2010 Functional and Structural Nanomaterials: Fabrication, Properties, Applications and Implications: Synthesis of Nanomaterials I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee

Program Organizers: David Stollberg, Georgia Tech Research Institute; Nitin Chopra, University of Alabama; Jiyoung Kim, University of Texas - Dallas; Seong Jin Koh, University of Texas at Arlington; Navin Manjooran, Siemens Corporation; Ben Poquette, Keystone Materials; Jud Ready, Georgia Tech

Tuesday AM Room: 214
February 16, 2010 Location: Washington State Convention Center

Session Chair: Navin Manjooran, Siemens AG; Jud Ready, Georgia Tech

8:30 AM Introductory Comments

8:35 AM Invited

Oxide Nanostructures: Synthesis, Characterization and Properties Evaluation: *Avanish Srivastava*[1]; [1]National Physical Laboratory

Important findings pertaining to ZnO, TiO2 and WO3 on novel growth morphologies and related spectroscopic, optical and electrochemical performance are elucidated. Moreover certain fundamental concerns on oxide nanostructures dealing with their preparation and correlating the ensuing microstructure with luminescence and electrochemical activity have been tackled to resolve queries of (i) effect of processing on size and morphologies, (ii) consequences of microstructures on optical performance and (iii) correlation of magnetic resonance and Raman spectroscopic measurements with fine scale microstructures and potential applications. A detailed correlation between nanostructures, their growth parameters, microstructural characterization and their property evaluation will be presented and discussed. References: 1.A.K.Srivastava, K.N.Sood, K.Lal, R.Kishore., Patent Filed Ref. No. 0773DEL2005 Dated 31st March, 2005. 2.A.K.Srivastava, N.Gupta, K.Lal, K.N.Sood, R.Kishore, J Nanosci Nanotech 7,1941 (2007). 3.M.Deepa, A.K.Srivastava, K.N.Sood, S.A.Agnihotry, Nanotechnology 17, 2625 (2006). 4.A.K.Srivastava, M.Deepa, S.Bhandari, H.Fuess, Nanoscale Res.Lett. (2009) in press.

8:55 AM

Effect of Processing Parameters on the Physical, Thermal, and Combustion Properties of Plasma-Synthesized Metallic Nanopowders: *Chris Haines*[1]; Darold Martin[1]; Joseph Paras[1]; Ryan Carpenter[1]; Deepak Kapoor[1]; [1]US Army ARDEC

US Army ARDEC has employed inductively-coupled inert gas condensation to synthesize a wide range of nanopowders. This paper focuses on metallic systems; specifically the effects of processing parameters on the particle size, thermal characteristics, and combustion behavior of these materials. A design of experiments was conducted which varied the plasma power, feed rate, system pressure, and quench gas conditions while holding all other parameters constant. The powders were characterized by X-ray diffraction, BET, FE-SEM, TGA/DSC, and bomb calorimetry. The role of each processing parameter on the various properties will be discussed and summarized with effects plots. The consistency of these effects across a number of material systems will be shown. Lastly, the concept of *tunability* will be discussed.

9:15 AM

Synthesis and Characterization of Single Crystalline Metal Nanowire Rings: *Yongjie Zhan*[1]; Hao Lu[1]; Yang Lu[1]; Cheng Peng[1]; Jiangnan Zhang[1]; Jun Lou[1]; [1]Rice University

In this paper, we reported a simple and effective solvothermal route to prepare copper and silver nanowire ring structures, demonstrating new possibility through hydrothermal/solvothermal route to prepare novel nanostructures. Single crystalline copper and sliver nanowire rings with wire diameters from tens to hundreds of nanometers and ring diameters around 15-40μm were synthesized through solvothermal process, in which cuprous chloride was used as copper resource and polyvinyl alcohol (PVA) used as reducing agent, and N-methyl-2-pyrrolidone (NMP) used to provide a water/NMP mixture reaction system was believed to be the key factor to form the novel closed structure. A growth-stress induced bending mechanism was suggested to explain the possible formation mechanism. The single crystallinity and perfectly closed structure of copper nanowire rings could potentially be explored for many interesting applications in the fields of nanoelectronics, nanophotonics and nanomechanics. Preliminary results on mechanical, optical and electrical characterizations of such nanostructures were also dicussed.

