B powder coated by carbon nano-tubes (CNT) has been prepared by using surfactant (surface active agent), having both hydrophobic and hydrophilic groups, when employing CNT with 15nm and 75nm diameter. It was compacted at room temperature by applying 600MPa pressure, and consolidated by hot extrusion. Pre-heating temperature of the green compact before extrusion was higher than a sublimation point of the binder used in the surfactant which is determined by differential thermal analysis (DTA). Optical microscope and FE-SEM observation and EDS analysis were carried out on the above composite alloys. The dependence of their mechanical properties on CNT content was also discussed.

Materials for Infrastructure: Building Bridges in the Global Community: Session I

Sponsored by: The Minerals, Metals and Materials Society, Indian Institute of Metals, Chinese Society for Metals
Program Organizer: Brajendra Mishra, Colorado School of Mines

Tuesday PM
March 11, 2008
Room: 272
Location: Ernest Morial Convention Center

Session Chair: Brajendra Mishra, Colorado School of Mines

2:00 PM Introductory Comments by Brajendra Mishra

2:10 PM Keynote
The Geology of the Katrina Disaster in New Orleans: *Stephen Nelson*[1]; [1]Tulane University

A combination of historical and geological factors combined with inadequate design of levees and floodwalls resulted in a series of levee overtoppings and catastrophic levee failures in the New Orleans area during the passage of Hurricane Katrina on August 29, 2005. Early in the morning of August 29, levees along the Mississippi River Gulf Outlet and Intracoastal Waterway were overtopped by the storm surge generated by Katrina resulting in the flooding of eastern New Orleans and St. Bernard Parish. Later in the morning storm surge entering New Orleans' Inner Harbor Navigational Canal overtopped levees and floodwalls on both sides of the canal, eventually resulting in the catastrophic failure of the floodwalls and the destruction of the Lower 9th Ward. Three drainage canals in New Orleans, originally constructed in the mid-1800s to drain rainwater from the city into Lake Ponchartrain to north, then became subject to storm surge entering from the Lake. The excess pressure of the surge combined with the weak geological material underlying the levees and floodwalls resulted in two levee breaches on the London Avenue Canal and one on the 17th St. Canal by mid-morning on August 29. These breaches resulted in flooding of about 80% of the city of New Orleans. The failures of levees on these drainage canals did not result from overtopping the floodwall system, but apparently from weaknesses in the design of the system that failed to adequately account for the underlying geologic conditions.

2:40 PM Invited
The Application of Steel in a New Residential Structural System: *Zigang Li*[1]; [1]Baoshan Iron and Steel Company, Ltd.

Baosteel, as a member company of Living Steel Project organized by IISI, aims at extending the use of steel in house buildings, and exerts deep research on the application of steel materials and components in new type residential structural systems. A new structural system with roll formed shape steel columns and high frequency welding H-section steel beams was studied through finite element analysis method and was testified with experiments.

3:10 PM Break

3:30 PM Invited
Emerging Role of Non-Ferrous Metals in Public Utilities and Infrastructural Projects: L. Pugazhenthy[1]; *Katragadda Sarveswara Rao*; [1]India Lead Zinc Development Association

After the economic liberalization in 1991, India's focus shifted to the infrastructural sector that covers national highways, power, telecom, aviation, ports, etc. In view of the consistent and impressive GDP growth rates, substantial funds have been spent on these sectors and the results are yielding. For instance, the rate of telecom subscriber growth – 8 million a month – is almost twice the population of Finland. The Planning Commission of India proposes to invest about US $500 billion during the Eleventh Five Year Plan period (2007–2012) in the infrastructural segment. In one sector alone ie., power, it is proposed to add 80,000 MW of electric power during the same period. Similar would be the growth in the other infrastructural sectors. The thrust on infrastructural projects in the country has given a great boost in demand for steel, non ferrous metals etc. Base metals like aluminium, copper, zinc and lead are used in a wide variety of applications in power generation and transmission, transportation, building, construction, irrigation, telecom, agriculture, public health etc. As a result of the growth trends in these areas, the non ferrous metals have been witnessing double digit growth rates in their usage every year. Zinc has been growing at a rate of 15% per year. After China, it is India that has been galloping in the demand for non ferrous metals. Accordingly metal producers, both primary and secondary, have been expanding their capacities. Each of the two industrial groups involved in Aluminium, Zinc, Copper has been planning to set up 1.0 million tonne smelters for these three metals. In order to achieve the targeted production levels, the non ferrous metal manufacturers are also acquiring mines in Zambia, Australia etc., so that there is an uninterrupted flow of input materials. The metal producers are also investing huge sums of money in survey, exploration and mining so as to find new resource bases. The Indian government has also been fine-tuning appropriate policies for domestic investment, foreign direct investment, taxation, environment protection so as to accelerate the pace of activity in the metals sector.

4:00 PM Invited
Opportunities for Aluminum Alloys in Restoration of Aging Bridge Decks: *Subodh Das*[1]; J. Randolph Kissell[2]; John Kaufman[1]; [1]Secat Inc; [2]TGB Partnership

Thousands of steel and concrete highway bridges in North America are deteriorating. Many have been load-limited due to this deterioration and because traffic and loads now exceed original designs. Yet replacing these spans is often slow, costly, and done at great inconvenience to drivers, including those of emergency vehicles. Aluminum alloys, with their 75 year record of successful use in bridges, offer the opportunity to address this challenge. Aluminum bridge decks have a high strength-to-weight ratio and can be prefabricated offsite and installed quickly with minimum disruption to traffic. In addition, because of their excellent corrosion resistance, aluminum decks do not have to be painted and require very little maintenance. This paper addresses how aluminum bridge decks might be developed and deployed.

4:30 PM Invited
Research and Development of a Ultra-Fine Grained Steel Rebar with Low Cost and High Performance: *Zhongmin Yang*[1]; [1]Division of Structural Materials, Central Iron and Steel Research Institute

In the past decade, "Ultra-fine grain" (UFG) has been paid more and more attention to seek for low cost high strength steels. People in Chinese steel academy and industry have devoted many of their efforts on the investigation to the ways to refine grains in steels and the mechanisms. Also, to make great efforts on develop to the industrial and application technology of ultra-fine grain steel products. Grain refinement has been as one of the effective ways of microstructure control to improve the performances of steel products. Compare with original steels, due to the increased strength, improved toughness and ductility, the ultra-fine steels has been gradually taken broad application. We have developed the DIFT (Deformation Induced Ferrite Transformation) rolling technology to refine ferrite grains in low carbon steel rebar. Plain low carbon steel rebars of 400MPa grade, with ferrite grains in the range of 4 – 8μm, have been developed. According to test results, ultra-fine steel rebars can be as the candidate to replace microalloyed steel rebars of 400 MPa grade. Therefore, it can be saving a great deal of microalloying elements (V, Nb, Ti. etc). In resent years, DIFT Technology has been applied in wide range of long-product lines with very economic production costs and the annual product of ultra-fine steel rebars was achieved 100 million ton.

Materials in Clean Power Systems III: Fuel Cells, Hydrogen-, and Clean Coal-Based Technologies: Metallic Interconnects in SOFCs: Oxidation, Protection Coatings

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee
Program Organizers: Zhenguo "Gary" Yang, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; K. Scott Weil, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University; Ayyakkannu Manivannan, National Energy Technology Laboratory

Tuesday PM	Room: 392
March 11, 2008	Location: Ernest Morial Convention Center

Session Chairs: Xingbo Liu, West Virginia University; Paul Gannon, Montana State University

2:00 PM Invited
The Effect of Substrate Impurities on Coated Metallic Interconnect Materials for Solid Oxide Fuel Cell Interconnects: *Christopher Johnson*[1]; Nina Orlovskaya[2]; Anthony Coratolo[3]; Caleb Cross[1]; Xingbo Liu[4]; Junwei Wu[4]; Randall Gemmen[1]; [1]National Energy Technology Laboratory; [2]University of Central Florida; [3]Drexel University; [4]West Virginia University

Substrate impurities can affect the performance of coated metallic interconnect materials used in solid oxide fuel cells (SOFC) applications. Impurities, such as silicon and aluminum can lead to increased area specific resistance (ASR), and a number of typical low level additives used in metallurgy to enhance the properties of the bulk materials for other applications can interact in detrimental ways when these substrates are coated to prevent chromium migration in SOFC. Coatings discussed in this work are applied by magnetron sputtering and by DC and pulsed plating techniques. Substrates are ferritic alloys with varying amounts of silicon, aluminum and other additives, and having CTEs that are relatively similar to that for typical SOFC (Crofer APU 22, and SS430) Samples are characterized by ASR measurements, X-ray Diffraction (XRD), scanning electron microscopy (SEM), and by energy dispersive X-ray analysis (EDX) before and after long term annealing under simulated cathode conditions.

2:30 PM Invited
Large Area Filtered Arc Deposited Coatings for SOFC Metallic Interconnects: *Paul Gannon*[1]; Vladimir Gorokhovsky[2]; Max Deibert[1]; Preston White[1]; Richard Smith[1]; Hui Chen[1]; W. Priyantha[1]; [1]Montana State University; [2]Arcomac Surface Engineering

Thin film (<5μm) MCrAlY oxide coatings were deposited on ferritic steels using large area filtered arc deposition (LAFAD) technology (M = Ti, Co and/or Mn). Coated and uncoated specimens were characterized as-received, and as a function of solid oxide fuel cell-interconnect (SOFC-IC) relevant exposures. Significant improvement of simulated SOFC-IC performance was observed in coated vs. uncoated specimens, including lower and more stable area specific resistance (ASR) and reduced Cr volatility. TiCrAlY oxide coatings exhibited the best high-temperature stability, while coatings containing Co and/or Mn exhibited the lowest ASR. Coating thickness and substrate steel surface finish were also observed to influence high-temperature corrosion behavior. Coating deposition processes, characteristics and SOFC-IC applicability are presented and discussed.

3:00 PM
Ceramic Coatings for Metallic SOFC Interconnects: *Jeffrey Fergus*[1]; [1]Auburn University

The two approaches for SOFC interconnect materials – ceramics and metals – each have advantages, but neither is ideal. Metallic chromia-forming alloys are most commonly used, but the formation of chromium-containing vapor species can lead to poisoning of the cathode after long operation times. The resistance to this degradation can be improved through a combination of the two approaches by coating the metallic interconnect with a ceramic. The coating serves three functions – it must i) limit the inward diffusion of oxygen to reduce the growth of the scale, ii) limit the outward diffusion of chromium to reduce chromium poisoning, and iii) transport electrons from the interconnect to the cathode. Performing all these functions, while simultaneously satisfying other requirements, such as chemical stability/compatibility and thermal expansion match, is a significant challenge. In this paper, the approaches used in the development of ceramic coatings for metallic SOFC interconnects will be reviewed.

3:25 PM
Novel Coatings for Stainless Steel Interconnects Utilized in SOFCs: *Douglas Ivey*[1]; Weifeng Wei[1]; Nima Shaigan[1]; Weixing Chen[1]; Anthony Wood[2]; Sofiane Benhaddad[2]; [1]University of Alberta; [2]Versa Power Systems

Intermediate temperature SOFCs operate at temperatures <800°C, permitting the use of ferritic stainless steels as interconects. Long term SOFC operation is limited, however, by the resistivity and volatility of the protective chromia scale that forms on the steel surface. To overcome these limitations, two novel methods of applying protective coatings are studied in this work. In the first approach, Mn-Co spinel oxides are anodically electrodeposited from aqueous solutions containing Mn and Co divalent ions. By controlling the solution composition and deposition parameters, dense and crack free deposits are obtained. Oxidation tests show significantly improved oxidation resistance compared with uncoated steels. In the second approach, composite coatings consisting of dispersed conductive lanthanum chromite particles in a Ni matrix are electrodeposited onto ferritic stainless steel substrates. Upon oxidation in air, a bilayer oxide scale forms, with NiO covering a protective chromia embedded with conductive chromite particles.

3:45 PM Break

4:00 PM Invited
Micro/Nanomechanical Characterization of Coatings and Its Applications to Fuel Cell Systems: *Xiaodong Li*[1]; [1]University of South Carolina

Reliability study is the key for practical applications and commercialization of today's advanced full cells. Coatings of different materials are structural and functional components in full cell systems. The mechanical failure of these coatings may cause malfunction or even break down of the full cells. Precise characterization of the mechanical properties of the coatings is required for proper design and structural integration of full cell systems. In this presentation, recent progress in micro/nanoscale mechanical characterization of coatings is briefly reviewed. Emphasis is placed on nanoindentation techniques and how they are used to study hardness, elastic modulus, scratch resistance, fracture toughness, fatigue, friction and wear properties of different coatings, especially those designed for use in full cell systems. The presentation ends with a discussion on directions for future research.

4:30 PM Invited
Thermal Stability and Oxidation Resistance of Protective Metal Oxide Coatings on Steel SOFC Interconnects: *Richard Smith*[1]; H. Chen[1]; W. Priyantha[1]; M. Kopczyk[1]; J. Lucas[1]; C. Key[1]; M. Finsterbusch[2]; P. Gannon[1]; P. White[1]; M. Deibert[1]; V. Gorokhovsky[3]; V. Shutthanandan[4]; P. Nachimuthu[4]; [1]Montana State University; [2]Technical University Ilmenau; [3]Arcomac Surface Engineering; [4]Pacific Northwest National Laboratory

The requirements of low cost and high-temperature corrosion resistance for interconnects in SOFC stacks have directed attention to the use of steel plates with electron conducting, oxidation resistant coatings. We studied coatings from the MCrAlYO family (where M = Co, Mn, or Ti) deposited on 430 steel coupons using filtered arc deposition technology at Arcomac Surface Engineering. Ion beam analysis, x-ray diffraction, and electron microscopy were used to characterize the thermal stability, composition, and structure of the coatings before and after high temperature annealing. Significant reductions in oxidation rates as well as reduced Cr volatility were seen for these coatings as contrasted with bare steel surfaces. To better understand the reduced Cr volatility for the CrAlO system, we studied volatility as a function of alumina content in chromia/alumina solid solutions. Supported through HiTEC, funded by DOE (PNNL) No.DE-AC06-76RL01830. Work in EMSL supported by OBER (DOE) DE-AC06-76RLO1830.

5:00 PM
Investigation of Thin Film Mn-Co-Fe Spinel Oxide Coatings Deposited by RF-Magnetron Sputtering on Metallic Interconnects for SOFC Applications: *Cezarina Mardare*[1]; Michael Spiegel[1]; Alan Savan[2]; Alfred Ludwig[2]; [1]Max-Planck Institut fuer Eisenforschung; [2]Centre for Advanced European Studies and Research

In order to improve the properties (i.e. oxidation resistance, conductivity) of ferritic stainless steels created for SOFCs applications, thin metallic films from the Mn-Co-(±Fe) system were deposited by RF-magnetron sputtering on the metal surface. The depositions were followed by a heat treatment for converting the metallic coatings (300 or 1000nm) to $(Mn,Co,\pm Fe)_3O_4$ spinel oxides, and by long-term annealing in cathodic-like atmospheres for assessing the stability of the coatings. The samples were analyzed by GIXRD and SEM/EDX to confirm the presence of the spinel structure on top of the steel, and by ToF-SIMS to investigate and characterize the evolution and growth of internal oxides at the steel/coating interface after the long time exposure. For the thin (300nm) coatings after 500h, Cr penetration was observed. The evolution of Cr diffusion profiles in thicker coatings (1000nm) together with ASR measurements at high temperature for the coated steels is currently under investigation.

5:20 PM
Pulse Plating of Mn/Co Alloys for SOFC Interconnects Application: Junwei Wu[1]; Christopher Johnson[2]; Yinglu Jiang[1]; *Xingbo Liu*[1]; [1]West Virginia University; [2]National Energy Technology Laboratory

(Mn,Co)3O4 spinel is a promising coating for SOFC interconnect application due to its high conductivity. Besides, Mn1.5Co1.5O4 spinel demonstrated a good thermal expansion match to ferritic stainless steels such as AISI 430, as well as the cathode compositions such as LSM, LSF. Slurry coating and PVD has been reported to apply the (Mn,Co)3O4 spinel for SOFC interconnect application. Electroplating and following oxidation offers another cheaper option. In this research, the effects of three parameters of pulse plating, namely peak current density, on-time and off-time, on the composition and morphology of Mn-Co coatings were investigated. By varying the parameters, Mn/Co alloys was deposited successfully, whose surface and cross section was characterized by SEM/EDX and XRD. Further on-cell tests prove pulse plating Mn/Co is an alternative cheap option for interconnects coatings

5:40 PM
Structure and Stability of Surface Coated UNS 430 Ferritic Steel for SOFC Interconnect Application: *Hui Chen*[1]; Chih-Long Tsai[1]; Weerashinge Pritantha[1]; Richard Smith[1]; Paul Gannon[1]; Max Deibert[1]; [1]Montana State University

The development of intermediate temperature solid oxide fuel cells enable ferritic stainless steels as possible candidates for interconnects. Those SOFC interconnect should be stable both in oxidizing and reducing atmosphere. The thermal stabilities of UNS 430 stainless steels coated with dense and well adherent Ni or Cu protecting layers were studied in air and hydrocarbon fuel gases. Scanning electron microscopy was utilized to characterize the microstructure of the coated steels before and after oxidizing and carburizing. XRD was used to characterize the evolution of crystal structure. Rutherford backscattering with He+ and non-Rutherford scattering with H+ were used to characterize the composition and the interdiffusion of the coatings. The chromium volatility of the coated steel plates at 800°C was measured using ion beam analysis. Significant reductions in oxidation rates as well as reduced Cr volatility were observed for the coated alloys.

Materials Informatics: Enabling Integration of Modeling and Experiments in Materials Science: Informatics and Combinatorial Experiments and Materials Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizer: Krishna Rajan, Iowa State University

Tuesday PM　　Room: 271
March 11, 2008　　Location: Ernest Morial Convention Center

Session Chair: To Be Announced

2:00 PM
Combinatorial Approach to the Synthesis of Carbon Nanotubes: *Pablo Caceres-Valencia*[1]; [1]University of Puerto Rico-Mayaguez

Carbon Nanotubes are synthesized with the aid of a catalyst via the VLS mechanism. The relationship between the catalyst composition and the characteristics of the carbon nanotube is not known. This can be study using composition spreads. Three well known catalyst such as Fe, Co and Ni have been studied using composition spreads. These elements were deposited using a magnetron sputtering technique at an angle. The chemical composition was determined using EDS-SEM and a GMS-program . Before the exposure to a carbon gas, the sample was annealed in the CVD and in selected samples the particle size was measured as a function of particle composition. The carbon nanotubes were synthesized using acetylene as a carbon source. Carbon nanotubes were formed preferentially at the Fe corner. Small additions of Co to Fe increase the deposition rate.

2:40 PM
Informatics and High Throughput Screening of Thermophysical Properties: *Robert Hyers*[1]; Jan Rogers[2]; [1]University of Massachusetts; [2]NASA Marshall Space Flight Center

The combination of computer-aided experiments with computational modeling enables a new class of powerful tools for materials research. A non-contact method for measuring density, thermal expansion, and creep of undercooled and high-temperature materials has been developed, using electrostatic levitation and optical diagnostics, including digital video. These experiments were designed to take advantage of the large volume of data (many gigabytes/experiment, terabytes/campaign) to gain additional information about the samples. For example, using sub-pixel interpolation to measure about 1000 vectors per image of the sample's surface allows the density of an axisymmetric sample to be determined to an accuracy of about 200 ppm (0.02%). A similar analysis applied to the surface shape of a rapidly rotating sample is combined with finite element modeling to determine the stress-dependence of creep in the sample in a single test. Details of the methods for both the computer-aided experiments and computational models will be discussed.

3:20 PM
Informatics for Imaging and Spectroscopy Databases: *H. Meco*[1]; M. Heying[1]; K. Boysen[1]; M. Stukowski[1]; W. Qin[1]; W. Hu[1]; Krishna Rajan[1]; [1]Iowa State University

Quantitative interpretation of large amounts of spectroscopy and imaging data is challenging due to very large amounts of data. Informatics provides techniques that not only help to manage data but also to track correlations in a systematic way, and also identify subtle features that otherwise are difficult to see. In this presentation we provide examples using a variety of material characterization techniques including electron microscopy, 3D atom probe tomography and diffraction. The integration of informatics with spectroscopy data in is shown as a means of significantly accelerating the interpretation of complex high dimensional data.

4:00 PM
Modeling of Titan Oxidation Process in Fluidized Bed: *Szota Michal*[1]; Jozef Jasinski[1]; Tomasz Mrozinski[1]; [1]Czestochowa Technical University

This paper presents neural network model used for designing leading of titan oxidation processes in fluidized bed. In this case the neural network structure is designed and prepared by choosing input and output parameters of process. The method of learning and testing neural network, the way of limiting nets structure and minimizing learning and testing error are discussed. Such prepared neural network model, after putting expected values of for example assumed microhardness curve and thickness of surface layer in output layer, can give answers to a lot of questions about running oxidizing process in fluidized bed. The neural network model can be used to build control system capable of on-line controlling leading process and supporting engineering decision in real time.

Materials Processing Fundamentals: Smelting and Refining

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Process Technology and Modeling Committee
Program Organizer: Prince Anyalebechi, Grand Valley State University

Tuesday PM — Room: 283
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chair: Prince Anyalebechi, Grand Valley State University

2:00 PM
Precipitation Rate of Hematite in Sulfate Media: *Maria Ruiz*[1]; Daniel Castillo[1]; Rafael Padilla[1]; [1]University of Concepcion

Hematite precipitation is a convenient alternative for the removal of iron from leaching circuits because of its chemical stability, high density and high iron content. The precipitation of hematite from ferrous sulfate solutions was studied in an autoclave. The precipitation occurs by two sequential steps: oxidation of the ferrous to ferric ions followed by hydrolysis of the ferric to hematite. The objective of this study was to determine the effect of operating variables on the two-step rate of hematite precipitation. The variables included: stirring speed (100 to 600 rpm), temperature (160 to 200°C), acid concentration (1 to 10 g/L), initial iron concentration (1 to 15 g/L), and oxygen overpressure (1 to 6 atm). The sulfur content and the particle size of the precipitates were also determined. The results showed that the rate of oxidation of Fe2+ to Fe3+ is the controlling step of the overall process.