9:35 AM

Spontaneous Growth of Novel Hexagonal Mn Nanowhiskers from Hydrogen Activated Laves Phase Alloys: *Erdong Wu*[1]; Xiumei Guo[1]; Wuhui Li[1]; [1]Institute of Metal Research, Chinese Academy of Science

Spontaneous growth of metal whiskers is a well-established phenomenon. Owing to its significant importance either as a hidden peril for electronic devices or a potential fabrication technique for complex microstructures, the phenomenon has been extensively studied for decades. However, only the whiskers of soft metals with relatively low melting points, such as Sn, Cd and Zn and primarily in a micrometer diameter scale, can spontaneously grow at room temperature. With the aid of activation of repeated hydrogenation/dehydrogenation, the crystalline whiskers of transition metal Mn in the shape of nanorod can segregate and grow spontaneously from the crystals of Zr1-xTixMnCr Laves phase alloys at room temperature. Moreover, the Mn atoms in the nanowhiskers form a novel hexagonal structured allotrope, which is the first non-cubic allotrope found for the Mn element. The morphology and structure of the Mn nanowhiskers are exhibited, and the mechanism and potential applications of the phenomenon are discussed.

9:55 AM

Geometry Dependence of the Strain-Driven Self-Rolling of Semiconductor Nanotubes: Ik Su Chun[1]; Huan Li[1]; Archana Challa[1]; *K. Jimmy Hsia*[1]; Xiuling Li[1]; [1]University of Illinois at Urbana-Champaign

Semiconductor nanotube is an emerging material system that has caught limited attention, yet possesses the potential to provide a wide range of functionalities (X. Li, J. Phys. D. 41, 193001, 2008). It is formed by a combination of bottom-up and top-down approaches through self-rolling of residually strained thin-films that are epitaxially grown and lithographically defined. This allows feasible large area assembly and integration with existing semiconductor technologies while maintaining control of tube size and heterojunction formation in the tube wall. The tube diameter is determined by the total layer thickness and the mismatch strain in the epitaxial layers. The self-rolling direction is controlled by the anisotropy of the Young's moduli and, for crystalgraphically equivalent directions, depends on the feature geometry of the thin film. In this presentation, we report on the experimental observations and FEM simulations of the geometry dependence of the self-rolling behavior of InxGa1-xAs - GaAs semiconductor nanotubes.

10:15 AM Break

10:30 AM

Mesoscale Simulations of Self-Assembly of Arbitrarily-Shaped Particles in Bulk and at Fluid-Fluid Interfaces: *Paul Millett*[1]; Yu Wang[2]; [1]Idaho National Laboratory; [2]Michigan Tech

Recent advances in the ability to experimentally control the size, shape, and composition of nanoparticles have significantly broadened the possibilities to create novel mesoscale structures as a result of their "bottom-up" assembly. Here, we present a novel mesoscale simulation approach that utilizes diffuse interface fields to capture and investigate the dynamic assembly processes for arbitrarily shaped particles with arbitrary charge density and/or dipole moment. Illustrative results demonstrating the method's ability to predict a wide variety of colloidal crystal structures, with a particular focus on binary lattices consisting of positively- and negatively-charged particles, will be presented. Furthermore, this mesoscale approach has also been extended to include the capillary forces experienced by particles segregated at fluid-fluid interfaces. We will present simulations of the complex evolutions of multi-phase fluid mixtures in which particle absorption to the interfaces significantly alters the dynamics of system.

10:50 AM

Morphological Evolution and Coarsening Process of a Strained Heteroepitaxial Thin Film during Constant Deposition: *Solmaz Torabi*[1]; Steve Wise[2]; Peng Zhou[1]; John Lowengrub[1]; [1]University of California Irvine; [2]University of Tennessee, Knoxville

Self-assembly semiconductor nanostructures such as quantum- dots are a promising inexpensive and effective approach to manufacture novel nanoscale electronic devices. Producing such quantum-dot-based devices, however, is still challenging. Consequently, we need to have a fundamental understanding of the self-organization process (nucleation, growth and coarsening) during epitaxial growth. So we study the morphological evolution of a strained heteroepitaxial thin film, during continuous mass deposition, on a substrate. Here, we use our new approach for modeling strongly anisotropic crystal and epitaxial growth using regularized, anisotropic Cahn-Hilliard-type equations. In order to model the misfit strains, we add the elastic energy to our diffuse model. Adding elastic energy modifies the equilibrium shape and in particular affects the shape of the corners. We can predict different Qdot shapes, such as pyramids and domes, based on the strength of the elastic interactions. We present 2D and 3D numerical results using an adaptive, nonlinear multigrid finite-difference method.