2:20 PM
A New Molten $CaCl_2$ Electrolysis Process for Titanium from High Titanium Slag: Fanke Meng[1]; *Huimin Lu*[1]; [1]Beijing University of Aeronautics and Astronautics

A new method for direct reduction of high titanium slag in molten $CaCl_2$ is studied in this paper. High titanium slag is made as cathode; an inert material is used as anode, and $CaCl_2$ as electrolyte. During the electrolysis, oxygen ion from high titanium slag cathode is reduced to oxygen on the surface of the anode; titanium is obtained on the cathode sinking underneath molten high titanium slag. The gas released on the anode is oxygen rather than carbon oxides and the cathode used is molten high titanium slag rather than solid titanium oxide, the electrolysis temperature is about 1700°C. This process is environment-friendly, low energy consumption and high current efficiency.

2:40 PM
Pressure Leaching of Enargite in Sulfate-Oxygen Media: *Rafael Padilla*[1]; Cristian Rivas[1]; [1]University of Concepcion

Enargite (Cu_3AsS_4) occurs normally associated with copper sulfides, mainly chalcopyrite. When the enargite content in chalcopyrite concentrates is high, direct smelting of the concentrate presents problems related to environmental pollution and contamination of the final metallic copper. Leaching processes are viable alternatives to treat copper concentrates with high content of enargite. Consequently, in this paper, the pressure leaching of enargite in sulfuric acid-oxygen system is discussed. The leaching rate was studied at 160 to 220°C and partial pressures of oxygen of 74 to 200 psi. The enargite dissolution increased substantially with temperature increase. Complete dissolution of enargite was obtained at 220°C, and 100 psi of oxygen pressure in 90 min. The shrinking core model controlled by surface reaction was used to analyze the kinetics of enargite dissolution. Activation energy of 70 kJ/mol was obtained for the temperature range 160 to 220°C.

3:00 PM
In-Situ Analysis of the Effect of Other Oxides for the Reduction Process of Wustite by Carbon in TEM: *Ishikawa Nobuhiro*[1]; Takeshi Aoyagi[1]; Takashi Kimura[1]; Kazuo Furuya[1]; Nayuta Mitsuoka[2]; Takashi Inami[2]; [1]National Institute for Materials Science; [2]Ibaraki University

The reduction of iron oxides has been one of the most frequently studied topics in iron smelting. We developed the in-situ observation of the reduction of iron oxides by solid state carbon in transmission electron microscope (TEM). The pure wustite (FeO) was used in the previous study as an iron oxide. But many kinds of additive is used to promote the reduction efficiency. The purpose of this study is to analyze the effect of additive during the reduction. We prepared several wt % of lime (CaO) and alumina (Al_2O_3) including wustite. Then the reaction between wustite and carbon was observed in TEM at about 800-1100K. The results were completely different from using pure wustite. Lime suppressed the reaction and Alumina promoted the reaction.

3:20 PM
Development of a Static Model for COREX Process: *Wang Chen*[1]; [1]Northeastern University

COREX process is briefly introduced. A static model combining a set of mass and heat balance equations has been developed. Taking the specified inputs from the graphical user interfaces, the model is capable of calculating the consumption of raw materials and fluxes, the volume and composition of the slag and the volume and composition of the generator gas. The model enables the examination of the changes of materials and energy flows induced by the variations of operational parameters and raw materials chemical analysis. The model allows a detailed process analysis and adjustment of the plant operation and can thus be used to improve plant operation.

3:40 PM Break

3:50 PM
Thermal Stability of Nano-Twins in Pulse Electrodeposited Cu: Experimental and Calculations: *Di Xu*[1]; Luhua Xu[1]; King-Ning Tu[1]; Vidvuds Ozolins[1]; [1]University of California, Los Angeles

Cu with high density of nano-twins has excellent mechanical strength and electrical properties at the same time. It has potential to improve the reliability of Cu interconnects for very-large-scale-integration of circuits in the near-future

Si microelectronic technology. The thermal stability of nano-twinning structure is of concern for large scale production and its future application. The annealing of pulse-electroplated Cu films shows that the thermal stability of nano-twins depends on the stability of {112} incoherent twin boundaries which are perpendicular to the {111} coherent twin planes. First principle calculations and MD simulations are used to study the twin boundary energy and mobility of twin planes. The simulations show a considerable mobility of {112} twin boundary while a near-zero mobility of {111} twin boundary, which is consistent with the experimental results.

4:10 PM
Thermodynamic and Kinetic Studies of Hydrochloric Acid Leaching Process of Panzhihua Ilmenite for Rutile Synthesis: *Jilai Xue*[1]; Zengjie Wang[1]; Haibei Wang[2]; Xunxiong Jiang[2]; [1]University of Science and Technology Beijing; [2]Beijing General Research Institute of Mining and Metallurgy

Thermodynamic and kinetic studies of hydrochloric acid leaching of Panzhihua ilmenite from China are presented. The Eh-Ph diagram for system Fe-Ti-Cl-H_2O has been constructed, where a reaction medium of high acidity is of necessity to make iron into solution as Fe2+. The leaching process is found to follow the spherical model f(a)=1-(1-a)1/3 and the apparent activation energy is calculated as 47.21 KJ/mol. The studies reveal different dissolving behaviors in various kinetic stages of the acid leaching. At the beginning stage the dissolved amount of both Fe and Ti increased monotonically with time of leaching, while about 30 minutes later the dissolved Ti in the form of $TiOCl_2$ began hydrolyzed into TiO_2 solid powder that reunited on the surface of ilmenite ore, as observed by SEM. The leaching process can end 4h later when most of Fe and other impurities have been dissolved and the hydrolyzing of $TiOCl_2$ almost finished.

4:30 PM
Thermodynamic Analysis and Its Application to Preparing Sm-Fe Alloy Oxide Precursor by Wet-Chemical Co-Precipitation: Ping Xue[1]; *Xueyi Guo*[1]; Qinghua Tian[1]; Rongquan Jiang[1]; [1]Central South University

Sm_2Fe_{17} alloy is an important precursor for the preparation of $Sm_2Fe_{17}N_x$ magnet, which possesses a higher Curie temperature, larger anisotropy field, better resistance to oxidation and thermal stability. In this paper, the wet chemical co-precipitation was developed for synthesis of the Sm-Fe alloy oxide precursor. Based on the principles of simultaneous equilibrium and mass balance, a series of thermodynamic equilibrium equations of Sm(β)-Fe(α)-NH_3-C_2O_{42}-H_2O system at ambient temperature were deduced theoretically and the thermodynamic diagrams of lg[M2+]-T (M=Sm,Fe) versus pH at different solution compositions were drawn. Then, the experiments were done to address the various effects on the wet chemical process, including solution pH value, reactant concentration, reaction temperature and aging time, etc. It is found that by precise control of the process, the Sm-Fe alloy oxide precursor was synthesized with fine crystallization and special size and size distribution.

Mechanical Behavior, Microstructure, and Modeling of Ti and Its Alloys: Microstructure/Property Correlation I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
Program Organizers: Ellen Cerreta, Los Alamos National Laboratory; Vasisht Venkatesh, TIMET; Daniel Evans, US Air Force

Tuesday PM Room: 384
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Charles Ward, US Air Force; Patrick Martin, US Air Force

2:00 PM Invited
Low-Temperature Tensile and Creep Deformation Behavior of Grade 7 Commercially Pure Titanium Alloy: Paul Oberson[1]; S. Ankem[2]; *Kuang-Tsan Chiang*[3]; Luis Ibarra[4]; [1]U.S. Nuclear Regulatory Commission; [2]Ankem Technologies Inc.; [3]Southwest Research Institute; [4]Center for Nuclear Waste Regulatory Analyses (CNWRA)

The drip shield design considered by the U.S. Department of Energy includes Titanium Grade 7 (Ti-0.2wt%Pd-0.13wt%O) components.The tensile and creep behavior of Grade 7 Titanium alloy was independently investigated by the Center for CNWRA at low temperatures (25 to 250°C).The tensile behavior exhibited flow stress drops that increased with an increase in temperature.This behavior was attributed to a rise in the mobile dislocation density.In regard to creep deformation, there was no change in the amount of creep strain with an increase in temperature at 85% of the respective yield stress.This behavior was attributed to a change in the deformation mechanisms from slip and twinning at low temperatures (25 to 50°C) to solely slip at higher temperatures. Disclaimer: This abstract is an independent product of the CNWRA and does not necessarily reflect the view or regulatory position of the NRC.

2:30 PM
Application of Thermodynamic Modeling Tools in the Prediction of Titanium Alloy Properties: *Manish Kamal*[1]; Vasisht Venkatesh[1]; [1]Titanium Metals Corporation

Thermodynamic modeling tools are becoming increasingly important in materials development and processing. However, the use of these tools in the Titanium industry is still in its early phase. This study details the validation study at TIMET of PANDAT, a thermodynamic modeling tool. PANDAT was used to generate beta transus and alpha approach curves for production billets. Validation of the thermodynamic model was carried out with experimental techniques using digital image analysis and manual point count method. Model predictions were compared for alpha-beta alloy (Ti-64) and a near alpha, alpha-beta alloy (Ti-6242). Effects of interstitial elements, ingot chemistry and product chemistry and top and bottom ingot chemistry on the approach curve will be discussed.

2:50 PM
Characterization and Neural Network Models of a Creep-Resistant β-Titanium Alloy Based on Ti Metal-21S: *Benjamin Peterson*[1]; Peter Collins[1]; Hamish Fraser[1]; [1]Ohio State University

Timetal 21S has been selected as a baseline composition for the development of a new high temperature β-Ti alloy. A combinatorial approach employing directed laser deposition has been used to produce test coupons with different compositions. In addition to variations in the amounts of the elements present in the unmodified alloy (Ti, Mo, Nb, Al, and Si), the alloys contain varying amounts Zr, Sn, W, B, C, and Ge. Subsequently, the creep properties (minimum creep rate) have been assessed and the microstructures characterized and quantified. This data is used to populate databases and subsequently train and test Bayesian Neural Network models for the prediction of creep properties. Advanced characterization techniques and computation tools are employed to identify the creep rate-limiting features. For example, SEM and TEM studies show a critical dependence of the size of a-denuded β regions on the creep properties in these β-Ti alloys.

3:10 PM
Correlating Fracture Topography and Underlying Microstructure in α/β Ti Alloys: *Adam Pilchak*[1]; Robert Williams[1]; Andrew Rosenberger[2]; Mary Juhas[1]; James Williams[1]; [1]Ohio State University; [2]US Air Force

High strength titanium alloys contain two ductile phases, the hcp α- phase and the bcc β-phases. The size and spatial distribution of these two constituents largely influence the strength and fracture related properties of these alloys. Consequently, the resulting fracture topography can be complex, even confusing. We have been developing or refining several methods for interpreting the fracture topography of Ti alloys. For example, using a focused ion beam to reveal underlying microstructure then using EBSD to determine constituent crystallographic orientation beneath particular fracture features. This talk will present and describe some examples of these methods for specimens fatigued to fracture under a variety of conditions and lifetimes. For example, it will be demonstrated that care must be taken to not confuse low ΔK fatigue fracture with traditional cleavage fracture observed in bcc metals. This work is sponsored by ONR under Project # N00014-06-1-0089, Dr. Julie Christodoulou, Manager.

3:30 PM
Effect of Microstructure on the Elevated-Temperature Low-Cycle Fatigue Behavior of Boron-Modified Ti-6Al-4V: *Wei Chen*[1]; Carl Boehlert[1]; Sesh Tamirisa[2]; Daniel Miracle[3]; [1]Michigan State University; [2]FMW Composite Systems, Inc.; [3]Air Force Research Laboratory

This study investigated the effect of boron addition on the low-cycle fatigue behavior of Ti-6Al-4V(wt.%) at elevated-temperature (455°C) for stresses between 200-550MPa (R=0.1; frequency=5Hz).The boron concentration of alloys was 0wt.%, 0.1wt.% and 1wt.% respectively, and alloys in two different processing conditions, as-cast and cast plus extruded, were tested.The results indicated that the boron additions improved the fatigue properties of Ti-6Al-4V.The cast-plus-extruded alloys exhibited significantly greater fatigue lives than the as-cast alloys.This was attributed to the microstructural changes caused by extrusion.S-N curves and the microstructure of each alloy will be presented, and the fracture and deformation behavior of the Ti-6Al-4V-xB alloys will be compared.

3:50 PM Break

4:10 PM Invited
Microstructure Development, Mechanics and Behaviour of Near-Beta and Beta-Titanium Alloys: *David Dye*[1]; Richard Dashwood[1]; Martin Jackson[1]; Seema Raghunathan[1]; Russell Talling[1]; Nicholas Jones[1]; [1]Imperial College

Near-beta and beta titanium alloys are of current interest from a practical point of view because of their using in landing gear and biomedical applications. Theoretically they are also interesting because of (i) the ability to form fine-scale (20nm) alpha, (ii) the vulnerability to omega formation and (iii) because of the tailorability of the single crystal elastic moduli with alloying. We will present recent results, chiefly from synchrotron x-ray diffraction, on the mechanics of these alloys. These show that (i) the elastic constants of the beta phase change dramatically when the beta is enriched by fine-scale alpha precipitation, (ii) that the single crystal elastic constants of GUM metal alloys can be characterised using synchrotron diffraction, (iii) that the dynamics of omega precipitation and subsequent alpha formation can be characterised, in situ, with ~2 second time resolution. In addition, some observations on the grain behaviour during hot working will be shown.

4:40 PM
Microstructural Investigation of Crack Initiation During Dwell Fatigue in Alpha/Beta Titanium Alloys: *Amit Bhattacharjee*[1]; S. Rokhlin[1]; M. Mills[1]; S. Ghosh[1]; J. Williams[1]; [1]Ohio State University

Several near alpha titanium alloys are used widely by various aero-engine manufacturers in the lower temperature and pressure region of an aircraft engine both as discs and blades. These alloys are prone to micro/macro texturing which causes debit in the dwell (where load is held at a constant value) time fatigue life and are also detrimental to other mechanical properties. A combination of X-ray radiography, orientation imaging microscopy (OIM) in a scanning electron microscope and polarized light microscopy has been utilized to characterize the local microtexture around internal secondary and primary fatigue cracks.This information is being used to generate image-based, finite element models of local strain and stress development, with the ultimate goal of determining a local crack initiation criteria for dwell fatigue.

5:00 PM
Multiscale and Multistage Characterization of Fatigue Damage in Ti-6Al-4V: *Rikki Teale*[1]; Andrea Keck[1]; Manuel Parra[1]; Pedro Peralta[1]; [1]Arizona State University

Fatigue damage on notched and "smooth" specimens of Ti-6Al-4V was monitored to account for effects of local microstructure on nucleation and propagation of short and long. Grain/colony sizes and crystallographic orientations around nucleation sites and along the potential fatigue crack propagation paths are characterized using Electron Backscattering Diffraction (EBSD) in the test specimens. The geometry of short cracks is examined using serial sectioning and correlated to the local crystallographic environment. In addition, in-situ loading experiments were performed under optical and scanning electron microscopes to obtain strain fields beyond the crack tip via digital image correlation (DIC). These tests will provide data to quantify the effects of local microstructure on crack nucleation and crack propagation. These data, in turn, will be use to build a progressive damage model of crack growth at the microscale. Work funded by a Structural Health Monitoring MURI, Department of Defense AFOSR Grant FA95550-06-1-0309, Victor Giurgiutiu program manager.

5:20 PM
Mechanical Behavior and Microstructure of Ti-6Al-4V-0.25O Rich Oxygen Titanium Alloy: Hui Wang[1]; *Huimin Lu*[1]; [1]Beijing University of Aeronautics and Astronautics

Self-propagating High-temperature Synthesis (SHS) with reduction process was used to fabricate the Ti-6Al-4V-0.25O powder from TiO_2-V_2O_5-Al-CaH_2 system. In this process, there was no chlorine and the oxygen content in the powder was about 0.2% ~0.3%. The Ti-6Al-4V-0.25O alloy material was prepared by powder injection method from the rich oxygen titanium alloy powder. Some mechanical behavior and microstructure of the rich oxygen titanium alloy material was superior to Ti-6Al-4V titanium alloy. This process for producing the rich oxygen titanium alloy material is environment-friendly and low-cost.

5:40 PM
Thermophysical and Structural Properties of a Gamma–TiAl Alloy in the Temperature Range of 50-900°C: *Rainer Wunderlich*[1]; Taishi Matsushita[1]; Robert Brooks[1]; Seshadri Seetharaman[1]; Hans-Jörg Fecht[1]; [1]Ulm University

Gamma-TiAl alloys offer great promise as a turbine blade material in jet engines because of increased specific strength and good creep resistance while issues like embrittlement at elevated temperature still need to be solved. Characterization of the material microstructure, its thermophyical properties and their temperature dependence is an important aspect in product development. Within the framework of the EC-FP6 IMPRESS project the thermophysical and microstructural properties of a Ti-45.5at%Al-8at%Nb have been investigated. The thermal diffusivity exhibited a pronounced maximum at T=750°C which could not be explained by the temperature dependence of the specific heat capacity or that of the electrical resistivity. A temperature dependent XRD-investigation revealed a change in the intensities of the γ-phase (111), (200),and (222) reflections as a function of temperature which otherwise would be typical for texture. This observation is interpreted as a temperature dependent reorientation of γ-lamellea resulting from twinning and the release of internal stress.

Mechanics and Kinetics of Interfaces in Multi-Component Materials Systems: Interfacial Microstructures and Effects on Mechanical and Physical Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee, TMS: Thin Films and Interfaces Committee
Program Organizers: Bhaskar Majumdar, New Mexico Tech; Rishi Raj, University of Colorado, Boulder; Indranath Dutta, US Naval Postgraduate School; Ravindra Nuggehalli, New Jersey Institute of Technology; Darrel Frear, Freescale Semiconductor

Tuesday PM Room: 279
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Nuggehalli Ravindra, New Jersey Institute of Technology; Corbett Battaile, Sandia National Laboratories

2:00 PM Invited
Effect of Rare-Earth Additions to Al-Sc Alloys on Precipitate Composition and Resistance to Dislocation Motion: *David Seidman*[1]; Richard Karnesky[1]; Marsha van Dalen[1]; David Dunand[1]; [1]Northwestern University

Al-Sc alloys display a high number density of nanosize trialuminide $L1_2$ precipitates with coherent interface with the Al matrix. The rare-earth (RE) elements Y, Sm, Gd, Dy, Er and Yb were found to partition to the precipitates. Local Electrode Atom Probe was used to follow the evolution of the RE concentration in the precipitates as a function of aging time at 300°C. RE segregate towards the center of the Al3(Sc,RE) precipitates, minimizing lattice mismatch with the matrix. Hardness and creep measurements were used to assess the effect of Sc replacement by RE within the precipitates. RE do not increase hardness of the alloys, as expected from the fact that matrix dislocation bypass precipitates by the Orowan mechanism at ambient temperature. RE however improve creep resistance of the alloy, which is explained by the increased matrix/precipitate lattice mismatch resulting in enhanced elastic interactions with matrix dislocations.

2:30 PM
Stress Effects on Hydrogen Trapping in the Proximity of TiC-Fe Interfaces: Masato Enomoto[1]; *Jong Lee*[2]; [1]Ibaraki University; [2]Michigan Technological University

In steels, the interfacial region between ferrite matrix and TiC inclusions is suspected of a major trapping site for hydrogen, but the trapping nature is unknown. Due to the lattice misfit, fine TiC particles (< 20 nm) are coherent, whereas coarse ones are incoherent. To understand the role of the misfit strain in hydrogen trapping, a micromechanics analysis is performed. Elastic stress within a TiC particle and in the Fe matrix at the interface is computed, and its geometric mean is defined as a trap stress for hydrogen. The results show that if the shape is plate-like, the trapping strength is greater for incoherent than for coherent particles. Within TiC particles, hydrogen atoms are proposed to replace some carbons due to the compressive stress. In the bcc Fe matrix, however, hydrogen atoms are to take on expanded interstitial sites for the coherent case and dislocation cores for the incoherent case.

2:55 PM Invited
Synchrotron X-Ray Microscopy Studies on Electromigration of a Two-Phase Material: *K. Subramanian*[1]; Andre Lee[1]; Cheng-En Ho[1]; Wenjun Liu[2]; [1]Michigan State University; [2]Argonne National Laboratory

Electromigration in multi-phase alloys has become important due to their utilization in microelectronic interconnects. Synchrotron X-ray microscopy has provided information regarding 2- and 3-dimensional crystallographic orientations and strain fields, providing documentation of microstructural evolution and surface features from current stressing of such alloys. Micro-beam X-ray fluorescence investigations indicated different conducting species move at different rates during electromigration. The continuous build-up of slow moving dominating species just behind the fast moving species causes significant compressive deformation in the anode region to push out the fast moving species towards the free surface forming the hillock. Micro-beam X-ray diffraction studies indicated the existence of significant compressive strain along the direction of electron flow near the anode, as well as tensile strains in directions perpendicular to the electron flow. Electron wind forces provide extra impetus for rapid morphological changes to produce larger grains of individual phases towards the anode.

3:25 PM
The Effect of Grain Boundary Character Distribution on Coble Creep: *Ying Chen*[1]; Christopher Schuh[1]; [1]Massachusetts Institute of Technology

As the properties of an individual interface largely depend on its atomic structure, the broad distribution of interfacial structures in polycrystalline materials presents a challenge to understanding the structure-property relationship. In this talk, we will discuss the effect of grain boundary network heterogeneity on grain boundary diffusional (Coble) creep. The grain boundaries are classified into slow-diffusing “special” boundaries and fast-diffusing “general” ones. The creep viscosity of the polycrystal is obtained from computer simulations, and is characterized as a function of the fraction of the different types of boundaries. This basically defines a new class of percolation problem where mass diffusion and force equilibrium are coupled. In addition to the percolation aspects, we also explore stress concentrations induced by the grain boundary character distribution, and an empirical effective medium equation that may be used with classical creep constitutive laws in order to predict the viscosity of a heterogeneous material.

3:50 PM Break

4:00 PM Invited
The Formation of Wear-Induced Microstructures during Sliding Contact: *Corbett Battaile*[1]; Somuri Prasad[1]; Joseph Michael[1]; Bhaskar Majumdar[2]; [1]Sandia National Laboratories; [2]New Mexico Tech

Friction can lead to complex mechanical and microstructural evolution near the worn surface, and these changes can impact the properties of the material. Recent results from tribological experiments on nickel single crystals reveal the formation of microstructural features ranging from nanometers (very near the surface) to microns in size. The formation and mechanical response of these zones is sensitive to crystallography, and can dramatically alter the frictional properties of the material itself. We have modeled these phenomena using combined treatments of crystal plasticity, microstructure formation, and grain boundary sliding. The loading conditions are adopted from an analysis of static frictional contact. A phenomenological treatment of wear debris and asperity-mediated contact is included to appropriately describe the mechanical mixing that occurs very near the contact interface. We will provide an overview of the experimental evidence, discuss the wear model in detail, and present results for kilocycle wear on nickel single crystals-in-different-crystallographic-orientations.

4:25 PM Invited
The Role of Interfaces on the Mechanics of Shape Memory Alloy (SMA) Fiber Reinforced Pb-Free Solder Composites: *Nikhilesh Chawla*[1]; J. Coughlin[1]; J. Williams[1]; [1]Arizona State University

It is well known that solder joints must retain their mechanical integrity under conditions of isothermal aging, thermal fatigue, and creep. In recent years, shape memory alloys (SMA) have received significant attention due to their ability to be deformed and return to their original dimensions, upon unloading and/or the application of heat. Recently, SMA has been used as a reinforcement phase to form “smart” composite materials. The unique properties of SMA are being exploited to design damage tolerant materials that exhibit a beneficial response under applied loading. In this paper, we report on the interfacial reactions and mechanics of NiTi shape memory alloy fiber reinforced Sn matrix composites. It will be shown that the reaction products and kinetics are quite complex. The load transfer to the fiber is governed by the strength of the interface, aspect ratio of the fiber, and yield stress of the matrix. The evolution of the microstructural constituents and the influence on deformation behavior will be discussed.

4:55 PM
Formation and Growth of Ag Crystallites in Screen-Printed Ag Contacts for Crystalline Si Solar Cells: *Kyoung-Kook Hong*[1]; Sung-Bin Cho[1]; Joo-Youl Huh[1]; JaeSung You[2]; Ji-Weon Jong[2]; [1]Korea University; [2]LG Chem, Ltd. Research Park

The PbO-based glass frits contained in Ag paste are known to play an important role in the screen-printed Ag contact formation. Recently, there is an environmental demand for the development of a Pb-free glass frit to replace the conventional PbO-based frit. In search of the PbO replacement, it is crucial to understand first the role of PbO in the Ag contact formation. However, it still remains unclear how PbO in Ag pastes plays a role in the formation and growth of Ag crystallites at the paste/Si interface. In this study, we investigated the reactions between leaded Ag pastes and n-type [100] Si wafer coated with SiNx antireflection layer in an tube furnace at 800°C for 5 to 30 min, in order to understand the mechanism for the formation and growth of the inverse pyramidal Ag crystallites at the interface between the Ag paste and Si during the firing process.

5:20 PM
A Study of Interfacial Growth Kinetics and Mechanics for an Fe Fibre Reinforced Al Alloy: *Scott Kenningley*[1]; Simon Barnes[2]; [1]Federal Mogul and Manchester University; [2]Manchester University

The incorporation of long Fe based fibres into Al alloys has shown promise for significantly increased levels of high temperature fatigue resistance. This fatigue resistance, in combination with a relatively inexpensive manufacturing route, makes this composite a strong candidate material for highly loaded automotive engine products such as pistons. One of the problems with using this composite at highly elevated temperatures, is the reaction between the Fe based fibres and the Al matrix to produce brittle intermetallic phases. In the current study, fibre-matrix interfacial intermetallic growth has been characterised and modelled as a function of fibre volume fraction, heat soak temperature and time. The influence of changing interface intermetallic composition, morphology and size on the high temperature fatigue resistance of the composite has also been examined in an attempt to produce an FE model to predict behaviour of the composite as a function of its loading and thermal history.

Minerals, Metals and Materials under Pressure: High Pressure Phase Transitions and Mechanical Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Richard Hennig, Cornell University; Dallas Trinkle, University of Illinois; Ellen Cerreta, Los Alamos National Laboratory

Tuesday PM — Room: 385
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Dallas Trinkle, University of Illinois; Richard Hennig, Cornell University

2:00 PM Invited
On Failure in Polycrystalline and Amorphous Brittle Materials: *Neil Bourne*[1]; [1]Los Alamos National Laboratory

Materials found across natural environments experience mechanical extremes of pressure, temperature and strain rate or survive electromagnetic loads of great violence. Reaching an understanding of such states and the response of materials subject to them is key in fully describing operating deformation mechanisms. Armed with physically based models, it is possible to contemplate designing not only structures to operate within such environments, but also to adopt strategies to engineer materials with optimized properties to survive there. Key features of the dynamic response of brittle solids will be reviewed leading to understanding of natural materials under dynamic loads and to a new understanding of material design for use in extreme environments.

2:30 PM
Shear Strength and Texture Evolution in Tantalum Foils: *Juan Escobedo*[1]; David Field[1]; David Lassila[2]; [1]Washington State University; [2]Lawrence Livermore National Laboratory

A new experimental apparatus has been developed for performing shear tests on specimens held under moderately high hydrostatic pressures. The experiments provide calibration data for models of materials subjected to extreme pressures such as the Steinberg-Guinan hardening model, among others. This paper reports the development of the experimental procedures and the results of experiments on thin foils of 111 textured polycrystalline Ta performed under hydrostatic pressures ranging from 1 to 5 GPa. Both yielding and hardening behavior of Ta are observed to be sensitive to the imposed pressure. EBSD was performed on the deformed specimens in order to determine what fraction of the specimen underwent a shear-type of deformation, these observations were correlated with the predicted texture that results by shearing a 111 texture material to a shear strain of 1 according to the visco-plastic self-consistent model. Finally burgers vector determination, dislocation cell structures were characterized by post-mortem TEM analysis.

2:50 PM Break

3:00 PM
Interpretation of Shock Damage Quantification Results through Polycrystalline Computer Simulations: *Davis Tonks*[1]; Veronica Livescu[1]; John Bingert[1]; [1]Los Alamos National Laboratory

Quantification of ductile incipient damage in Tantalum samples from plate impact experiments has been performed using serial sectioning, EBSD, and optical microscopy. The influence of local crystal grain structure on void nucleation and growth and the role of plastic flow localization were investigated and will be reported on in another paper in this meeting (Livescu et al). This analysis is incomplete, however, without including the effects local stress and strain histories on damage creation. In this work, we compare the quantified damage results with computer simulations in which the experimental crystal grain structure has been explicitly represented in the mesh and the crystal grain plasticity is modeled phenomenologically. Void growth is treated in a simplified way. The calculated results reveal the stress and strain concentrations, which induce damage. The calculations and quantified damage will be analyzed for systematic trends that will then be modeled.

3:20 PM
First-Principles Studies of Structural and Mechanical Properties of XB_2 (X = W, Os, Re): *Xing-Qiu Chen*[1]; C. L. Fu[1]; [1]Oak Ridge National Laboratory, Materials Science and Technology Division

Transition-metal diborides, XB_2 (X = W, Os, Re), have attracted great interest due to their hard, ultra-incompressible behavior. Using ab initio density functional approach, we have studied the structural, electronic, and elastic properties of these compounds. The orthorhombic OsB_2 is found to undergo a phase transition into the ReB_2-type hexagonal phase above 2.5 GPa. For WB_2, we predict a ReB_2-type hexagonal structure at zero pressure; above 62 GPa, however, WB_2 transforms into the experimentally reported AlB_2-type structure. Among all of the studied structures, the ReB_2-type hexagonal structure exhibits the largest incompressibility along the c-axis, comparable to that of diamond. The origin of the ultra-incompressibility correlates not only with the strong covalence between B-B and X-B but also with the local buckled structure interconnected with covalent bonds. Research sponsored by the Division of Materials Sciences and Engineering, U.S. DOE, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

3:40 PM Break

4:00 PM Invited
High Pressure Materials Research Using Advanced Third Generation Synchrotron X-Ray: *Choong-Shik Yoo*[1]; [1]Washington State University

Compression energy ($P\Delta V$) at 100 GPa (10^6 atmospheres or 1 Mbar) often exceeds 1 eV or 100 KJ/mol, rivaling the energy of strong chemical bond. Therefore, the application of such a high pressure significantly alters chemical configuration, electronic and crystal structure, thermal, mechanical and chemical properties of solid and, in turn, provides a way to test condensed matter theory and to exploit novel properties and exotic materials. Furthermore, recent advances in diamond-anvil cell (DAC) high-pressure technologies coupled with advanced third-generation synchrotron x-ray offer unprecedented opportunities to discover exotic states of matter at high pressure-temperature conditions of Earth and other Giant planetary interiors. In this paper, I will discuss several recent results of high-pressure chemistry arising from pressure-induced electron delocalization in simple low-z molecular solids and in strongly correlated f-and d-transition metals at pressures of 100 GPa, following the brief descriptions of modern DAC technologies coupled with laser-heatings and synchrotron x-ray diffraction and spectroscopy. Also presented are the future directions of high-pressure synchrotron materials research in an emerging/complementary phase and time scales of shock and static high pressures.

4:20 PM

Delta to Alpha-Prime Phase Transformation in a Pu-Ga Alloy under Hydrostatic Compression: *Adam Schwartz*[1]; Mark Wall[1]; Daniel Farber[1]; Kevin Moore[1]; Kerri Blobaum[1]; [1]Lawrence Livermore National Laboratory

Delta-phase Pu-Ga specimens, 2.3 mm diameter by 100 microns thick were compressed to approximately 1 GPa in a large volume moissanite anvil cell to induce the transformation to the alpha-prime phase. The recovered samples were characterized at ambient pressure with optical microscopy, x-ray diffraction, and transmission electron microscopy. Optical microscopy revealed a very fine microstructure that appears to be single phase. This preliminary conclusion was supported by x-ray diffraction, which showed only the monoclinic reflections from the alpha-prime phase. However, transmission electron microscopy revealed small regions of delta-phase with a very high dislocation density. From these results, we conclude that hydrostatic compression to 1 GPa is not fully sufficient to form and retain 100% alpha-prime. This work was performed under the auspices of the United States Department of Energy by the University of California, Lawrence Livermore National Laboratory, under Contract No. W-7405-Eng-48.

4:40 PM Break

4:50 PM

Pressure Induced Phase Transitions in PTFE: *Eric Brown*[1]; Philip Rae[1]; George Gray[1]; Dana Dattelbaum[1]; David Robbins[1]; [1]Los Alamos National Laboratory

We present an experimental study of crystalline structure evolution of polytetrafluoroethylene (PTFE) due to pressure-induced phase transitions in a semi-crystalline polymer using soft-recovery, shock-loading techniques coupled with mechanical and chemical post-shock analysis. Gas-launched, plate impact experiments have been performed on pedigreed PTFE 7C, mounted in momentum-trapped, shock assemblies, with impact pressures above and below the phase II to phase III crystalline transition. Below the phase transition only subtle changes were observed in the crystallinity, microstructure, and mechanical response of PTFE. Shock loading of PTFE 7C above the phase II–III transition was seen to cause both an increase in crystallinity from 38% to ~53% (by Differential Scanning Calorimetry, DSC) and a finer crystalline microstructure, and changed the yield and flow stress behavior. Results are discussed in the context of class shock hugoniot measurements and diamond anvil cell measurements on polymers.

5:10 PM

High-Pressure Amorphization of Boron Carbide: *Mingwei Chen*[1]; [1]Tohoku University

Super-hard materials with strong covalent bonds can withstand very high stress prior to failure. However, the underlying mechanisms of high-pressure deformation and damage that lead to the failure of super-hard materials are poorly known. Boron carbide, B_4C, is one of the hardest materials and has been demonstrated to have outstanding ballistic armor properties. A variety of constitutive models have been developed to predict its performance at high shock pressures. However, the results have been mixed because the actual deformation/damage micromechanisms remain indefinite, which have been the subject of much intense work over the last 30 years. Here we report unusual depressurization amorphization in single-crystal B_4C subjected to diamond anvil cell experiments with nonhydrostatic pressures up to 50 GPa. Our findings provide a comprehensive portrayal on the high-pressure damage mechanisms of B4C that will allow for more robust models and may be generically applicable to other super-hard materials in similar environments.

Neutron and X-Ray Studies for Probing Materials Behavior: Recrystallization

Sponsored by: National Science Foundation, The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee

Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter K. Liaw, University of Tennessee

Tuesday PM — Room: 391
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Gabrielle Long, Argonne National Laboratory, Advanced Photon Source; Dorte Jensen, Riso National Laboratory

2:00 PM Keynote

Neutron and X-Ray Studies of Recrystallization: *Dorte Jensen*[1]; [1]Riso National Laboratory

Neutron and x-ray methods offer complementary information to electronmicroscopy investigations of recrystallization. Of particular importance is the possibility of in-situ bulk studies. By following texture changes, overall kinetics information may be achieved and synchrotron x-rays allows studies of the local structural evolution in the bulk. These in-situ investigations have given results of importance for validation and for new developments of recrystallization models. Experimental methods, results and new modelling approaches are presented and discussed.

2:30 PM Invited

Spatially Resolved Elastic Strains in Dislocation Cell Structures Measured Using Sub-Micron X-Ray Beams: *Lyle Levine*[1]; Jonathan Tischler[2]; Bennett Larson[2]; Michael Kassner[3]; Peter Geantil[3]; Wenjun Liu[4]; [1]National Institute of Standards and Technology; [2]Oak Ridge National Laboratory; [3]University of Southern California; [4]Argonne National Laboratory

X-ray line profile measurements of deformed metals have long been used to study the elastic strains (and thus stresses) within dislocation cellular structures. These conventional X-ray studies use sample volumes that are much larger than the dislocation structures being examined. Thus, only volume-averaged information can be obtained and attempts to interpret these results in terms of stress distributions within the underlying dislocation structures require ad hoc assumptions. We have used energy scanned, submicrometer X-ray beams to directly measure the axial elastic strains within individual dislocation cells in copper single crystals deformed in both tension and compression. Depth resolution was provided by translating a depth profiling wire through the diffracted beams and using triangulation to determine the depths of the diffracting volumes. More recent experiments have studied the distributions of elastic strains within individual dislocation cell walls and looked at elastic strains within extended contiguous sample volumes containing numerous dislocation cells.

2:55 PM

Study on the Texture and Micro-Structural Characters of IF Steel at Early Stage of Recrystallization by High-Energy X-Ray: *Yandong Liu*[1]; Tong He[1]; Qiwu Jiang[2]; Yang Ren[3]; Yandong Wang[1]; Gang Wang[1]; Liang Zuo[1]; [1]Northeastern University; [2]Anshan Iron and Steel Group Corporation; [3]Argonne National Laboratory

High-energy synchrotron diffraction offers great potential for experimental study of recrystallization kinetics. A fine experimental design to study the recrystallization mechanism of Interstitial Free (IF) steel was implemented in this work. In-situ annealing process of cold-rolled IF steel with 80% reduction was observed using high-energy X-ray diffraction. The results shown that, at the early stage of recrystallization, new grains with {111}<110> orientation occurred at first and {111}<112> in sequence; the a-fiber texture component decreased with the annealing temperature increased, {111}<110> texture component does not change, while {111}<112> texture component increased.

3:15 PM
The Structure Dependence of Deformation Behaviour of TRIP Steel Monitoring by In-Situ Neutron Diffraction: *Jozef Zrnik*[1]; Ondrej Muransky[2]; Peter Sittner[3]; E.C. Oliver[4]; [1]COMTES FHT Sro; [2]Nuclear Physics Institute; [3]Institute of Physics; [4]ISIS, CCLRC Rutherford Appleton Laboratory

Low alloyed TRIP steels are a class of multiphase steels offering an attractive combination of strength and ductility. By variation of thermomechanical processing parameters the transformation of conditioned austenite results in different structure characteristics. Qualitative microstructural characterization of the structure was carried out by SEM and TEM of thin foils. The volume fraction of retained austenite was measured by neutron diffraction and it varies in dependence of austenite conditioning. The tensile tests were performed at room temperature and different mechanical behaviour with different complex structure was found. The retained austenite mechanical stability during incremental loading was investigated by in-situ neutron diffraction. The results show that mechanical properties evaluated in-situ and by the standard tensile tests differ in total elongation and yield strength achieved. From evolution of phase strain was detected that the retained austenite bears a significantly larger load than the ferrite during the steel deformation.

3:35 PM
An In-Situ Investigation of Strain Partitioning and Variant-Selection Processes in Ti-6Al-4V at Elevated Temperatures Using Synchrotron Radiation: *Michael Glavicic*[1]; S.L. Semiatin[2]; Gordon Sargent[1]; [1]Air Force Research Laboratory (UES Inc.); [2]Air Force Research Laboratory

The modeling of texture evolution during the hot working of two-phase, alpha/beta titanium alloys is complicated by several factors. First, the flow-stress behaviors of the hexagonal-close-packed (hcp) alpha phase and the body-centered-cubic (bcc) beta phase differ substantially and exhibit different dependences on temperature. Such differences result in an unequal partitioning of the imposed strain. Second, the beta phase decomposes to form alpha during cooling from the hot working temperature. At moderate cooling rates, this phase transformation follows a Burgers-type orientation relationship, in which there are twelve distinct possible variants that can form from a single orientation of a prior beta-phase grain. In this work, the partitioning of strain and the predisposition for one or several of the twelve variants to form preferentially over the others during the phase transformation from beta to alpha were investigated using novel in-situ high-temperature x-ray diffraction and x-ray line-broadening techniques.

3:55 PM Break

4:05 PM
Investigation of Fatigue Crack Growth in Superalloy Single Crystals by In Situ High Brilliance X-Ray Techniques: *Liu Liu*[1]; Naji Husseini[2]; Divine Kumah[2]; Chris Torbet[1]; Roy Clarke[2]; Tresa Pollock[1]; J. Wayne Jones[1]; [1]University of Michigan, Material Science and Engineering; [2]University of Michigan, Physics

A portable ultrasonic fatigue instrument was designed and installed at the Advance Photo Source, Argonne National Laboratory to image, in situ, the initiation and propagation of fatigue cracks in the nickel-base superalloy CMSX-4 at 20 kHz with axial loading in the <001> direction. CMSX-4 specimens with 200 μm thickness have been examined. High brilliance x-ray undulator radiation was used to image (through-thickness) the initiation and growth of fatigue cracks from femtosecond laser machined notches and their interaction with the cast single crystal microstructure. Fatigue crack propagation occurred mainly on octahedral {111} planes, and for some specimens, the transition from a mixed-loading-mode crystallographic crack on an octahedral plane to a mode I non-crystallographic crack was also observed. Images of crack morphology from x-radiation are compared with topographical information obtained by post-test fractography and optical microscopy. Extensions of this work to investigate fatigue damage accumulation in complex alloys and coatings is described.

4:25 PM
Texture Development in Transformation-Induced Plasticity (TRIP) Steels Studied by Neutron and X-Ray Diffraction: *Sheng Cheng*[1]; X.-L. Wang[2]; Z. L. Feng[2]; Y. D. Wang[1]; S. Vogel[3]; J. J. Wall[1]; H. Choo[1]; P. K. Liaw[1]; X. Sun[4]; [1]University of Tennessee; [2]Oak Ridge National Laboratory; [3]Los Alamos National Laboratory; [4]Pacific Northwest National Laboratory

Complete texture studies by in situ neutron diffraction in two commercial C-Mn-Si TRIP steels with different amount of retained austenite were conducted at High-Pressure Preferred Orientation Neutron Diffractometer (HIPPO) of Los Alamos Neutron Science Center (LANSCE). The texture evolution as a function of plastic strain will be analyzed and compared with other typical steels. The effect of retained-austenite fraction on texture evolution will be discussed. The texture of these commercial TRIP steels will also be compared to that of stainless TRIP steels, and their implications will be discussed. This work was supported by the National Science Foundation Major Research Instrumentation (MRI) Program (DMR-0421219) and International Materials Institutes (IMI) Program (DMR-0231320), respectively. Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Freedom CAR and Vehicle Technologies, as part of the Automotive Light weighting Materials Program, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:45 PM
Effect of Cold-Rolling on Texture Evolution and Mechanical Properties of a Ti-IF Steel Sheet: *Yongfeng Shen*[1]; Wenying Xue[2]; Yanhui Guo[2]; Yandong Wang[1]; [1]Northeastern University, Key Laboratory for Anisotropy and Texture of Materials (Ministry of Education); [2]Northeastern University, The State Key Lab of Rolling and Automation

The dependence of crystallographic texture, deformation microstructure and flow stress on cold-rolling reductions in an interstitial-free (IF) steel sheet was investigated using X-ray diffraction (XRD), scanning electron microscope (SEM) and transmission electron microscope (TEM) techniques as well as tensile tests. The development of texture components was characterized for the specimens cold-rolled to various reductions from 13% to 75%. It was further observed that deformation microstructures consist of the geometrically necessary boundaries (GNBs) and incidental dislocation boundaries (IDBs). Dislocation and grain boundary hardening leads to an increase in flow stress with increasing rolling reduction. A fall in ductility found for the specimens with the rolling reductions between 13% and 46% is attributed to the onset of localized shearing. The increased ductility is closely related to the more uniform size distribution of cell blocks with increasing rolling reduction from 46% to 75%.

5:05 PM
In-Situ Tracing the Anomalous Recrystallization in the Ultrafine-Grained FeCo by High-Energy X-Ray Diffraction: Jianning Deng[1]; Chenchen Yuan[1]; Yang Ren[2]; Ru Lin Peng[3]; Yanling Yang[1]; Gang Wang[1]; Dezhi Zhang[4]; *Yandong Wang*[1]; [1]Northeastern University; [2]Argonne National Laboratory; [3]Linköping University; [4]Institute of Metal Research

Annealing texture in the traditional cold-rolled body-centered-cubic (BCC) metals or alloys consists mainly of {111}//ND. However, in a severely rolled FeCo alloy (with BCC structure) anomalous enhancement in the rotated-cube (100)[011] texture component and a decrease in the {111}//ND components were found after annealing, which is contrast to the recrystallization behaviors reported in traditional BCC metals. So far the intrinsic physical nature of the anomalous recrystallization behavior in this material remains mysterious in the community of textures. In this investigation, the high-energy X-ray diffraction technique was used to trace the in-situ evolution of grain size, microstrain, and texture components during recrystallization. In combination with EBSP analysis in the SEM, the physical nature is revealed and will be presented in this talk.

5:25 PM
Influence of Hydrides on Fatigue Behavior of Zircaloy-4: *Elena Garlea*[1]; Hahn Choo[1]; Gongyao Wang[1]; Peter K. Liaw[1]; Don Brown[2]; Jungwon Park[1]; Phillip Rack[1]; [1]University of Tennessee; [2]Los Alamos National Laboratory

The effect of zirconium hydrides, introduced by hydrogen-gas charging, on the mechanical behavior of Zircaloy-4 was investigated macroscopically using conventional mechanical testing and microscopically using neutron diffraction technique. Compact tension specimens were employed for which the specimen fatigue history plays a role on the distribution of hydrides and, thus, the crack propagation rate and fatigue life. It was observed that the hydrides were localized around a pre-existing crack, due to the stress gradient at the crack-tip, leading to faster crack propagation rates compared to the case when the hydrides were homogeneously spread if no flaws pre-existed in the sample. The synergetic effects of hydrides and crack tips on lattice strains of zirconium were observed by measuring the residual strain around the crack tip. The results from the hydrogenated specimens were compared with the as-received condition, allowing the evaluation of the stress produced at the crack-tip by the zirconium hydrides.

5:40 PM Invited
Novel Approaches for Non-Destructive 3D Grain Mapping: Wolfgang Ludwig[1]; *Erik Lauridsen*[2]; Søren Schmidt[2]; Henning Poulsen[2]; [1]European Synchrotron Radiation Facility; [2]Riso National Laboratory

Knowledge of the three-dimensional nature of material microstructures is of great importance for understanding, and optimization, of the microstructure – property relationship. This talk will provide an introduction to two novel approaches for 3D characterization of undeformed polycrystals. The two new approaches, termed topotomography and diffraction contrast tomography (DCT), respectively, are based on diffraction imaging using synchrotron radiation. Advantages and limitations of the two techniques are discussed as well as their relation to existing 3D x-ray based characterization techniques.

Particle Beam-Induced Radiation Effects in Materials: Ceramics and Nuclear Fuel Materials

Sponsored by: The Minerals, Metals and Materials Society, American Nuclear Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee
Program Organizers: Gary Was, University of Michigan; Stuart Maloy, Los Alamos National Laboratory; Christina Trautmann, Gesellschaft fur Schwerionenforschung; Maximo Victoria, Paul Scherrer Institute and Lawrence Livermore National Laboratory

Tuesday PM   Room: 389
March 11, 2008   Location: Ernest Morial Convention Center

Session Chairs: Jian Gan, Idaho National Laboratory; Lumin Wang, University of Michigan

2:00 PM Invited
Accumulation of Radiation Damage in Ceramic Oxides: *Lionel Thomé*[1]; Jacek Jagielski[2]; Sandra Moll[1]; Frederico Garrido[1]; [1]Nuclear Spectrometry and Mass Spectrometry Center-National Center for Scientific Research; [2]Institute of Electronic Materials Technology

Ceramic oxides are employed in hostile media where efficient use of energy is a prime need. In most of applications ceramic oxides are submitted to intense irradiations which lead to strong modifications of their physico-chemical properties. The collect and analysis of data dealing with the production and recovery of radiation damage are thus tasks of prime interest. Ion beams provide very efficient tools for the simulation of the interactions involved during the slowing-down of energetic particles. A rather broad panoply of experimental techniques (RBS/C, TEM, XRD) can be implemented to monitor the damage build-up and to characterize the nature of created defects. The aim of the present paper is to revisit the domain by the analysis of the damage accumulation in irradiated crystals in the framework of a new model (MSDA) based on the hypothesis that the damage results from multiple processes of atomic rearrangements occurring in successive steps.

2:40 PM
Bombardment of YSZ Thin Films with GeV U Ions: Alessio Lamperti[1]; Anna Paola Caricato[2]; Paolo Maria Ossi[3]; *Christina Trautmann*[4]; Lia Vanzetti[5]; [1]University of Durham; [2]Università di Lecce; [3]Politecnico di Milano; [4]Gesellschaft fur Schwerionenforschung; [5]Instituto Trentino di Cultura-Center for Scientific and Technological Research

The physico-chemical properties of Yttria Stabilised Zirconia (YSZ) make it a candidate for the containment of high activity nuclear wastes. To enhance the material stability under heavy irradiation, the evolution of its microscopic structure under extreme conditions has to be better understood. Amorphous and polycrystalline cubic YSZ thin films, about 400 nm thick, were deposited on (100) Si at RT and 673 K by UV pulsed laser ablation (248 nm; 20 ns; 4 Jcm^{-2}), in oxygen atmosphere (1 Pa). The samples were exposed to 2.6 GeV U^{+} ions, at fluences between 2 and 8×1011 cm^{-2}, completely penetrating the samples. The films were characterized, before and after irradiation, using SEM, GIXRD and XPS. In the crystalline films irradiation consistently broadens the diffraction reflections. A change is observed of Zr3d, Y3d and O1s core lines. The structural stability of polycrystalline and amorphous YSZ is discussed.

3:00 PM
Comparison of the Damage in Sapphire Due to Implantation of Boron, Nitrogen, or Iron: *Carl McHargue*[1]; Eduardo Alves[2]; Carlos Marques[2]; [1]University of Tennessee; [2]Instituto Tecnologico e Nuclear

The damage microstructure and optical properies of sapphire implanted with boron, nitrogen, and iron were examined by RBS-C, TEM, and optical absorption. Implantations were conducted at RT and 1000 C at 150 keV and fluences from 3E16 to 1E17 ion/cm^2. Optical absorption measurements indicate that the boron-implanted samples contain the higher number of F-type centers and the nitrogen-implanted samples the fewest. The microstructure of the boron-implanted samples shows only “black-spot” defect clusters, as did the iron-implanted samples at low fluences. At higher fluences, the iron samples revealed the presence of nanometer sized single crystal bcc iron particles that contributed to additional optical scattering. Bubbles formed in the nitrogen-implanted samples at low fluences of nitrogen. A second damage region is apparent in the RBS-C spectra for higher fluences of nitrogen.

3:20 PM
Radiation Induced Effects in Magnesia and Dual Phase Magnesia-Zirconia Compounds: *Hannah Yount Swoboda*[1]; Todd Allen[1]; Kurt Sickafus[2]; [1]University of Wisconsin-Madison; [2]Los Alamos National Laboratory

Magnesia and magnesia zirconia dual phase ceramics are candidates for use as inert matrix materials in advanced reactor designs. Irradiations are being performed at Los Alamos National Laboratory (LANL) and the University of Wisconsin-Madison (UW) on magnesia and magnesia-zirconia dual phase ceramics to determine their radiation resiliency. The ceramics were irradiated with heavy ions to 1 dpa and 10 dpa and with protons to 1 dpa. Nanoindentation, microhardness analysis, XRD analysis, and TEM analysis was completed to investigate changes in lattice parameters, the presence of dislocations, and phase changes. Initial XRD results show broadening of the peaks and an increase in lattice parameter of less than 1.0% after irradiation. Initial Knoop microhardness analysis indicates a decrease after irradiation from 9.8 to 9.5 GPa at 10 dpa in the dual phase ceramic and 9.0 to 7.6 GPa at 10 dpa in the MgO.

3:40 PM Break

4:00 PM
Particle Beam-Induced Radiation Effects in Strontium Titanate: *Yanwen Zhang*[1]; William Weber[1]; [1]Pacific Northwest National Laboratory

Strontium titanate ($SrTiO_3$) is of technological interest in microelectronics industries, where knowledge of particle beam-induced radiation effects, dynamic recovery and nanostructure evolution is critical. Irradiation-induced amorphization and recrystallization in $SrTiO_3$ under ion and electron irradiation have been studied in situ to characterize defects, dose rate effects, recrystallization rates and temperature dependence. The damage accumulation is consistent with a disorder accumulation model that describes interstitial and amorphous atom contributions. Significant dynamic recovery of the ion-induced damage occurs as irradiation temperature increases, and damage accumulation ratedecreases dramatically at higher temperatures. The damage accumulation kinetics is consistent with irradiation-enhanced and thermal recovery processes with activation energies of 0.1 and 0.7 eV, respectively. Fast epitaxial recrystallization occurs through regrowth at a/c interface under electron-beam irradiation, with regrowth rates several orders magnitudes higher than thermal epitaxial growth, which may be attributed to localized electronic excitations that lower the energy barriers for rearrangement of interfacial atoms.

4:20 PM
Radiation Damage in Nanocrystalline UO_2: *Tapan Desai*[1]; Dilpuneet Aidhy[2]; Paul Millett[1]; Taku Watanabe[2]; James Tulenko[2]; Dieter Wolf[1]; Simon Phillpot[2]; [1]Idaho National Laboratory; [2]University of Florida

Displacement cascades in nanocrystalline UO_2 are studied using large-scale molecular dynamics (MD) simulation. A heavy cation (Uranium) is used as a primary knock-on atom (PKA) with energies ranging from 1 keV to 20 keV. Large number of point defect formation from radiation damage in single-crystal UO_2 have been studied previously [1]. To understand the ion irradiation damage at the grain boundaries, a nanocrystalline material consisting of three dimensional grains of 10-40 nm diameter size is used. Due to the radiation, we expect large-scale defect formation in the grains followed by segregation of these defects to the grain boundaries. We compare the effects of different interatomic potentials for the description of the interactions in UO_2 on the defect evolution. The effect of the temperature on the defect formation/diffusion is also characterized. [1]L.V. Brutzel, M. Rarivamanantsoa and D. Ghaleb, Journal of Nuclear Materials, 354, (2006), 28.

4:40 PM
Charged Particle Beam Irradiation Creep Behavior of Pyrolytic Carbon: *Anne Davis*[1]; Zhijie Jiao[1]; Rongsheng Zhou[1]; Gary Was[1]; Lumin Wang[1]; [1]University of Michigan

The creep behavior of pyrolytic carbon (PyC) in TRISO fuel particles under high temperature and neutron irradiation is critical to the fuel integrity of the Very High Temperature Reactor. To determine the irradiation creep behavior of PyC, experiments were conducted on thin (<70 µm) strip samples using a 3 MeV proton beam. The samples were loaded to stresses of 20.9 and 14.2 MPa at temperatures between 800 and 1200°C. The specially-designed irradiation stage in the Tandetron accelerator at the Michigan Ion Beam Laboratory includes a laser speckle extensometer for determining sample extension and a two-dimensional thermal imager for temperature control. The temperature dependence of the creep rate under proton irradiation is compared to that under neutron irradiation at similar temperatures and loads. The relation between the creep behavior and the irradiated microstructure will also be discussed.

5:00 PM
Study of Silver Diffusion in Silicon Carbide: *Erich Friedland*[1]; Johan Malherbe[1]; Nick van der Berg[1]; [1]University of Pretoria

Fuel elements of modern high-temperature nuclear reactors are encapsulated by CVD-layers of pyrolitic carbon and silicon carbide to reduce fission product release. Release of Ag-110m presents a major problem as the transport mechanism through polycrystalline silicon carbide is not well understood. The aim of this study is to obtain information on the importance of volume and grain boundary diffusion as well as their dependence on radiation damage. For this purpose silver diffusion in single crystal and in polycrystalline silicon carbide is compared at temperatures up to 2000 K. The contribution of grain boundary diffusion is extracted by correlating the diffusion results for single- and polycrystalline samples with the distributions of grain sizes and boundary thicknesses of the latter samples obtained by SEM analyses. Diffusion coefficients are obtained from the broadening of implantation profiles using RBS analysis. Different implantation temperatures are used to study the effect of radiation damage.

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials VII: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Sinn-wen Chen, National Tsing Hua University; Srinivas Chada, Medtronic; Chih Ming Chen, National Chung-Hsing University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-Ho Lee, Hong Ik University; Kejun Zeng, Texas Instruments Inc; Katsuaki Suganuma, Osaka University

Tuesday PM — Room: 278
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Chih Ming Chen, National Chung-Hsing University; Daniel Lewis, Rensselaer Polytechnic Institute

2:00 PM Invited
Pronounced Electromigration of Cu in Molten Sn-Based Solders: J. Huang[1]; *C Robert Kao*[1]; [1]National Taiwan University

The high local temperature in flip-chip solder joints of microprocessors has raised concerns that the solder, a low melting temperature alloy, might locally liquefy and consequently cause failure of the microprocessors. This presentation reports a highly interesting electromigration behavior when the solder is in the molten state. A 6.3x103 A/cm2 electron current was applied to molten Sn3.5Ag solder at 255°C through two Cu electrodes. The high current density caused the rapid dissolution of the Cu cathode. The dissolved Cu atoms were driven by electrons to the anode side and precipitated out as a thick and sometimes continuous layer of Cu6Sn5. The applied current caused the dissolution rate of the Cu cathode to increase by one order of magnitude. An equation for the minimum chemical potential gradient that is needed to balance the electromigration flux is derived.

2:20 PM
Measurement of Electromigration Activation Energy of the Eutectic SnPb Flip-Chip Solder Joints on Cu/Ni Thick-Film UBM Using Kelvin Probes: *TsungHsien Chiang*[1]; Chih Chen[1]; [1]National Chiao Tung University

Activation energy of electromigration is investigated using Kelvin probes. The eutectic SnPb flip-chip solder joints with thick under bump metallization (UBM) of 5-µm Cu/3-µm Ni has been adopted for the electromigration test. We fabricate the Kelvin probes to monitor the bump resistance during the current stressing, and define the electromigration failure as the bump resistance increase reaches 20% of its initial value. Most of the previous studies defined the failure of the electromigration when the stressing circuit was open. Three-dimensional (3D) finite element modeling with void formation was established to compare with the experiment results to confirm the increase of bump resistance. The stressing conditions were done on a hotplate with temperatures at 160°C, 150°C, and 140°C, and the current was 0.9 A. Also, the failure mode and the variation of the microstructure during various stages of electromigration will be discussed.

2:35 PM
Effect of Electromigration on the Mechanical Properties of Sn-3.5Ag Solder Joints with Ni and Ni-P Metallization: *Zhong Chen*[1]; Aditya Kumar[2]; Chee Cheong Wong[1]; Subodh Mhaisalkar[1]; Vaidhyanathan Kripesh[3]; [1]Nanyang Technological University; [2]Nanyang Technological University/Institute of Microelectronics; [3]Institute of Microelectronics

The effect of moderate electric current density (1 x 10^3 to 3 x 10^3 A/cm^2) on the mechanical properties of Ni-P/Sn-3.5Ag and Ni/Sn-3.5Ag solder joints were investigated after thermal aging at 160 degree for 100 hours. It was found that aged samples without current stressing failed mostly inside the bulk solder with significant plastic deformation in a tensile. The passage of current caused brittle failure to occur at the joint interface, and the tendency of such brittle failure increases with increasing current density. The fracture energy in the brittle facture decreased more rapidly with the Ni joint than the Ni-P joint. The IMC growth rate was also affected more in the Ni joint than the Ni-P joint.

2:50 PM
Recrystallization Induced Microstructure Change in Eutectic SnPb Flip Chip Solder Joints by Electromigration: *Fan-Yi Ouyang*[1]; K. Tu[1]; [1]University of California, Los Angeles

We observed frequently a refinement of the lamellar microstructure in eutectic SnPb solder joints in electromigration. We propose that the refinement is a consequence of recrystallization of the solder joint. Electromigration has both atomic flux and electron flow. The latter generates joule heating and the former causes strain. A strained solid at high temperature will undergo recrystallization. Since the eutectic solder is a two-phase alloy, a fine lamellar microstructure can be created, provided that the strain energy is much higher than the total interfacial energy. We calculated that the strain energy induced by electromigration can be three times of magnitude larger than the interfacial energy. Thus, recrystallization of a fine lamellar structure can occur in eutectic SnPb solder joints driven by electromigration.

3:05 PM
Study on the Electromigration Behavior in Sn-37Pb Flip Chip Solder Joint: *Sang-Su Ha*[1]; Jong-Woong Kim[1]; Seung-Boo Jung[1]; [1]Sungkyunkwan University

Electromigrtion of solder joint under high current stressing has been an important concern of the reliabilities in the solder joint system, since the required device density and power of flip chip package increase. Thus, the size of the bumps shrinks continuously, causing rapid increase in the current density passing through the bumps. Recently, flip chip solder joint, when the current density reached the range of 104A/cm[2], drift of metal atoms occurred seriously, causing dissolution of under bump metallization at the cathode and the accumulation of the IMCs at the anode, meanwhile the current crowing accelerated at migration process. During the high current stressing in the solder joints, the effect of current crowing and Joule heating has been found to be responsible for the failure in the chip/anode side of the solder joints. In this study, electromigration behavior in Sn-37Pb solder bumps have been investigated for flip chip structure.

3:20 PM
Dual-Mode Failure Mechanisms of Electromigration in Pb-Free Snag Solder Joints: *Hsiao-Yun Chen*[1]; Chih Chen[1]; [1]National Chiao Tung University

The effect of under bump metallization on electromigration failure mechanism has been investigated for eutectic SnAg composition under 9 x 10^3 A/cm2 at 150°C. The thickness of the Cu UBM is 5 µm while the metallization on the substrate is 5 µm electroless Ni. Failure analysis revealed that Cu consumption in flip-chip solder joint structures plays important role in the failure mechanism. It is found that Cu would migrate to substrate end thus caused UBM depletion on the chip side regardless of the current direction, causing the intermetallic compounds spalled into solder. On the contrary, there would be no UBM depletion under current stressing for solder joints with a 5-µm Cu/3-µm Ni UBM. Typical pancake type void occurred when electron flow from chip side to substrate, while there is void formation near substrate with electron flow upward. More details on the failure mechanism will be discussed in the presentation.

3:35 PM Break

3:50 PM Invited
Undercooling of Pb-Free Solders on Various UBMs: *Moon Gi Cho*[1]; Sung Kang[2]; Hyuck Mo Lee[1]; [1]Korea Advanced Institute of Science and Technology; [2]IBM Corp

The undercooling behavior of pure Sn, Sn-0.7Cu, Sn-3.5Ag and Sn-3.8Ag-0.7Cu solder alloys was observed in terms of various under bump metallurgies (UBMs). Four different UBMs (electroplated Cu, electroplated Ni, electroless Ni-P and electroless Ni-P/immersion Au) were employed. The amount of the undercooling of Pb-free solder alloys were reduced when reacted with Cu UBM and Ni-based UBMs, and the Ni-based UBMs was more effective than Cu UBM. When Ni_3Sn_4 was formed in the interfacial reactions with Ni-based UBMs, the reduction of undercooling was significant, especially for pure Sn and Sn-3.5Ag. The effects of UBMs on the undercooing of Pb-free solder alloys are discussed by comparing intermetallic compounds (IMCs) as well as the compositional change of each solder before and after interfacial reaction with UBMs. In addition, the microstructural changes of four solders on UBMs are discussed, which could be related to their undercooling behaviors.

4:10 PM Invited
Microstructural Evolution of Alloy Powder for Electronic Materials with Liquid Miscibility Gap: Ikuo Ohnuma[1]; Takuro Saegusa[1]; Yoshikazu Takaku[1]; Ryosuke Kainuma[1]; *Kiyohito Ishida*[1]; [1]Tohoku University Graduate School of Engineering

Alloy powders manufactured from immiscible melts by a rapid quenching method exhibit characteristic morphologies, a so-called in situ composite, such as the egg-type, uniform second-phase dispersion microstructures reported by C.P. Wang et al., Science, 297 (2002), 990-993. In this paper, the microstructural evolution of rapidly quenched alloy powders, which are applicable for novel electronic materials, will be discussed based on a two-liquid miscibility gap predicted in the calculated phase diagrams using a thermodynamic database, ADAMIS (Alloy Database for Micro-Solders). Specifically, the microstructural evolution and properties of Bi-base Pb-free solders for high temperature use and Ag- or Au-base micro-powders with a high electric conductivity produced by a gas-atomizing method will be presented.

4:30 PM
Growth of SnPb Composite Whisker under Electrical Current Stressing: *Cheng-Chang Wei*[1]; Pei Chiung Liu[1]; Chih Chen[1]; King Ning Tu[2]; [1]National Chiao Tung University; [2]University of California, Los Angeles

The spontaneous growth of whiskers has become the most serious reliability problems in Pb-free electronic package industry. According to the previous literature, the optimum growth temperature for whisker is 60°C. And, it is hard to grow Sn whiskers in SnPb solder, since Pb can mitigate the growth of the Sn whiskers. In this study, composite SnPb whiskers were found to grow from the eutectic SnPb solder stripes. By employing FIB, solder stripes of various lengths, including 5µm, 10µm, 15µm and 270µm can be fabricated. Above the high current density of 5x104 A/cm2 at 100°C, the high compressive stress gradient exists in the SnPb solder and urges the composite whiskers formation. Pb-rich-phase whiskers grew first, followed by the growth of the Sn-rich-phase whiskers. According to the diffraction pattern of the Pb part in the composite whisker, the growth orientation was identified [110], [1-11] and [112].

4:45 PM
Improvement on Thermal Fatigue Properties of Sn-Ag-Cu Lead-Free Solder Interconnects on Casio's Wafer Level Packages Based on Morphology and Grain Boundary Character Distribution Changes: *Shinichi Terashima*[1]; Taro Kohno[2]; Aiko Mizusawa[3]; Kazuyoshi Arai[3]; Osamu Okada[3]; Takeshi Wakabayashi[3]; Masamoto Tanaka[1]; Kohei Tatsumi[1]; [1]Nippon Steel Corporation; [2]Ome Bumping Service Center, Nippon Steel Materials Corporation; [3]Casio Computer Corporation

It has been well known that fatigue properties of lead-free Sn-Ag-Cu solder interconnects are affected by their silver contents; lower silver contents (ex. Sn-1Ag-0.5Cu) show better reliability at mechanically shock test than those with higher silver (ex. Sn-3Ag-0.5Cu) by absorbing shock energy, but have shorter thermal fatigue lives due to tin-grain coarsening caused by strain-induced dynamic recrystallization. In this presentation, thermal fatigue properties of commercial low-silver solder, LF35 (based on Sn-1.2Ag-0.5Cu), are evaluated as compared with those of both low (Sn-1Ag-0.5Cu) and high silver (Sn-3Ag-0.5Cu) solders on Casio's wafer level packages. It is clarified that thermal fatigue properties of LF35 was remarkably improved from that of Sn-1Ag-0.5Cu, and moreover, LF35 showed longer fatigue life than Sn-3Ag-0.5Cu in spite of its lower silver content. This improvement can be explained from the viewpoints of both morphology and grain boundary character distribution changes.

5:00 PM
Mechanical and Electrical Properties of Cu-Sn Intermetallic Micro-Wire: *Luhua Xu*[1]; King-Ning Tu[1]; [1]University of California, Los Angeles

The size of solder joint in flip chip solder is becoming smaller, with a typical diameter of 50-100 micron. In through wafer vias, the diameter of copper interconnect and solder joint are as low as 20 micron. Solder and copper pad may rapidly react during both solder reflow and solid state aging. As a result, a high portion of intermetallic occupies in the small solder joint. It can even become a pure intermetallic joint. In this work, Cu-Sn intermetallic micro-wires were prepared by 100 micron copper wires reflowed with SnAgCu solder paste and followed by high temperature thermal annealing. Uni-axial tensile test on the intermetallic(Cu6Sn5+Cu3Sn) showed the combined elastic modulus is 98

GPa, which is in agreement with previous result measured by nano-indentation. The resistivity of the intermetallic wire was determined by four probe method. Electromigration test on the Cu-IMC-Cu micro-wire showed a re-distribution of Cu6Sn5 and Cu3Sn.

5:15 PM
Effect of Co Addition on Undercooling, Microstructures and Microhardness of Sn-3.5Ag Solder and Interfacial Reactions with Ni-P UBM: *Dong Hoon Kim*[1]; Moon Gi Cho[1]; Hyuck Mo Lee[1]; [1]Korea Advanced Institute of Science and Technology

The effect of Co addition, from 0.01 to 0.7%, on undercooling, microstructure and microhardness of Sn-3.5Ag solder (number are all in wt.%) and interfacial reactions with Ni-P UBM are investigated. For addition of higher than 0.02%, the undercooling of Sn-3.5Ag solders was significantly reduced together with coarsened microstructures and increased eutectic regions. The hardness value also increased with increased alloying amount. The interfacial reaction with Ni-P UBM showed that spalling of intermetallic compounds (IMCs) during reflow was prevented in the Sn-3.5Ag-xCo (x=0.02%). And, the addition of higher than 0.05% Co changed the morphology of IMC from bulky (Ni_3Sn_4) to needle-shape (Ni-Sn-Co ternary compound). The optimum level of Co addition is also discussed.

Recycling: Micro-Organisms for Metal Recovery

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Christina Meskers, Delft University of Technology; Greg Krumdick, Argonne National Laboratory

Tuesday PM — Room: 280
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Christina Meskers, Delft University of Technology; Joseph Pomykala, Argonne National Laboratory

2:00 PM
Chromate Reduction by a Novel Leucobacter Sp. Isolated from Chromite Ore Processing Residue Disposal Site of China: Zemin Ma[1]; Wenjie Zhu[1]; *Liyuan Chai*[1]; [1]Central South University

A novel chromate [Cr(VI)] reducing bacterium was isolated from chromite ore processing residue disposal site of China and identified as a Leucobacter sp. by 16S rRNA gene sequence homology. The minimum inhibitory concentration of strain CRB1 was 820 mg l-1 of Cr(VI). The reduction of Cr(VI) occurred only under aerobic conditions. With an initial concentration of 2.5×108 cells ml-1, 1800 mg l-1 of Cr(VI) was reduced entirely within 38 h under the optimum conditions, at 30°C and pH 9.0. Increasing Cr(VI) concentrations prolonged reduction process and higher cell densities meant quicker reduction. A blue precipitate was produced during chromate reduction of strain CRB1, which was binding to the surface of bacterial cells and determined as trivalent chromium with electron paramagnetic resonance (EPR) spectrometry. The conspicuous chromate reduction ability of Leucobacter sp. CRB1 suggested a potential for bioremediation of Cr(VI) containing wastes.

2:20 PM
Detoxification of Chromite Ore Processing Residue with Novel Chromate Reducing Bacteria Leucobacter Sp. Ch1: Wenjie Zhu[1]; *Liyuan Chai*[1]; Zhihui Yang[1]; [1]Central South University

Chromite ore processing residue (COPR) is a hazardous waste containing high concentration of chromium. We describe here a pioneer trial of detoxification of COPR in column reactor with an indigenous chromate reducing bacterium Leucobacter sp. Ch1, which showed high chromate reducing ability at high pH. Under the optimum conditions, at 30 °C and pH 10.0, pH of leachate decreased from 10.3 to 8.3, and Cr(VI) concentration in solution decreased to zero mg L-1, and 76.4% of Cr(VI) was removed from COPR samples. The leaching toxicity of detoxified residue was 1.1 mg L-1 that was lower than national standard of China.

2:40 PM
Effects of Cr(VI) on the Population of Soil Microorganisms: ShunHong Huang[1]; Zhihui Yang[1]; Peng Bing[1]; Liyuang Chai[1]; Licheng Zhou[1]; *Qing-hua Tian*[1]; [1]Central South University

To improve understanding the effect of heavy metal chromium on soil biota, laboratory incubation experiment was carried out in the soils contaminated artificially with five concentrations of chromate (50, 250, 500, 1000 and 2000 mg kg^{-1} soil) over time (35 days). The population and diversity of bacteria, fungi and actinomycetes in soils were measured as indicators of soil microbial processes in the disturbances mentioned above. Incubation experiment results elucidated that the population and diversity of microorganisms decreased with increasing Cr (VI) concentration. Furthermore, various microorganisms showed different sensitivity to total Cr contamination. The sensitivity of microbial population to chromium contamination was in the order: actinomycetes >bacteria>fungi. The resulted implied that the microorganism can be used as an indicator of chromium contamination.

3:00 PM
Influence of Cr (VI) on the Activity of Soil Enzymes: ShunHong Huang[1]; Zhihui Yang[1]; Bing Peng[1]; Liyuan Chai[1]; *Qing-hua Tian*[1]; [1]Central South University

Soil microorgainisms are sensitive to ambient environment and the changes of enzyme activities can indicate the pollution severity in soils. Field samples and incubation experiments were used for investigating the effects of Cr (VI) on enzyme activities in soils. Soil samples were taken from chromium slag site and nearby region in XiangXiang Iron-Alloy factory in Hunan province. The results showed that the effect of Cr (VI) on activities of catalase, polyphenol oxidase and alkaline phostphaste was not significantly distinct. However, Cr (VI) showed a significantly inhibitory effect on dehydrogenase activity. Its value decreased with the increasing Cr (ε) concentration. The result imply that of dehydrogenase activity could be used as an indicator for the chromium pollution level in the area of Iron-Alloy factory and for the stability level of the soil ecosystem.

Structural Aluminides for Elevated Temperature Applications: Processing and Microstructure Control

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Young-Won Kim, UES Inc; David Morris, Centro Nacional de Investigaciones Metalurgicas, CSIC; Rui Yang, Chinese Academy of Sciences; Christoph Leyens, Technical University of Brandenburg at Cottbus

Tuesday PM — Room: 394
March 11, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Kouichi Maruyama, Tohoku University; Birgit Skrotzki, BAM

2:00 PM Invited
Phase Transformations Studies of TiAl Based Alloys Solidifying through the β Phase: *Marc Thomas*[1]; [1]ONERA

Following alloy development programs undertaken by ONERA with the aim to reduce the L->α columnar as-cast structure, a new β-solidification TiAl (G4) alloy has been developed as a casting material with equiaxed microstructure for high temperature gas turbine applications. Since then, such a multi-phase material was found to induce a limited influence of aluminium content on mechanical properties. Further microscopic insights were however needed for a better understanding in the phase transformations mechanisms for such β solidification alloys. This paper highlights what are the structural factors, either related to the β solidification or to the subsequent cooling, that can be used to identify the precise step reactions from the liquid for this category of TiAl-based alloys. Then, emphasis is given to the various solid-state transformations involving the β phase such as β->γ, β->α2 and β->ω, those depending on the alloy chemistry and the heat treatment conditions.

2:30 PM
Recent Development in Gamma Titanium Aluminides Forging: Jacques Tschofen[1]; *Nicolas Rizzi*[1]; [1]Manoir Aerospace -Forges de Bologne

The gamma TiAl alloys present interesting properties to replace Ni base alloys for either rotanional or hot components. They exhibit high temperature properties up to approx. 750°C combined with a low density. This study presents recent work in forging of several grades. It shows difficulty to get good forgings and gives trends to have a more reliable forging route.

2:50 PM
Near Net Shape Manufacturing with Gamma Titanium Aluminide: *Edward Shelton*[1]; A. Wisbey[1]; J.W. Brooks[1]; [1]QinetiQ

Alloys based on the intermetallic phase gamma titanium aluminide, TiAl, could replace other metallic materials in a range of applications. The principal benefits associated with TiAl are its low density and excellent properties at elevated temperatures. Commercial exploitation of the alloys has been restricted by technical difficulties and high costs associated with manufacturing components using conventional processing routes. The potential to produce components from TiAl alloys by thixoforming has been investigated. This process produces near net shape components by forcing the alloy into a mould while partially molten and is a well-developed technology applied to lower melting point alloys. There are significant technological difficulties to be overcome in developing it for TiAl alloys with melting points in excess of 1400°C. Process development studies are reported. Experimental trials have produced thixoformed specimen components from a TiAl-based alloy. Fundamental microstructural and mechanical properties of this thixoformed TiAl are reported.

3:10 PM
Towards the Thermodynamic Equilibrium of Titanium Aluminides after Consolidation by Back Pressure Equal Channel Angular Pressing: Ross Whitfield[1]; *Klaus-Dieter Liss*[1]; Wei Xu[2]; Thomas Buslaps[3]; LaReine Yeoh[1]; Xiaolin Wu[2]; Kenong Xia[2]; [1]Australian Nuclear Science and Technology Organisation; [2]University of Melbourne; [3]European Synchrotron Radiation Facility

Mixtures of 50-Al and 50-Ti powders (atomic %) have been consolidated using the Back Pressure Equal Channel Angular Pressing (BP-ECAP) method starting with both raw and ball milled powders. We present in-situ high-energy X-ray diffraction studies with continuous Rietveld analysis obtained upon a heating ramp from 300 K to 1075 K performed after the consolidation process. Initial phase distributions contain all intermetallic compounds of this system but Al, with distribution maxima in the outer regions of the concentrations (α-Ti, $TiAl_3$). Upon annealing, we follow in detail the phase evolution and lattice parameter changes due to chemical segregation which is in favor for the more equilibrated phases such as γ-TiAl, α_2-Ti_3Al and $TiAl_2$. An unexpected phase transition at about 625 K upon heating creates an intermediate β-Ti phase which gradually transforms into the final products.

3:30 PM Break

3:40 PM Invited
Processing and Properties of Gamma TiAl Sheet from Atomised Powder: *Rui Yang*[1]; Lei Xu[1]; Yuyou Cui[1]; Wei Sun[1]; Chunguang Bai[1]; Dong Liu[1]; [1]Shenyang National Laboratory for Materials Science, Institute of Metal Research CAS

Sheets made of light high-temperature materials such as gamma TiAl alloys have a variety of applications in aerospace structures. The compositional homogeneity of preforms inherent in the powder metallurgy approach helps to ensure uniform microstructure and mechanical properties of the sheet, especially at large dimension. This paper summarises our successful effort of sheet rolling using atomised powder of gamma alloys. Emphasis will be placed upon impurity pick-up and control of the atomised powder, the influence of degasing/HIPing parameters on the soundness of the preform, as well as identification of optimal combinations of rolling parameters. Temperature distribution in the preform at different stage of the rolling operation was accurately simulated using a finite element method. Thermomechanical deformation map of the gamma alloys obtained on a Gleeble simulator proves to be an excellent guide to identifying the "processing window" in which sheets with satisfactory mechanical properties can be produced.

4:10 PM
Solidification of Nb-Rich TiAl-Based Alloys: Grain Refinement by Boron Additions and the Role of Peritectic Growth: *Ulrike Hecht*[1]; Victor Witusiewicz[1]; Anne Drevermann[1]; [1]Access Materials and Processes

Boron is an effective grain refiner for TiAl-based alloys, however its beneficial effect is sensitively depending on alloy composition and solidification conditions. In this paper we present a comprehensive analysis of the grain refinement mechanism and its limits, based on thermodynamic and kinetic aspects of solidification and subsequent phase transformations: The analysis is based on unidirectional solidification experiments performed on samples with different composition, selected from within the range 43 to 46 at.% Al and 5 to 8 at.% Nb, both with and without boron additions. An adequate quenching technique allowed us to conserve high temperature phases, specifically the alpha-(Ti) phase, in all solidified samples. A clear distinction between peritectic alpha-(Ti) and alpha-(Ti) grown from solid state beta-(Ti) was possible. EBSD analysis of the grain orientation of alpha-(Ti) was directly feasible and proved to be the key for understanding grain refinement.

4:30 PM
Grain Refinement in TiAl- Based Materials Investigated by Directional Growth Experiments and Phase-Field Simulations: *Ingo Steinbach*[1]; Ulrike Hecht[1]; Anne Drevermann[1]; Janin Eiken[1]; Christian Hartig[2]; Daniel Gosslar[2]; Robert Guenther[2]; Ruediger Bormann[2]; [1]Rheinisch-Westfälische Technische Hochschule-Aachen; [2]Technische Universität Hamburg-Harburg

Grain refinement of TiAl-based alloys can be achieved by thermal treatment or during solidification, the latter being commonly attributed to the effect of boron additions. The detailed mechanism of grain refinement by boron is however a subject of controversial discussion in literature, especially with regard to the role of boride phases on the nucleation of beta(Ti) and on nucleation the of peritectic alpha(Ti). In this talk we present phase field simulations and well controlled directional growth experiments that focus on nucleation and subsequent growth of primary beta(Ti) and peritectic alpha(Ti) in the presence of third phases that may act as heterogeneous nucleation sites for either beta(Ti) or alpha(Ti). The microstructure evolution is compared to that of a reference TiAl-based alloy that does not contain, or form, third phases.

4:50 PM
Texture Formation in High Niobium Containing γ-TiAl Alloys during Hot Rolling: *Andreas Stark*[1]; Arno Bartels[1]; Daniel Gosslar[1]; Frank-Peter Schimansky[2]; Rainer Gerling[2]; Helmut Clemens[3]; [1]Hamburg University of Technology; [2]GKSS Research Centre; [3]Montanuniversität Leoben

The texture formation during hot rolling was studied in Nb-rich γ-TiAl based alloys with compositions of Ti-45Al-(5-10)Nb-(0-0.5)C (at.%). Hot rolling is performed with texture-free powder compacts at temperatures in the upper range of the (α+γ) phase field. Additionally a third phase, β-Ti(Al), is present at that temperatures in Ti-45Al-10Nb. Samples for texture analysis were taken after various pass sequences of the rolling process as well as after additionally annealing. In the as-rolled samples the textures of γ-TiAl and α-Ti(Al) show components which are crystallographically correlated by the Blackburn orientation relationship. However this correlation is not caused by phase transformation but indicates co-deformation during hot rolling. For the first time we could measure the texture of β-phase in Ti-45Al-10Nb which exhibits components, typically observed in bcc-metals after rolling. In addition, the high number of slip systems which can be active in bcc β-Ti(Al) results in improved formability during the rolling process.

5:10 PM
Development of TiAl-Based Alloys for High Temperature Applications: *Fereshteh Ebrahimi*[1]; Sonalika Goyel[1]; Michael Kesler[1]; Orlando Rios[1]; Hans Seifert[2]; [1]University of Florida; [2]Technische University Bergakademie

Polycrystalline gamma-TiAl has shown promising properties for high temperature applications below 1000C. Precipitation of a second intermetallic phase can be exploited to extend its applicability to higher temperatures. Furthermore, since TiAl has limited workability, it is desirable to have a high temperature cubic phase, which upon cooling transforms to the aluminide phase. We have investigated additions of Nb and Cr to identify a compositional region that satisfies the above goals. Arc melted buttons using pure elements were prepared. Phase evolution in the alloys was evaluated by examination of samples heat treated in purified argon atmosphere and then quenched from critical temperatures identified by DSC experiments. Selected alloys were aged to study the phase stability at elevated temperatures. In this presentation the extension of the cubic beta phase field and the evolution of the two-phase gamma-TiAl and sigma-Nb2Al will be discussed. Financial support through NSF-DMR 0605702 grant is greatly appreciated.

Ultrafine-Grained Materials: Fifth International Symposium: Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Yuri Estrin, Monash University and CSIRO Melbourne; Terence Langdon, University of Southern California; Terry Lowe, Los Alamos National Laboratory; Xiaozhou Liao, University of Sydney; Zhiwei Shan, Hysitron Inc; Ruslan Valiev, UFA State Aviation Technical University; Yuntian Zhu, North Carolina State University

Tuesday PM Room: 395/396
March 11, 2008 Location: Ernest Morial Convention Center

Session Chairs: Terence Langdon, University of Southern California; Sergey Dobatkin, Russian Academy of Sciences, A.A.Baikov Institute of Metallurgy and Materials Science; Terry Lowe, Los Alamos National Laboratory; Karl Theodore Hartwig, Texas A&M University

2:00 PM Invited
Functional Properties Produced by Severe Plastic Deformation: *Zenji Horita*[1]; [1]Kyushu University

Severe plastic deformation (SPD) through Equal-Channel Angular Pressing (ECAP) is applied to a Cu-6.5mass%Co alloy containing a fine dispersion of ferromagnetic Co precipitates in the Cu matrix. Changes in microstructures and crystal structures are examined using X-ray diffraction (XRD) analysis and transmission electron microscopy (TEM). Measurements of coercive force are carried out after ECAP with different numbers of passes and different processing routes. It is shown that the application of ECAP leads to an increase in the coercive force and intouduces magnetic anisotropy. SPD through High-Pressure Torsion (HPT) is further applied to intermetallic $MgNi_2$ powders and the hydrogen storage capability of the intermetallic is examined. Quantitative analysis of hydrogen content including microstructural analyses using XRD and TEM show that HPT is effective to modify a non-hydrogen absorber, $MgNi_2$, to a hydrogen absorber without addition of ternary elements.

2:20 PM
Significant Size Effect in the Mechanical Behavior of Nanostructured Copper: Yonghao Zhao[1]; Cheng Xu[2]; Terence Langdon[2]; *Yuntian Zhu*[3]; [1]University of California, Davis; [2]University of Southern California; [3]North Carolina State University, Department of Materials Science and Engineering

Nanostructured materials are usually characterized with small samples. Here we report that small sample sizes have unexpected size effects on the mechanical behavior, including on the ductility and work-hardening rate. Specifically, in addition to the size effect normally expected from the cross-section and gauge length, we demonstrate also that the gauge length significantly affects the strain hardening rate and the uniform elongation. The implications of these observations suggest that some of the literature data on mechanical behavior of nanostructured materials, especially the strain hardening rate and uniform ductility, may be partially incorrect. Also, the strain rate sensitivity may be affected by the sample size, casting some doubts on fundamental properties derived from this parameter such as the activation volume. In summary, the mechanical properties of nanostructured materials are not easily compared unless the sample sizes are the same.

2:35 PM Invited
Common Trends in Flow Anisotropy and Fatigue Response of Ultra-Fine Grained HCP and BCC Materials: *Ibrahim Karaman*[1]; M. Haouaoui[1]; G.G. Yapici[1]; H.J. Maier[2]; I. Beyerlein[3]; C.N. Tome[3]; [1]Texas A&M University; [2]University of Paderborn; [3]Los Alamos National Laboratory

Recent findings show that large strains and abrupt strain path changes alter the microstructure and crystallographic texture of HCP and BCC materials, leading to considerable flow stress anisotropy. We have recently conducted an extensive investigation on the effect of strain path changes of a few HCP and BCC materials during and after severe plastic deformation on the resulting uniaxial (tensile and compressive) flow anisotropy and low cycle fatigue (LCF) response. In particular, we have used Equal Channel Angular Extrusion (ECAE) to severely process: 1) commercial purity (CP) HCP Ti; 2) pure HCP Zr billets with strong basal texture; 3) Nb-1%Zr BCC alloy; and 4) interstitial free (IF) BCC steel, most of them processed at room temperature. A Visco-Plastic Self Consistent (VPSC) polycrystal plasticity model was used to predict the flow behavior and texture evolution after post-processing uniaxial experiments. In this talk, we will summarize the common trends and subtle differences on the texture evolution, flow anisotropy, and LCF response in these materials while also elaborating on the microstructural mechanisms responsible for these phenomena.

2:55 PM
Superplastic Deformation of Ultrafine-Grained Mg-Alloys Produced by Micro-Alloying and Equal Channel Angular Extrusion: *Florian Dalla Torre*[1]; Anja Hänzi[1]; Maciej Krystian[2]; Jörg Löffler[1]; Peter Uggowitzer[1]; [1]Swiss Federal Institute of Technology; [2]ARC Seibersdorf Research GmbH

The 'grain growth restriction'-concept via the addition of elements such as Zn, Y, Ca, and Zr was used to cast and extrude Mg-alloys with mean grain sizes of < 10 micrometer. In combination with equal-channel-angular-extrusion (ECAE) processing, the mean grain size was further reduced to 200-300 nm. Tensile testing at room temperature of samples prior to ECAE showed a substantial increase in ductility and strength. After ECAE the yield strength increased further to values of 300 to 400 MPa, while maintaining a high strain to failure of 15-28%. Texture and mechanical anisotropy decreases with decreasing grain size. Above 250°C superplastic deformation with strains larger than 100% and with strain-rate-sensitivities of up to 0.6 were measured. These properties, combined with the stabilization of the grain size in the micrometer range, are very beneficial for superplastic net-shape-forming. In addition, their good biocompatibility makes these alloys potential candidates for biomedical applications.

3:10 PM Invited
Low-Temperature Superplastic Flow of Ultrafine Ti-6Al-4V: *S. Semiatin*[1]; Gordon Sargent[2]; [1]Air Force Research Laboratory; [2]UES, Inc

The superplastic-flow behavior at low temperatures (650 – 800°C) and a range of strain rates (10-4 to 10-2 s-1) of Ti-6Al-4V with an ultrafine microstructure has been established. Program materials comprised both billet product manufactured by warm isothermal ('abc') forging and sheet fabricated by warm rolling. Extensive metallography on undeformed and deformed samples water quenched from the various test temperatures was conducted to characterize microstructure stability. Despite the low deformation temperatures, both lots of material showed similar (measurable) dynamic coarsening, whose kinetics mirrored the flow-hardening observed during compression and tension tests. The plastic-flow phenomenology was interpreted in the context of the classical Bird-Mukherjee-Dorn relation. The stress and grain-size exponents of the strain rate were ~1.4-2 and ~2, respectively, for strain rates of 10-4 and 10-3 s-1. The constitutive analysis suggested that multiple mechanisms (dislocation glide-climb, diffusion) control deformation, thus complicating the interpretation of the apparent activation energy.

3:30 PM Invited
Quasistatic and High-Strain Rate Response of Ultra-Fine Grained Copper: *Marc Meyers*[1]; A. Mishra[1]; B. Kad[1]; M. Martin[1]; N. Thadhani[1]; [1]University of California

The quasistatic and high-strain rate mechanical response of UFG copper processed by ECAP was characterized by compressive, tensile, reverse Taylor impact and Hopkinson-bar experiments. Upon impact, the samples were found to undergo heat-induced static recrystallization at a calculated temperature of 360K, indicating that the UFG copper is thermally unstable. Reverse Taylor tests were conducted on as-received OFHC Cu rod and ECAP specimens with sequential ECAP passes (2 and 8). The dynamic deformation of the samples is modeled using AUTODYN-2D and a modified Johnson-Cook constitutive equation was found to well capture the dynamic response. Both the dynamic experiments and analysis from the Reverse Taylor tests indicate an enhanced strain-rate sensitivity in comparison with conventional polycrystalline copper, in agreement with predictions of reduced activation volume. Similar to the compression test results, the impacted front of the samples are found to recrystallize extensively and preferentially.

3:50 PM Break

4:05 PM Invited
Towards Enhancement of Fatigue of Ultra-Fine Grain Metals - Past and Future: *Alexei Vinogradov*[1]; Satoshi Hashimoto[1]; [1]Osaka City University

Common aspects and features of fatigue of UFG materials produced by ECAP are discussed. Special attention is paid to the influence of strain path, amount of strain and post-processing annealing on the structure, strength and ductility, fatigue life and cyclic hardening/softening. Available results on the grain size effect on high-cyclic and low-cyclic properties are reviewed aiming at understanding of the reasons for materials degradation. Mechanisms of plastic flow and degradation during fatigue are discussed from the standpoint of initial UFG structure and its evolution upon cycling. The role of dislocation accumulation and grain reduction is highlighted. It is shown that high fatigue limit can be achieved after first ECA-pressings if a uniform fine grain structure is formed during ECAP and post-processing annealing. Results of phenomenological modeling of the monotonic and cyclic response of UFG metals are presented in terms of dislocation kinetics and a satisfactory agreement with experiments is demonstrated.

4:25 PM
Mechanical and Service Properties of Nano- and Submicrocrystalline Low Carbon Steels at Temperatures between (-40) and (+700)°C: *Sergey Dobatkin*[1]; Pavel Odessky[1]; Evgeny Naidenkin[1]; Svetlana Shagalina[1]; [1]Russian Academy of Sciences, A.A.Baikov Institute of Metallurgy and Materials Science

The thermal stability, mechanical and service properties of low-carbon ferritic-pearlitic and martensitic 0.09%C-Mn-Si, 0.1%C-Mn-V-Ti and 0.06%C-Mo-Nb-V steels have been studied after severe plastic deformation (SPD) by high-pressure torsion (HPT) and equal-channel angular pressing (ECAP). HPT and ECAP of low carbon steels produce nanocrystalline (50-100nm) and submicrocrystalline (SMC) (200-300nm) structures, respectively. The initial martensitic state compared to the initial ferritic-pearlitic state of low carbon steels promotes the formation of finer structure, less intense grain growth upon heating after SPD up to 700°C and more significant strengthening. The high-strength state of the 0.1%C-Mn-V-Ti and 0.06%C-Mo-Nb-V steels after ECAP is retained up to a temperature of 500°C. The strength properties at 600°C (fire resistance) of these SMC steels are higher by 20-25% than those of the undeformed steels. A significant increase in impact toughness especially at subzero temperatures is shown. The work was supported by RFBR (project no. 07-03-00342).

4:40 PM Invited
The Effect of ECAP with Back-Pressure on Enhanced Superplasticity of Magnesium Alloy AZ31: *Rimma Lapovok*[1]; Michael Popov[2]; Stuart Rundell[1]; Yuri Estrin[1]; [1]Monash University; [2]Technical University of Clausthal

Enhanced superplastic behaviour of magnesium alloy AZ31 was studied as a function of the number of ECAP passes and the level of back-pressure. After homogenisation at 420°C for 4 hours the processing by ECAP with a back-pressure leads to microstructures more favorable for enhanced superplasticity, notably bi-modal grain structures. The role of back-pressure and temperature for obtaining bi-modality of microstructure has been studied. It was shown that a decrease in the temperature of ECAP processing and concurrent increase of the back-pressure result in a bi-modal structure which consequently leads to an increase in elongation-to-failure to a record level as compared to the reported results for this material. Samples processed by six passes of ECAP at 150°C and the applied back-pressure of 218 MPa demonstrated enhanced superplastic behaviour with maximum strain-to-failure above 1200%, which is by more than 300% higher than the best results published on AZ31 superplasticity to date.

5:00 PM
The Flow Behavior of Superplastic Materials Processed Using Severe Plastic Deformation: Megumi Kawasaki[1]; *Terence Langdon*[1]; [1]University of Southern California

When metallic alloys are processed using severe plastic deformation, the grain sizes are reduced to the submicrometer range and the materials exhibit exceptional properties including superplastic flow at elevated temperatures. There are many examples to date documenting the occurrence of superplastic behavior in materials processed by procedures such as equal-channel angular pressing. This presentation examines the characteristics of this behavior with special reference to a two-phase Zn-22% Al eutectoid alloy. Emphasis is placed on the variation of the flow stress with strain rate, measurements of the strain rate sensitivity and the role and significance of internal cavitation.

5:15 PM
Ductility Enhancement of Ultrafine Grained Aluminium Processed by Constrained Groove Pressing: *Jozef Zrnik*[1]; Tomas Kovarik[2]; Miroslav Cieslar[3]; Libor Kraus,[4]; [1]COMTES FHT Sro; [2]West Bohemian University; [3]Charles University; [4]Comtes FHT, Ltd.

The constrained groove pressing was used to refine pure aluminium coarse structure. The cumulative effective strain eeff becomes ~ 9.3. The impact of the groove pressing is investigated upon microstructure changes with transmission electron microscopy. The coarse grain structure is successfully refined to the mixture of subgrain and polygonized grains of submicron size. However, the structure nonhomogeneity is apparent regardless the number of passes performed. The changes in mechanical properties were evaluated by performing tensile tests and by hardness measurement. The substantial impact of straining upon the tensile strength increase was already observed after the first pressings. With increasing straining only small enhancement in strength and concomitant loss of ductility was apparent. To improve plastic properties of ultrafine grained Al a further annealing and deformation experiment was carried out. The results from tensile tests indicate the small decrease in YS and UTS but increase in the elongation.

5:30 PM
Mechanical Properties of Ultrafine Grained Ferritic Steel Sheets Fabricated by a New Process without Severe Plastic Deformation: *Yoshitaka Okitsu*[1]; Naoki Takata[2]; Nobuhiro Tsuji[2]; [1]Honda R&D Company, Ltd.; [2]Osaka University

It's been difficult to apply UFG metals to automobile body parts, because dimensions of the samples fabricated by SPD methods are limited. Recently the authors have developed a new process to produce UFG steel sheets through conventional rolling and annealing procedures. The purpose of this presentation is to show the process and the mechanical properties of the prototyped steels. A low-carbon hot-rolled steels composed of ferrite and martensite were cold-rolled by 90% reduction, and annealed in a salt bath set at 600°C to 700°C. The final microstructure consisted of sub-micrometer ferrite and fine cementite, and the ferrite grain size changed from 0.5µm to 1µm by changing the annealing temperature. The difference in flow stress between dynamic and quasi-static deformation of the obtained UFG steel was much larger than that of conventional high strength steels. It was concluded that the UFG steel has high potential of crash energy absorption.

5:45 PM
Enhancement of Fatigue Properties in Ultrafine-Grained Titanium Rods Produced by Means of Severe Plastic Deformation: *Irina Semenova*[1]; Gulnaz Salimgareeva[1]; Vladimir Latysh[1]; Terry Lowe[2]; Ruslan Valiev[1]; [1]Ufa State Aviation Technical University; [2]Los Alamos National Laboratory

Our investigations are focused on the development of pilot technology to fabricate long-length ultrafine-grained (UFG) titanium rods. The technology combines equal channel angular pressing (ECAP) for the formation of UFG structure with additional thermomechanical treatments to further increase strength and produce rods of sufficient length for subsequent automated machining and forging of UFG titanium products. Among the set of properties fundamentally important for these products is fatigue resistance. This work summarizes experimental measurements of the fatigue properties of long-length rods of Grade 4 titanium. Fatigue tests were performed on smooth and notched samples. The formation of homogeneous UFG structures in commercial purity Grade 4 titanium rods is shown to enhance high cycle fatigue strength by 50%, exceeding the fatigue strength of Ti-6Al-4V. Additional experiments were conducted to evaluate how low-temperature annealing can further increase fatigue and other properties of UFG titanium.

Ultrafine-Grained Materials: Fifth International Symposium: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Yuri Estrin, Monash University and CSIRO Melbourne; Terence Langdon, University of Southern California; Terry Lowe, Los Alamos National Laboratory; Xiaozhou Liao, University of Sydney; Zhiwei Shan, Hysitron Inc; Ruslan Valiev, UFA State Aviation Technical University; Yuntian Zhu, North Carolina State University

Tuesday, 6:30-8:00 PM Room: 395/396
March 11, 2008 Location: Ernest Morial Convention Center

Cold and Hot Severe Plastic Deformation of an Al-3wt%.Mg Alloy: *Martin Hafok*[1]; Reinhard Pippan[1]; [1]Erich Schmid Institute of Materials Science

In order to examine the effect of deformation temperature on materials processed by high pressure torsion, an Al-3wt.%Mg alloy was chosen to analyse the evolution of microstructure and microtexture. By using a single phase aluminium alloy instead of pure aluminium the static recrystallization and recovery, which may occur in pure aluminium even at room temperature, is suppressed after quenching the sample from elevated temperatures or after heating the sample to room temperature from cryogenic temperatures. The characteristic development of microstructure and microtexture of the alloy deformed in a temperature range between -196°C and 450°C were investigated by SEM, EBSD and TEM technique in order to reveal the transition between cold working, dynamic recovery and dynamic recrystallization during HPT. In the experiments a characteristic microtexture and microstructure was found which is associated with the torsion deformation, the dynamic recovery and the dynamic recrystallization.

Continuous Equal-Channel Angular Pressing for Producing Long Nanostructured Ti Semi-Products: *Georgy Raab*[1]; Yuntian Zhu[2]; Terry Lowe[2]; Ruslan Valiev[1]; [1]Ufa State Aviation Technical University; [2]Los Alamos National Laboratory

We have analysized major factors that influence the formation of ultrafine-grained structure and evaluated the effectiveness of a continuous equal-channel angular pressing (ECAP) method, ECAP-Conform, for producing nanostructured CP-Ti long rods. In this work we have built a pilot ECAP-Conform machine to process rods with length up to 3 m and diameter up to 7.5 mm. The nanostructured Ti rods produced here have superior strength and fatigue properties, which is of great interest to advanced medical applications in stomatology and traumatology.

Contribution of Texture and Grain Size to Hall-Petch Relation of a Wrought Magnesium Alloy AZ31: Jun Tao[1]; *Jingtao Wang*[1]; Deliang Yin[1]; Jinqiang Liu[1]; [1]Nanjing University of Science and Technology, Department of Materials Science and Engineering

Because of the mixed contribution from both texture and grain size to mechanical properties of wrought Mg alloys, there was a confusion on the slope of Hall-Petch relation of wrought magnesium alloys, especially in those processed by equal channel angular pressing (ECAP). The experiment is designed in this investigation so that the effect of texture and grain size on yield strength of an AZ31 alloy could be clearly separated. It is concluded that Hall-Petch relation holds valid for the effect of grain size on yield strength in the experimental alloy, as long as no significant difference in the texture of the samples with different grain size. The effect of crystallographic texture on yield strength manifests itself by the different interception on stress axis of Hall-Petch lines with significant difference in texture intensity.

Corrosion and Fretting Wear Behaviour on UFG Ti-13Nb-13Zr Alloy in Ringer's Solution: Anbarasan Viswanathan[1]; *Geetha Manivasagam*[1]; C. Richard[2]; Sathyam Suwas[3]; R. Asokamani[1]; C. Kowandy[2]; J. Landoulsi[2]; [1]Vellore Institute of Technology-University; [2]Université de Technologie de Compiègne; [3]Indian Institute of Science

Corrosion and wear are the prime consideration for a biomaterial that is to be used in the human body, because metal ion released mainly associated with toxicity of surgical implants and can adversely affect the biocompatibility and mechanical integrity. Earlier works clearly indicate that refinement of grain size may enhance the corrosion and mechanical properties in comparison with coarse grain (CG) counterparts. Hence in this work we have attempted to study the corrosion and fretting wear behavior of ultra fine grain (UFG) Ti-13Nb-13Zr developed by Equal channel angular pressing (ECAP) process in simulated body fluid (Ringer's solution). Potentiodynamic anodic polarization and open circuit potential were used to evaluate the corrosion behavior of UFG Ti-13Nb-13Zr in simulated body fluid at 37ºC. In addition, fretting wear behavior of UFG Ti-13Nb-13Zr alloys against bearing steel is also evaluated. The results of the corrosion and fretting wear studies will be presented in this paper.

Crack Growth in Ultrafine-Grained AA6063T6 Produced by Equal Channel Angular Pressing: Lothar W. Meyer[1]; *Kristin Sommer*[1]; Thorsten Halle[1]; Matthias Hockauf[1]; [1]Chemnitz University of Technology

Crack growth behaviour of ultrafine-grained AA6063T6, processed by equal channel angular pressing (ECAP) via route E at room temperature, were evaluated with special emphasis on the effect of grainsize distribution and work hardening. A bimodal, two times ECAPed state and a monomodal ultrafine-grained state after eight ECA-extrusions are compared with the coarse grained initial T6 state. Crack growth behaviour is investigated by using SE(N)B-specimens and described by a Paris-Erdogan-Ratwani-equation, covering crack growth behaviour from the threshold region up to the high rate region approaching the critical stress intensity. Depending on the number of ECA-extrusions, the ECAPed material shows significantly lower threshold values (ΔK_{th}) and higher crack growth rates (da/dN) than its coarse grained counterpart. SEM micrographs of crack propagation surfaces reveal reduced grain size as major key to increased crack growth rates of the ECAPed material, as it influences roughness-induced crack closure and crack deflections.

Creep Response and Deformation Processes in Nanocluster Strengthened Ferritic Steels: Taisuke Hayashi[1]; *Peter Sarosi*[1]; Joachim Schneibel[2]; Michael Mills[1]; [1]Ohio State University; [2]Oak Ridge National Laboratory

Mechanically alloyed Oxide Dispersion Strengthened (MA/ODS) ferritic alloys are considered as candidate structural materials for fission and fusion power plants applications because of their excellent creep strength at high temperature and good resistance to irradiation-induced swelling. An alloy designated 14YWT containing 100nm sized grains and nanometer sized clusters (Y and Ti containing oxides) was subjected to high temperature annealing followed by creep at 800°C. Microstructural characterization using scanning and transmission electron microscopy to understand the microstructure stability during annealing as well as the deformation processes during creep of the MA/ODS steel was performed. A detailed examination of the grain growth, nanocluster distribution and dislocation analysis will be discussed.

Deformation and Fracture of AZ31 Magnesium Alloy during Equal Channel Angular Pressing: Feng Kang[1]; *Jing Tao Wang*[1]; Yong Peng[1]; [1]Nanjing University of Science and Technology

The deformation and fracture characteristics of AZ31 magnesium alloy during equal channel angular pressing (ECAP) were established. The isothermal behavior of AZ31 magnesium alloy was determined at temperatures between 150 and 250°C and ram speeds producing average effective strain rates between 0.001 and 0.25s-1. AZ31 magnesium alloy was particularly susceptible to shear localization during ECAP, uniform flow occurred only at high temperatures and low strain rates. Observations of shear banding and shear fracture were interpreted in terms of the tendency for strain concentration as quantified by the flow localization parameter, or the ratio of the normalized flow softening rate to the strain-rate sensitivity. These understandings of the effect of material properties on flow localization tendency are helpful for the selection of processing parameters with uniform flow during ECAP.

Deformation Behavior of Nanocrystalline Metals and Alloys Investigated by Mini-Tensile Test: *Lilia Kurmanaeva*[1]; Yulia Ivanisenko[1]; Jörg Weissmüller[1]; Jürgen Markmann[2]; Ruslan Valiev[3]; Hans-Jörg Fecht[4]; [1]Forschungszentrum Karlsruhe GmbH; [2]Universität des Saarlandes; [3]Ufa State Aviation Technical University; [4]University of Ulm

The recent past has seen an increasing interest in studies of mechanical properties of nanocrystalline materials (nc). NC materials offer wide application as structural materials thanks to their outstanding mechanical properties. A novel method for the preparation of bulk nanocrystalline materials with a grain size <30 nm using the combination of inert gas condensation and subsequent high pressure torsion was developed. Here, we present results on a comprehensive investigation of the microstructure and mechanical properties of nanocrystalline metals and alloys, namely Pd and Pd-Au, with a mean grain size of 15 nm. Microstructure was investigated by Transmission Electron Microscope (TEM). Mechanical properties of the obtained specimens were studied in tensile test using a dedicated tensile machine for miniature specimens. It was shown that nanocrystalline Pd and Pd-Au alloy exhibits a very high yield stress and a microhardness with sufficient ductility. The obtained results of mechanical properties and microstructure are discussed.

Direct Metal Particulate Production Using Modulation-Assisted Machining: *James Mann*[1]; Chris Saldana[1]; Srinivasan Chandrasekar[1]; W. Compton[1]; Kevin Trumble[1]; [1]Purdue University

Continuous production of Al 6061-T6 particulate using modulation-assisted machining (MAM) is demonstrated. Superimposition of a controlled, low-frequency modulation in conventional machining causes chips to form as discrete particles. By adjusting the conditions, equiaxed, platelet, and fiber shaped particles having narrow size distributions can be produced. Large-strain deformation leads to microstructure refinement and enhanced hardness. The process is applicable to a wide range of alloys and appears to be intrinsically scalable for large-volume production.

Effect of Initial Microstructure on Strain Hardening Behavior of Ultrafine Grain Dual-Phase Steel Produced by Severe Plastic Deformation: *Young Gun Ko*[1]; Dong Hyuk Shin[2]; [1]Massachusetts Institute of Technology; [2]Hanyang University

Strain hardening behavior of ultrafine grained (UFG) dual phase (DP) steels via equal channel angular (ECA) pressing and subsequent intercritical annealing followed by water quenching was investigated at ambient temperature. A series of tensile tests were carried out for three distinct DP steels, i.e., CG-DP, UFG-DP and UFG-DPV steels. In contrast to conventional UFG steels with a lack of strain hardening, UFG-DPV steel showed significant strain hardening rate, resulting from uniform distribution of island-typed martensite as well as grain refinement of each constituent phase throughout the microstructure. Also, UFG-DPV steel containing 0.06% of vanadium exhibited the good combination of high strength and sufficient strain hardening, because initial microstructure of that was consisted of the fine martensite through equal channel angular pressing. Strain hardening behavior of these steels was discussed in relation to modified C-Janalysis based on Swift relationship.

Effect of Strain Rate, and Deformation Temperature on the Microstructure of an Equal Channel Angular Extrusion (ECAE) Processed Ti-6Al-4V Alloy: *Rabindra Mahapatra*[1]; Shankar Sastry[2]; [1]Naval Air Systems Command; [2]Washington University

The Ti-6Al-4V alloy was ECAE processed to produce ultra-fine grains of 1-2µm. The uni-axial compression experiments to simulate forging parameters subsequent to ECAE processing were carried out to elucidate whether, the ultra-fine grained microstructure of the alloy can be sustained after the deformation. The ECAE processed Ti-6Al-4V alloy showed no significant grain growth when deformed at 750°C, at a strain rate of 0.1”/sec., where as when deformed at same temperature, a strain rate of 0.001”/sec., both the recovery and grain growth were observed.

Effect of Subsequent Annealing Treatment on Dynamic Deformation Behavior of Ultrafine Grain Al-Mg Alloy: *Young Gun Ko*[1]; Yang Gon Kim[2]; Dong Hyuk Shin[3]; Sunghak Lee[2]; Chong Soo Lee[2]; [1]Massachusetts Institute of Technology; [2]Pohang University of Science and Technology; [3]Hanyang University

A study was made to investigate the dynamic deformation behavior of ultrafine grain Al-Mg alloy with subsequent annealing treatments at ambient temperature. By imposing an effective strain up to 8 via equal-channel angular pressing (ECAP), most grains were refined from to 300 nm with a non-equilibrium nature of grain boundaries. Upon several subsequent annealing treatments, the ultrafine microstructure was stable at temperature up to 473 K, whist normal grain growth was found to take place above 523 K. In order to understand the effect of annealed microstructure (ultrafine grain with unstable grain boundary vs. fine grain with stable grain boundary) on dynamic deformation behavior, torsional tests were carried out for four samples i.e., as-ECAPed and 473, 523 and 573 K after ECAP process, using a Kolsky bar. Such mechanical response was discussed in relation to microstructure, tensile property and fracture mode associated with the occurrence of adiabatic shear bands.

Evolution of Microstructure and Property of a Pure Iron during Equal Channel Angular Pressing: Yue Zhang[1]; Jingtao Wang[1]; *Songming Wang*[1]; [1]Nanjing University of Science and Technology

Equal channel angular pressing (ECAP) was conducted on a pure iron up to 8 passes via route A and Bc. Evolution of microstructure and mechanical property during ECAP were characterized by tensile testing and TEM observation. While strong thin lamellae structure with an average thickness of 0.2~0.4 micrometer were observed after 8 pass of ECAP via route A, as one expected; similar structure is also observed, mixed with equiaxed fine grain structures in the sample after 8 pass of ECAP via route Bc. Immediate necking after yielding is observed in tensile test of all the samples after ECAP, although an obvious plastic deformation is observed before failure. A high tensile strength of ~880MPa could be achieved through ECAP by both route A and Bc.

Flow Properties of an Aluminum Alloy Processed by Equal Channel Angular Pressing: *Sivaraman Arjunan*[1]; Uday Chakkingal[1]; [1]Indian Institute of Technology Madras

Equal Channel Angular Pressing (ECAP) process is an important process for producing ultra fine grained microstructures in bulk metals and alloys. The microstructures developed after ECAP represent high energy configurations; hence they are inherently unstable and susceptible to flow softening depending upon strain paths employed in subsequent deformation. In the present work aluminum alloy AA 6063 samples were subjected to ECA pressing for up to three passes with a die angle of 105 degree. Compression testing was used to determine the subsequent flow behaviour. Two types of compression test specimen orientations; one parallel to the axis of pressed sample and the other at 45 degree to the axis of the pressed sample were used for the study. The flow curves were plotted and comparative flow properties, flow softening and anisotropic behavior have been studied with respect to number of passes and processing routes. These are correlated to the observed microstructures.

Forging Parameter Effects on the Mechanical Behavior of Cryomilled Al 5083: *Troy Topping*[1]; Byungmin Ahn[2]; A. Newbery[3]; Enrique Lavernia[1]; [1]University of California, Davis; [2]University of Southern California; [3]MillStrong Ultra, LLC

Aluminum alloys with nanocrystalline (NC) and ultra-fine grain (UFG) size are of immense interest because of their high strength – typically 30% stronger than conventionally processed alloys of the same composition. For this study, the microstructure and mechanical behavior of UFG Al 5083 plate, produced by the quasi-isostatic forging of cryomilled powder, has been investigated and compared to course-grained Al 5083 - in particular, the ability to strengthen the UFG material further by tuning the forging parameters. Experimental forging parameters were used on six plates with approximate dimensions of 10” in diameter and 0.75” in thickness. The effort focuses on increasing strength through reduced grain growth during processing, while maintaining ductility by breaking up prior particle boundaries (PPBs) with high forging pressures. Mechanical tests reveal increased strength in proportion to decreased grain growth, while ductility is maintained at the level of conventional alloys.

Grain Growth in Ultrafinegrained Aluminium Processed by Hydrostatic Extrusion: *Malgorzata Lewandowska*[1]; Tomasz Wejrzanowski[1]; Krzysztof Kurzydlowski[1]; [1]Warsaw University of Technology

Ultrafinegrained materials may be produced by a number of techniques involving severe plastic deformation. These materials are generally thermally unstable and undergo grain growth at elevated temperature driven by a large surface area of grain boundaries. The kinetics of growth at early stage of the process (in fine grain size range) depends on microstructural features, such as a high fraction of high angle grain boundaries and grain size uniformity. This implies a correlation between the SPD technique used for grain refinement and thermal stability of processed materials. In the present work, the changes in grain size and grain boundary characteristics during annealing at various temperatures were evaluated quantitatively for technically pure aluminium processed by hydrostatic extrusion. The experimental kinetics of grain growth have been analyzed in terms of the influence of initial fraction of high angle grain boundaries and grain size homogeneity and by simulation based on Monte Carlo method.

Improvement in Both Strength and Ductility in Nanostructured Carbon Steel Produced by High Pressure Torsion and Annealing: Shaohua Xia[1]; Lilia Vichikganina[2]; *Jingtao Wang*[1]; Igor Alexandrov[2]; Ruslan Valiev[2]; [1]Nanjing University of Science and Technology; [2]Ufa State Aviation Technology University

Nanostructured carbon steels of Fe-0.45%C and Fe-0.65%C were obtained by high pressure torsion and annealing. Combination of high strength and improved uniform elongation was achieved in Fe-0.45%C resulted from formation of bimodal distribution in grain size. By comparing the microstructures and mechanical properties between nanostructured Fe-0.45%C and Fe-0.65%C, we can conclude that in-situ formed micro-meter sized grains in ferrite phase during annealing provide extra strain hardening ability to sustain the uniform elongation as their coarse-grained counterpart. Estimation of the relationship between volume fraction of micro-meter sized grains and mechanical properties in bimodal steel was also made by associated analysis of results from previous investigations in low carbon steel processed by equal channel angular pressing and annealing.

Improving the Superplastic Properties of an AZ31 Magnesium Alloy by Equal-Channel Angular Pressing: *Roberto Figueiredo*[1]; Terence Langdon[1]; [1]University of Southern California

The interest in improving the properties of magnesium alloys has led to the development of technology to refine the grain structure especially through the use of severe plastic deformation. In the present paper, extruded billets of a commercial alloy, AZ31, were processed by Equal-Channel Angular Pressing (ECAP). Optical microscopy and tensile tests were used to evaluate the grain structure and high temperature mechanical properties in order to determine the effect of processing by ECAP. An analysis of grain structure shows that ECAP effectively reduces the average grain size to the range of a few micrometers. High temperature tensile tests demonstrate that ECAP reduces the flow stress of the AZ31 alloy and improves the strain rate sensitivity and the elongation to failure. Superplastic elongations up to more than 1000% were achieved after ECAP with low flow stresses and high strain rate sensitivity.

In-Situ Observation of Deformation of Nanocrystalline Al-Mg Alloy with Bimodal Grain Structure: *Byungmin Ahn*[1]; Enrique Lavernia[2]; Steven Nutt[1]; [1]University of Southern California; [2]University of California, Davis

The tensile properties and deformation response of nanocrystalline Al-Mg alloy (Al 5083) were investigated using a micro-straining unit. Atomized Al 5083 powder was ball-milled in liquid N2 to obtain a nanocrystalline structure, and blended with 15% unmilled coarse-grained powder to achieve bimodal structure. The blended powder was hot vacuum degassed to remove residual contaminants, consolidated by either cold (CIP) or hot isostatic pressing (HIP), and then forged. The microstructure was observed using an optical microscope. The investigation of tensile and fracture of bimodal structure suggests unusual deformation mechanisms and interactions between ductile coarse-grain bands and nanocrystalline regions.

Influence of Phase Separation on the Mechanical Properties of Pre-Oxidized PM 2000 Alloy: *Carlos Capdevila-Montes*[1]; Michael Miller[2]; Jesus Chao[1]; Jose Gonzalez-Carrasco[1]; [1]Centro Nacional de Investigacines Metalurgicas (CENIM-CSIC); [2]Oak Ridge National Laboratory

In the last few years, the ultrafine-grained Fe-based oxide dispersion strengthened (ODS) PM 2000 alloy has been shown to be a viable biomaterial as result of its outstanding combination of mechanical properties and corrosion resistance. After pre-oxidation at 1100°C and phase separation upon aging at 475°C, the room temperature tensile and fatigue properties are suitable for achieving the required biofunctionality for load-bearing implants. Atom probe tomography has revealed that phase separation into Fe-rich alpha and Cr-enriched alpha-prime phases is responsible for the increases in the yield and ultimate tensile strength. However, despite the loss of some ductility, PM 2000 shows ductile behaviour in the necked zone of tensile specimens. This ductility contrasts with the brittle failure observed during the so-called "475°C embrittlement" of other ferritic alloys. Moreover, PM 2000 aged at 475°C exhibits a higher fatigue limit than that of unaged pre-oxidised material.

Influence of Rolling Direction on Strength and Ductility of Aluminium and Aluminium Alloys Produced by Accumulative Roll Bonding: *Irena Topic*[1]; Heinz Werner Höppel[1]; Mathias Göken[1]; [1]Friedrich-Alexander University of Erlangen Nürnberg

The accumulative roll bonding (ARB) process has been recognised as a successful severe plastic deformation method for production of ultrafine-grained (UFG) materials with superior mechanical properties compared to their conventionally grained counterparts. The biggest advantage of this process is that it can be easily adapted in industry to produce large scale UFG sheets, especially interesting for light weight construction in automotive industry due to high potential of cost reduction and energy savings. In this work, accumulative roll bonded AA1050 and AA6016 showed significantly increased specific strength paired with high ductility. Despite a strongly elongated grain structure, tensile testing of samples oriented 45° to the rolling direction (RD) revealed considerable improvement in elongation to failure compared to the samples parallel to RD. Hydraulic bulge testing showed a tendency to higher achievable burst pressures and strains indicating good formability. Friction stir welding proved to be a successful method for UFG sheet material.

Influence of SPD on the Magnetic Properties of Soft Magnetic Materials: Stephan Scheriau[1]; Klemens Rumpf[2]; Siegfried Kleber[3]; Heinz Krenn[2]; Reinhard Pippan[1]; *Martin Hafok*[1]; [1]Erich Schmid Institute of Material Science; [2]Karl-Franzens-University Graz; [3]Böhler Edelstahl GmbH

Industrial available FeSi, FeCo and FeNi alloys with an initial grain size of 20-50µm were subjected to Severe Plastic Deformation (SPD) by high pressure torsion at both ambient temperature (293 K) and liquid nitrogen temperature (77 K). The strain levels were chosen in that way where a saturation of the microstructural refinement is observed. The microstructure of the severely deformed states is analysed by Back Scattered Electrons (BSE) micrographs captured in a SEM. Additionally samples that were deformed at 77 K are examined in a Transmission Electron Microscope (TEM). The magnetic properties were characterised by means of SQUID-magnetography providing information of the magnetic behaviour of the material in the as processed state. Depending on the deformation temperature the mean microstructural sizes in the SPD-state are about 120nm and 50nm at 293 K and 77 K, respectively. The microstructural size influences significantly the magnetic properties of these materials. The initial soft-magnetic behaviour of the coarse grained state shifts towards a hard-magnetic with decreasing crystallite size. By dropping below a crystallite size of ~50nm the magnetic properties become again soft-magnetic.

Mechanical Properties and Corrosion Behavior of Ultrafine-Grained AA6082 Produced by Equal Channel Angular Pressing: *Matthias Hockauf*[1]; Lothar W. Meyer[1]; Daniela Nickel[1]; Gert Alisch[1]; Thomas Lampke[1]; Bernhard Wielage[1]; Lutz Krüger[2]; [1]Chemnitz University of Technology; [2]Technische Universität Bergakademie Freiberg

The mechanical properties and corrosion behaviour of AA6082 with ultrafine-grained (UFG) microstructure are investigated and compared with the coarse grained (CG) AA6056 with much higher Cu-content that has been increasingly used for automotive applications. The AA6082 was processed by equal channel angular pressing (ECAP) up to eight extrusions at room temperature in a die with an internal angle of 90° following Route E with active backpressure. Besides the peak-aged temper, which gave maximum strengths and strongly reduced ductility, the solution heat treated condition was considered as well. Combined with post-ECAP ageing, an optimum of high strength, ductility and toughness was achieved. Polarisation tests showed a slightly more positive corrosion potential for the UFG-conditions compared to the CG counterparts. The results indicate that UFG low-Cu Al-alloys like AA6082 are capable to replace Cu-containing Al-alloys like AA6056 for applications in automotive industry, for example in high strength screws.

Mechanical Properties of Ultra Fine Grained Aluminum and Iron Produced by Accumulative Roll Bonding Method: *Saeed Tamimi*[1]; Mostafa Ketabchi[1]; Nader Parvin[1]; [1]Tehran Polytechnic University

This work aims to investigate whether accumulative roll bonding (ARB) is an effective grain refinement technique for ultra-low-carbon steel strips containing 0.002% C and pour aluminum. For this purpose, a number of ARB processes were performed at 500°C for IF and 200°C for pour aluminum, with 50% reduction of each rolling pass. The mechanical properties after rolling were obtained. Aluminum and iron's yield and tensile strengths increased by 200–300%. Variations of hardness along the thickness of the samples were obtained using micro hardness tests. It was found that both the grain size achieved, as well as the degree of bonding, depend on number of rolling pass and reduction of area as a whole. In IF steel, mean grain size was obtained about 300nm. The rolling process was stopped in 7th cycle for pour aluminum and 10th cycle for IF steel, when cracking of the edges became pronounced.

Mechanisms of Deformation and Refinement of Grains in Metals by Severe Plastic Deformation: *Georgy Raab*[1]; Farid Utyashev[2]; [1]Ufa State Aviation Technical University; [2]Russian Academy of Sciences, Institute for Metals Superplasticity Problems

Grains refinement is considered as an effect of mechanisms of fragments boundaries and bands formation as a result of crystallographic and non-crystallographic shears at bending and/or torsion of a sample. During ECAP and HPT the sample's bending-torsion in a localized deformation center grows and that leads to a non-monotonic deformation and an increase in the angular misorientations of boundaries of the formed bands and fragments. It was shown that the depth of structure refinement depends on the contributions of the required mechanisms in total strain that depend, in their turn, on a scale factor – the dimensions of the deformation center and sample.

Microstructural Characteristics of Leadframe Cu Alloys after Accumulative Roll Bonding Process: *Chayong Lim*[1]; Seungzeon Han[1]; Seonghee Lee[2]; [1]Korea Institute of Materials Science; [2]Mokpo National University

Mechanical properties and formation of nano-sized grains in Cu and Cu-Fe-P alloys by accumulative roll bonding (ARB) process were investigated. Nano-sized grains were successfully obtained in OFC and PMC-90 alloys by ARB process after third cycle. Once the 200 nm grains formed, further reduction in the grain size was not observed up to the 8 ARB process cycles. For both alloys, the tensile strength values increased drastically in the initial stage of ARB process. The tensile strength values of both alloys tended to saturate after the third ARB process cycle. The tensile elongation value greatly decreased by 1 cycle of ARB process due to the strain hardening. After the third cycle of ARB process, each alloy showed a gradual increase in tensile elongation due to the dynamic recovery. For PMC-90 alloy, the strength value is higher than that of OFC due to addition of the alloying elements.

Microstructure and Mechanical Properties of ECAE Processed Two-Phase Zinc-Aluminum (Zn-8%Al) Alloy: *Majid Al-Maharbi*[1]; Mohammed Haouaoui[1]; Ibrahim Karaman[1]; Gencaga Purcek[2]; [1]Texas A&M University; [2]Karadeniz Technical University

A two-phase zinc-aluminum alloy (Zn-8%Al) has been subjected to severe plastic deformation by equal channel angular extrusion (ECAE). The alloy was successfully extruded at homologous temperatures of 0.52 to 0.55 through different strain paths. The as-cast dendritic structure was eliminated and the α-phase particles were fragmented and dispersed more uniformly into the η-matrix leading to a more homogeneous microstructure. TEM micrographs show that grain sizes were significantly reduced to the ultrafine grain (UFG) sizes. An average increase in strength and elongation of about 53% and 1420% of as-cast values was achieved. Noticeable softening was observed after the first pass, and was attributed to chemical composition homogenization occurring with increasing strain level. The homogenization is assisted by the simultaneous long range diffusion and the refinement of the two phases as revealed by EDS and microhardness measurements which show considerable changes in composition and properties of the individual phases after ECAE.

Microstructure and Mechanical Properties of Nanocrystalline Fe-C Alloys Processed by Mechanical Milling and Spark Plasma Sintering: *Keiichiro Ohishi*[1]; Bonta Rao[2]; Kazuhiro Hono[1]; [1]National Institute for Materials Science; [2]University of Tsukuba

Bulk nanocrystalline Fe-C alloys containing different carbon contents were fabricated by mechanical milling and spark plasma sintering (SPS). The samples consolidated by SPS at temperatures in the range of 650-675°C exhibited a good combination of strength and plastic strain in compression. The values of yield and maximum compressive strengths increased with carbon content, while the plastic strains decreased. The microstructure has been characterized using transmission electron microscopy (TEM) and a three-dimensional atom probe (3DAP) to understand the origin of the unusually high yield strength and plastic strain. A bimodal grain structure consisting of fine and coarse grains was observed for all the samples showing high strength and plastic strain. The fine grained region was found to be a duplex phase structure comprised of ferrite and cementite grains. From TEM and 3DAP analyses, the presence of fine oxide particles containing chromium was confirmed.

Microstructure and Mechanical Properties of Nanostructured Metal Matrix Composites: *Timothy Lin*[1]; Fei Zhou[1]; Quan Yan[1]; Chunfu Tan[1]; Bob Liu[1]; Adolphus McDonald[2]; [1]Aegis Technology Inc.; [2]U.S. Army Aviation and Missile Command

Over last few years Al-based nanostructured metal matrix composites (NMMCs) has attracted increasing interests because of their great potentials in strength enhancement. Presently Aegis Technology is developing a novel class of NMMCs funded by U.S. Army Small Business Innovative Research (SBIR) project, "Light-weight Material for Ballistic Armor". This class of NMMCs, which is based on submicron SiC particulates reinforced nanostructured Al alloy matrix, can be used for not only lightweight armors but also lightweight structures (e.g. anti-wear engine components). Aegis has successfully developed a cost-effective, scalable processing route for the fabrication of NMMCs plates, billets and near-net-shape components. In this poster, Aegis will report its detailed investigations on the characterization of the NMMCS, including (1) Microstructures using TEM, SEM and X-Ray, and (2) Mechanical properties (Tensile, compression, fatigue, fracture toughness, creep and ballistic penetration). These investigations will provide a solid foundation for the further development of this class of NMMCs for a variety of potential applications.

Microstructure and Mechanical Properties of Ultra Fine Grained Cu-Al and Cu-Zn Alloys: *Martin Heilmaier*[1]; V. Subramanya Sarma[2]; [1]Otto Von Guericke University; [2]Indian Institute of Technology Madras

Recent studies on ultrafine grained (ufg) electro-deposited Cu containing controlled densities of nanoscale twins revealed a unique combination of very high yield strength (≈850 MPa) and good tensile ductility (≈14%). Here we aim at producing bulk Cu-5wt.%Zn and Cu-5wt.%Al alloys with ufg matrix and controlled densities of nano/sub-micron twins through varying (i) alloying additions (reducing the stacking fault energy SFE compared to pure Cu), (ii) deformation (rolling) temperatures and (iii) annealing treatments. Grain sizes were found in the range of 0.6 to 1.2 µm (including twin boundaries in the evaluation). The Cu-Al alloy shows significantly improved strength in comparison to the Cu-Zn alloy at tensile comparable ductilities >30%. This is attributed to the much stronger contribution of Al to solid solution strengthening. Contrary, the yield stress dependence on grain size is much stronger for Cu-Zn. Possible reasons will be discussed incorporating strain rate sensitivity tests to understand the deformation mechanisms.

Microstructure Development Ultrafine Grained Tantalum Produced by Machining: *Mert Efe*[1]; Hyun Jun Kim[1]; Wilfredo Moscoso[1]; W. Dale Compton[1]; Srinivasan Chandrasekar[1]; Kevin Trumble[1]; [1]Purdue University

Severe plastic deformation (SPD) of pure tantalum and subsequent microstructure development in annealing has been studied. Bulk plates of 1- to 6-mm thickness with plastic strains of 1 to 4 have been achieved in a new process called Large Strain Extrusion Machining (LSEM), in which the imposed plastic strain and plate thickness can be controlled controlled independently in a single stage deformation process. Significant grain refinement and corresponding hardening of the tantalum has been measured. High vacuum annealing with and without zirconium gettering, and metallographic analysis were used to characterize the recrystallization and grain growth behavior of the UFG tantalum. The results are discussed relative to the corresponding behavior in conventional ingot Ta.

Microstructure of a FeCoV Alloy after Equal Channel Angular Pressing and Tempering: Zhongze Du[1]; *Jingtao Wang*[2]; Qingjuan Wang[1]; [1]Xi'an University of Architecture and Technology; [2]Nanjing University of Science and Technology

Equal channel angular pressing (ECAP) of a FeCoV alloy was carried out at room temperature via route A up to 4 passes. The microstructure of the FeCoV alloy after ECAP and subsequent tempering were characterized by transmission electron microscopy (TEM). The FeCoV alloy transformed into a fine lath structure with the width of lath decreasing obviously with ECAP passes. The average width of lathes was 90nm after four passes of ECAP, with dislocation tangles inside the lath. These lath structure would breaks and transforms into a fine lath, when increasing ECAP passes. Although tempering after four passes ECAP had little effect on its lath shape of the microstructure, carbide particles and nanometer precipitates appeared inside the lathes after tempering, indicating the decomposition of the microstructure.

Microstructure Refinement of Ti-6Al-4V Titanium Alloy during Warm Deformation: *Sergey Zherebtsov*[1]; Maria Murzinova[1]; Sergey Mironov[1]; Alexander Pshenichnuk[1]; Gennady Salishchev[1]; Lee Semiatin[2]; [1]Institute for Metals Superplasticity Problems; [2]Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson Air Force Base

Microstructure evolution and mechanical behaviour of Ti-6Al-4V titanium alloy during uniaxial compression at 600°C to a height strain of 70% has been investigated. Initially the alloy has a lamellar microsrtucture. During deformation lamellar microstructure transforms into globular one with a grain size of around 0.4 micrometers. Mechanical behavior of the material is described by the deformation curve with strengthening, softening and steady state flow stage. The microstructure evolution with emphasis on grain boundaries misorientation and mutual turn of alpha and beta phases has been studied. The orientation of phases was determined by means of the EBSD-technique and so-called "single-reflex" method which based on computation of microdiffraction patterns. It have been revealed that transformation of lamellar microstructure into globular one is associated with loss of coherence of interphase boundaries. As soon as the coherence is lost the deformation localizes within each phase intensifying division of the lamellae into equiaxial fragments.

Nano-Grained Copper Strip Produced by Accumulative Roll Bonding Process: *Mohammad Reza Toroghinejad*[1]; Mahnoosh Shaarbaf[1]; [1]Isfahan University of Technology

Accumulative roll bonding (ARB) process is a severe plastic deformation process that has been used for pure copper (99.9%). The ARB process up to 8 cycles was performed at ambient temperature under unlubricated conditions. Microstructural characterizations were done by transmition electron microscopy and electron backscattered diffraction. It was found that continuous recrystallization resulted in microstructure covered with small recrystallized grains with an average diameter below 100 nm. The tensile strength and hardness of the ARB processed copper has become two times higher than initial value. On the other hand, the elongation dropped abruptly at the first cycle and then increased slightly. Strengthening in ARB processed copper may be attributed to strain hardening and grain refinement. In order to clarify the failure mode, fracture surfaces after tensile tests were observed by scanning electron microscopy. Observations revealed that failure mode in ARB processed copper is shear ductile rupture with elongated small dimples.

Novel Microstructures from Severely Deformed Al-Ti Alloys Created by Chip Formation in Machining: Jiazhao Cai[1]; Andreas Kulovits[1]; *Ravi Shankar*[1]; Jörg Wiezorek[1]; [1]University of Pittsburgh

We present some consequences of Severe Plastic Deformation (SPD) of Al-Ti alloys by chip formation in machining that can enable novel opportunities for creating materials with unprecedented properties. Chips cut from Al-6wt%Ti are composed of a refined dispersion of the fragmented remains of a hitherto coarse Al_3Ti embedded in a nanostructured matrix. This multi-phase nanostructured chip material demonstrates considerable resistance to coarsening owing to the thermally-stable dispersion of ultra-fine Al_3Ti precipitates and thus has promise in structural alloy applications. Furthermore, the Al-Ti machining chips are shown to possess excellent grain-refining characteristics, leading to microstructurally refined and homogeneous Al castings. This realization enables a low-cost route for enhancing the efficiency of the grain refiner Al(Ti) master alloy systems by exploiting SPD during chip formation.

OIM Study of Microstructure and Texture Heterogeneity during ECAP of Copper: *Alexander Zhilyaev*[1]; Azat Gimazov[2]; Shrinivasan Swaminathan[3]; Terry McNelley[3]; [1]Centro National de Investigaciones Metallurgicas (CENIM), CSIC; [2]Russian Academy of Sciences, Institute for Metals Superplasticity Problems; [3]Naval Postgraduate School

The heterogeneous deformation of annealed copper subjected to equal channel angular pressing (ECAP) was studied by Orientation Imaging Microscopy. The microstructure and microtexture of a partially pressed Cu billet has been analyzed at the inner and outer corners in the region of the die channel intersection as well as in the middle of the shear zone. Distinctly inhomogeneous deformation of prior annealing twins by interpenetrating slip bands was observed, and some twin-matrix interfaces displayed stair-like offsets. The intense local deformation within the annealing twins is characteristic of dislocation slip and not deformation twinning.

On the Texture Analysis of High Pressure Torsion - Deformed Mg and Cu: *Bartlomiej Bonarski*[1]; Erhard Schafler[1]; Michael Zehetbauer[1]; Borys Mikulowski[2]; [1]University of Vienna; [2]AGH-University of Science and Technology

Polycrystalline pure magnesium (99.8%) and copper (99.99%) have been subjected to High-Pressure Torsion (HPT) at room temperature. A special technique was developed in order to enable the HPT of Mg up to very high shear strains $\gamma \leq 120$ and different hydrostatic pressures (1–8 GPa). The texture development has been analyzed as a function of strain and hydrostatic pressure by systematic X-ray macrotexture investigations and compared with corresponding microhardness measurements. The texture evolution could be described in terms of volume fractions of the following components: (i) {0001}-, {10-11}- and {10-12}- fibres for the case of Mg; (ii) the typical components of shear textures for the case of Cu. From the textures observed, there is evidence for the occurrence of dynamic and static recovery, which turns out to depend not only on the strain but also on the hydrostatic pressure of HPT.

Precipitation Hardening and Grain Refinement in an Al-4.3wt%Mg-1.2wt%Cu Alloy Processed by ECAE: *Vanessa Vidal*[1]; Zheng-Rong Zhang[2]; Bert Verlinden[1]; [1]KULeuven; [2]University of Electro-Communications

In this study the influence of severe plastic deformation on the microstructure and the properties of a precipitation hardenable Al-4.3wt%Mg-1.2wt%Cu alloy was investigated by room temperature compression tests, EBSD and TEM. Samples in under aged, peak aged and over aged condition were deformed in an ECAE die at 180°C. After four ECAE passes fine equiaxed grains with an average size of approximately 200 nm with some traces of remnant elongated grains were obtained. It was observed that peak aged and over aged samples lose much of their strength during ECAE due to fragmentation of the precipitates. The highest strength after four ECAE passes (route Bc) was obtained with samples in under-aged condition, although in this case dissolution of precipitates into the matrix occurred. Formation of new round precipitates with small size were detected during post-ECAE annealing at 180°C. However, this re-precipitation resulted in a softening of the severely deformed samples.

Polymer Bonding Ultrafine Grained Al 6061-T6 Particulate Produced by Machining: Boum-seock Kim[1]; James Mann[1]; Srinivasan Chandrasekar[1]; *Kevin Trumble*[1]; [1]Purdue University

Plane-strain machining has been used to produce 20 to 200 µm size Al6061-T6 particulate having grain sizes less than 100 nm and 50% higher hardness than the bulk Al6061-T6. Several routes for low-temperature (~100°C) densification and bonding the particles using epoxy resins will be presented. Metal fractions greater than 90 vol % have been achieved with no loss of hardness (in the particulate) during the epoxy cure. Microstructure based modeling of composite hardness will be presented and tensile test results will be discussed. Potential structural applications for these composite materials will be elaborated.

Processing and Ballistic Performances of Lightweight Armors Based on Ultra-Fine-Grain Aluminum Composites: *Timothy Lin*[1]; Fei Zhou[1]; Quan Yan[1]; Chufu Tan[1]; Bob Liu[1]; Adolphus McDonald[2]; [1]Aegis Technology Inc.; [2]U.S. Army Aviation and Missile Command

Over last few decades Al-based metal matrix composites (MMCs) have become a promising material of choice for lightweight armors in vehicles. Recent development in ultra-fine-grain (UFG) and nanostructured material technology provides a new opportunity for the substantial strength enhancement of MMCs unattainable with the conventional microstructure of microscale, leading to significant weight reduction in armor packages. In this paper, Aegis Technology will present its latest development of a novel class of nanostructured metal Matrix composites (NMMCs) based on submicron SiC particulates reinforced nanocrystalline Al alloys, which is sponsored through an U.S. Army Small Business Innovative Research (SBIR) project. In this project, Aegis has successfully demonstrated the fabrication of large-dimension NMMCs plates by using a cost-effective synthesis and consolidation process that can be scaled up for the mass production. In this presentation, Aegis will report the microstructure, processing, mechanical properties and their correlations of this class of NMMCs. Particularly Aegis will present a detail investigation of the ballistic behaviors of the NMMCs under high-speed bullets of rifle and machine guns, in which a physical model and the associated simulation have been also developed to predict the penetration depth and identify the key influential parameters.

Room Temperature Relaxation in Copper under High Pressure Torsion Detected by In-Situ Synchrotron Diffraction: *Askar Kilmametov*[1]; Gavin Vaughan[2]; Alain Yavari[3]; Ruslan Valiev[1]; [1]Ufa State Aviation Technical University; [2]European Synchrotron Radiation Facilities; [3]Institut National Polytechnique de Grenoble

Structural relaxation in severely deformed Cu has been investigated in real time diffraction experiments at room temperature during in situ high pressure torsion (HPT) in high energy synchrotron light. Simultaneous relative changes in Bragg peak's broadening and crystal lattice expansion were under study in loading-unloading regime of torsion straining. Experimental results are consistent with the attribution of the annihilation of crystalline defects generated during HPT. Relaxation kinetics assumed to be controlled by diffusion; therefore the enhanced diffusivity has been estimated due to extremely high excess vacancy concentration, which is typical for those at thermal equilibrium near the melting point.

Solid State Amorphization of Cu+Zr Multi-Stacks by ARB and HPT Technique: *Yufeng Sun*[1]; Yoshikazau Todaka[2]; Minoru Umemoto[2]; Nobuhiro Tsuji[1]; [1]Osaka University; [2]Toyohashi University of Technology

A series of CuZr binary alloys with wide composition range were fabricated through ARB and HPT technique using pure Cu and Zr metals as the starting materials. Bulk alloy sheets with thickness of about 0.8 mm after ARB process and alloy disks with 0.35mm in diameter and 10 mm in diameter after HPT process can be obtained respectively. The structures of all the alloys were found to be gradually refined with the increasing of ARB cycles or HPT rotations. As a result, nanoscale multiple-layered structure was formed for the 10 cycled ARBed specimens, which could partially transform into amorphous phase after low temperature annealing. While for the as-HPTed sample, the alloy will be completely amorphrized after 20 rotations without any heat treatment. The thermal stabilities of the amorphous alloys were studied. The deformation behavior and the amorphization mechanism during the ARB and HPT process were put forward and discussed.

Stored Energy and Recrystallization Temperature of High Purity Copper after Equal Channel Angular Pressing: Yue Zhang[1]; *Jingtao Wang*[1]; [1]Nanjing University of Science and Technology

Equal channel angular pressing (ECAP) was conducted at room temperature to impose high strain into high purity copper. Differential Scanning Calorimeter (DSC) was used to estimate the store energy from ECAP and recrystallization temperature. It was found that the stored energy increases upon ECAP processing until a peak reached at 12 passes, and a slight decrease in stored energy was observed at higher ECAP passes. The recrystallization temperature decreases upon the increase of stored energy up to ~50 J/mol, and reaches a stable valve of ~210°C. Partial annealing of an ECAP processed (8 passes) sample by heating to ~185°pC at a heating rate of 20°C /min released the stored energy from ~55J/mol to ~18J/mol, without substantial change on the recrystallization temperature of the sample. A model was proposed to help understanding the recrystallization mechanism of ultrafine-grained copper and the observations above.

Strain-Assisted Grain Refinement of Co-Fe Alloys upon Room-Temperature Compression: Lai-Chang Zhang[1]; Mariana Calin[1]; Flora Paturaud[2]; *Jürgen Eckert*[1]; [1]Leibniz Institute for Solid State and Materials Research (IFW) Dresden; [2]W.C. Heraeus GmbH

It is well-known that severe plastic deformation methods, including equal channel angular pressing and high-pressure torsion, have been widely used to produce bulk ultrafine- and/or nanoscale-grained metals and alloys. However, the mechanism of the deformation-induced grain refinement is strongly related to the crystal structure of the investigated metallic materials. In this work, instead of applying severe plastic deformation, pronounced grain refinement to ultrafine or nanoscale has been achieved upon the room-temperature conventional compression of initially coarse-grained single-phase bcc Co-xFe (x=25 and 35 wt%) alloys. These alloys exhibit large plasticity over 140% without fracture at room temperature. The grains refine with increasing of the deformation strains during compression. The possible mechanism for the strain-induced grain refinement under compression is a consequence of the shear deformation and dramatic deformation-enhanced atomic diffusion during deformation.

Superplastic Ultrafine-Grained Sheet Produced out of the Al-Li-Mg-Sc Alloy with Enhanced Mechanical Properties Introduced by ECAP and Rolling: *Nina Yunusova*[1]; Rinat Islamgaliev[1]; Nikolay Krasilnikov[2]; Gulnaz Nurislamova; Ruslan Valiev[1]; [1]Ufa State Aviation Technical University; [2]Ulyanovsk State University

ECAP at elevated temperatures was applied to produce bulk textureless billets with equiaxed grains with a size less than 1 µm in size out of Al-Li-Mg-Sc alloy. The billets demonstrated the effect of high-strain-rate (10^{-1} s^{-1}) superplasticity (780%) at a relatively low (400°C) temperature. The sheet fabricated out of the billet by warm rolling retained its equiaxed structure with minor grain refinement and exhibited superplasticity with elongation 530% at the same temperature and the strain rate equal to 10^{-2} s^{-1}, when compared to the ductility (80%) of a sheet fabricated out of a coarse-grained billet. Short-time annealing of the superplastic sheet at a temperature of solid solution treatment with subsequent aging allowed to use potential of UFG structure and dispersion hardening to achieve very high UTS (690 MPa) retaining the initial ductility.

Synthesis of Al-$Al_{85}Ni_{10}La_5$ Nanocomposites by Mechanical Milling: *Zhihui Zhang*[1]; Yizhang Zhou[1]; Enrique Lavernia[1]; [1]University of California, Davis

Mechanical milling has been widely used to fabricate metal matrix composites and dispersion-strengthened alloys with the advantage of improved dispersion, enhanced interfacial bonding and refined microstructure. This study reports on the synthesis of an Al-$Al_{85}Ni_{10}La_5$ nanocomposite by milling a mixture of Al (mean particle size ~60 µm) and amorphous $Al_{85}Ni_{10}La_5$ powder (particle size 10~25 µm) at a cryogenic temperature. The microstructural evolution during the milling process was investigated using XRD, DSC, SEM and TEM. The results show that nanocrystaline Al matrix with a grain size about 26 nm was obtained and the amorphous powder was fractured and homogeneously distributed in the Al matrix with a whisker shape (1~2 µm thick with an aspect ratio of 1~5). The mechanical behavior of the powder and consolidated bulk composite was also discussed.

Tailoring the Microstructures of Ultrafine Grained Aluminum through a Two-Step Annealing Process: Naoya Kamikawa[1]; *Xiaoxu Huang*[1]; Niels Hansen[1]; [1]Riso National Laboratory

Due to microstructural and textural heterogeneities, annealing of nanostructured metals is difficult to control in order to avoid non-uniform coarsening and recrystallization. The present research demonstrates a method to delay such process by annealing at low temperature before annealing at high temperatures. By this two-step process the structure is homogenized and the stored energy is reduced significantly during the first annealing step. As an example high purity aluminum has been deformed to a total reduction of 98.4% by accumulative roll-bonding at room temperature. Isochronal annealing for 0.5 h of the deformed sample shows initiation of recrystallization at about 200°C. However, when introducing an annealing step for 6 h at 175°C the initiation of non-uniform coarsening and recrystallization is significantly delayed. To underpin these observations the structural evolution has been characterized by TEM, showing that extensive annihilation of low-angle dislocation boundaries characterizes the low-temperature annealing step.

Texture Analysis of Materials Subjected to Equal Channel Angular Pressing: Katrina Houston[1]; Srinivasan Swaminathan[1]; Jianqing Su[1]; *Terry McNelley*[1]; [1]Naval Postgraduate School

The correlation between the microstructure and texture for aluminum alloys processed by equal channel angular pressing (ECAP) has been studied. The microstructure was characterized using optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The local textures were discerned using orientation imaging microscopy (OIM) and compared to macro-texture measurements made by X-ray diffraction. Specific emphasis has been given to understanding the long-range arrangement of texture variants in the microstructure. The interfaces between texture variants are pre-cursors to high angle boundaries in repetitive ECAP. The microstructure-mechanical property relations were also examined.

The Effect of Deformation History on the Microstructure and Texture of Annealed ECAE Processed Copper: *Ana Erb*[1]; Daudi Waryoba[1]; Peter Kalu[1]; [1]Florida A&M University-Florida State University College of Engineering

Grain refinement by equal channel angular extrusion (ECAE) has recently attracted attention due to its effectiveness of producing ultrafine-grained structures in bulk materials. In the present investigation, OFHC Copper ECAE processed at room temperature via route B_C (where the billet is rotated 90° in the same direction between consecutive passes) has shown refinement of the grain size to about 1µm at 4 passes. On the other hand, a billet swaged first to a 30% reduction and ECAE processed to 4 passes shows a lower grain size refinement to about 3µm. Both deformation history, however, show a similar grain boundary structure, consisting of about 25% high angle grain boundaries (HAGBs) and 75% low angle grain boundaries (LAGBs). The effect of deformation history on the microstructure and texture of these materials is discussed.

The Influence of Impurities on Structure and Mechanical Properties of Nanostructured Titanium: *Rinat Islamgaliev*[1]; Vil Kazyhanov[1]; Askar Kilmametov[1]; Alfred Sharafutdinov[1]; Ruslan Valiev[1]; [1]Ufa State Aviation Technical University

High pressure torsion (HPT) is a well established severe plastic deformation technique for producing nanostructured metallic materials. Using new HPT installation we have investigated the microstructure (grain size, content of omega-phase) and mechanical properties of commercially pure titanium with different content of impurities (VT1-00, VT1-0, Grade 4) processed at various HPT regimes. It was found that content of high pressure omega-phase depends strongly on purity of initial material influencing on strength, ductility and thermal stability of grain structure. Special attention has been paid to investigation of the influence of thermomechanical treatments on nanostructure and mechanical properties.

Thermal Stability of Ultrafine Grains Size of Pure Copper Obtained by Equal-Channel Angular Pressing: *Nayar Lugo*[1]; Nuria Llorca[2]; Joan Suñol[3]; Jose Cabrera[1]; [1]Universidad Politecnica de Catalunya; [2]Universitat de Barcelona; [3]Universitat de Girona

Ultrafine grains size of pure copper 99.98% have been obtained by severe plastic deformation using the Equal-Channel Angular Pressing (ECAP) method. Copper samples were ECAPped from 1 to 8 passes developing a finer microstructure showing grain sizes of 250 nm after the 8th pass through the die. Important enhancement in the mechanical strength properties was obtained in the material processed by this technique. Subsequent heat treatments were carried out to evaluate the grain size thermal stability of the ECAPped samples. Microstructure and mechanical properties together with Differential Scanning Calorimetric (DSC) tests were carried out in order to evaluate thermal recovery and recrystallization temperature as well as activation energy. Good correlation was obtained with the microstructure and the mechanical properties. Heat treatment produced significant changes in the behaviour of the material.

Transmission Electron Microscopy (TEM) Study of the Effect of Machining Parameters on Grain Refinement in Aluminum Alloy Machine Chips: *Lei Dong*[1]; Judy Schneider[1]; [1]Mississippi State University

The resulting microstructure of machined chips of AA 2195 and AA 2219 were characterized by Transmission Electron Microscopy(TEM). By varying the specimen diameter and rotation and feed speed, different strains and strain rates were imposed on the metal during the cutting process. Dependant on the metal cutting conditions, the resulting microstructure was found to contain either elongated or ultra-fine grains. These observations suggest that the grain refinement mechanisms were influenced by the metal cutting or hot working conditions. The use of metal cutting theory is being explored as a basis of quantifying the optimal conditions for grain refinement during the friction stir welding process.

XRD Characterisation of Ultrafine Grained (UFG) Al-Mg Alloys: *Markus Dinkel*[1]; Florian Pyczak[1]; Mathias Göken[1]; [1]University Erlangen - Nürnberg

The characterisation of ultrafine grained materials by X-ray diffraction (XRD) is a suitable and convenient method to acquire information about this material class. In this work examples from different areas of interest are covered. The effect of impurities on the crystallite sizes and dislocation densities of ECAP – processed AlMg alloys is studied. It can be shown that with increasing Magnesium content the achievable reduction in crystallite size with ECAP eventually reaches a saturation state and a further reduction of the structural size seems unlikely. Simultaneously the dislocation density increases to a plateau level with increasing Mg content. In annealing experiments the microstructural stability of AlMg0.5 and the resulting changes are determined by XRD. As a result it becomes evident that the annealing leads to a moderate increase in crystallite size up to a temperature where accelerated crystallite growth begins. Also XRD results prior and after fatigue testing are presented.