11:10 AM

Development and Characterization of Nanoporous Carbon Coated ZnO Powders Prepared by Pyrolysis of Spray Dried ZnO-PVA Mixtures: *Burak Özkal*[1]; Seyma Duman[1]; Osamu Yamamoto[2]; [1]ITU; [2]Akita University

It is known that some carbons remain as residue during inert atmosphere pyrolysis of polymers and type and polymerization degree of the polymer are very important parameters leading different final properties. Starting from homogeneous polymer and powder mixtures, it is possible to produce composite powders and structures where the outer phase is amorphous carbon. This coating layer is very attractive especially for nanopowders and nanofibers where encapsulation is necessary. In this study, development and characterization of the nanoporous carbon coated ZnO powders were performed consecutive processes of spray drying and polymer pyrolysis technics. For this purpose spray drying process conditions of ZnO powders having different amounts of PVA were optimized and final products were characterized via advanced powder characterization technics. Spray dryed powders were subjected to polymer pyrolysis under protective atmosphere and then integrity of ZnO granules and properties of nanoporous carbon coating were characterized.

11:30 AM

Low-Temperature Synthesis of NiFe2O4 Spinel Nanopowder via Solid-State Reactions: *Zhigang Zhang*[1]; Guangchun Yao[1]; Jia Ma[1]; Zhongsheng Hua[1]; Lei Wang[1]; [1]Northeastern University

Nickel ferrite (NiFe2O4) nanopowder was successfully synthesized via solid-state reactions at low temperature. The precursors prepared by rubbing FeSO4•7H2O, NiSO4•6H2O, NaOH and dispersant sufficiently at room temperature were calcined at different temperature for different holding time. Effects of content of dispersant, calcining temperature and holding time on morphology and grain size of nanopowder were researched by orthogonal experiment. Single factor experiment was carried out successively according to range analysis. The phase, surface morphology and grain size were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM) and laser particle analyzer. The solid-state reaction mechanism was discussed according to the XRD analysis of the precursors. The results indicated the NiFe2O4 spinel nanopowder prepared by adding 20wt% NaCl (dispersant) in the rubbing process and calcining at 800° for 1.5 hour was of pure cubic spinel structure with the grain size about 40-70 nm.

11:50 AM Concluding Comments

Advances in Composite, Cellular and Natural Materials: Metal Foams

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee

Program Organizers: Yuyuan Zhao, The University of Liverpool; David Dunand, Northwestern University

Tuesday AM Room: 305
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Antonia Antoniou, Georgia Institute of Technology; Ki-Ju Kang, Chonnam National University

8:30 AM

Fabrication of Ti-6Al-4V Open-Cellular Foams by Additive Manufacturing Using Electron Beam Melting: *L. Murr*[1]; S. Gaytan[1]; F. Medina[1]; E. Martinez[1]; L. Martinez[1]; R. Wicker[1]; [1]University of Texas at El Paso

Methods to fabricate Ti or Ti-alloy foams suffer from their high melting point issues, extreme chemical affinity with atmospheric gases, and other reactivity issues. In this work, we have utilized computerized tomography (CT) scans of more common aluminum alloy foams to develop build elements in CAD models from which Ti-6Al-4V prototypes have been fabricated by electron beam melting (EBM). This strategy has allowed a wide variation of open-cellular foams of Ti-6Al-4V to be fabricated in density or porosity variations and associated low stiffnesses. Complex, monolithic arrays have been fabricated composed of various density regimes and including solid, fully dense components. These prototypes have been characterized by optical and electron microscopies. Applications in biomaterials implant areas and aeronautics and aerospace will be illustrated. While this program is specific to Ti-6Al-4V as a precursor powder, any metal or alloy prototype may be fabricated.

8:50 AM

Compression-Compression Fatigue of LENS-Processed Cellular Titanium: *Nikolas Hrabe*[1]; B. Vamsi Krishna[2]; Amit Bandyopadhyay[2]; Rajendra Bordia[1]; [1]University of Washington; [2]Washington State University

Cellular titanium is a candidate material for a range of biomedical and aerospace applications. It has been fabricated over a range of relative densities (0.53-0.73) using laser-engineered net shaping (LENS) and found to display monotonic mechanical properties suitable for the intended application of load-bearing bone replacement implants. For all relative densities tested, the compression-compression fatigue strength at 1 million cycles was found to be rather high (approximately 150% of the yield strength). This talk will include a correlation of monotonic and fatigue results to the Gibson-Ashby model for cellular solids as well as a discussion on the proposed deformation mechanism based on results from scanning electron and optical microscopy, and micro-computed x-ray tomography.

9:10 AM

Properties of Energetic Materials Reinforced by Open-Cell Metal Foams: *Dmitriy Kiselkov*[1]; Ravil Yakushev[1]; [1]Institute of Technical Chemistry

One of the most challenging ways of improving of the energetic materials performance is reinforcement of energetic polymer composition by heat conducting elements. Widely used methods of filling the energetic material by different types of metallic components (fibers, wires, meshes, cells, bars etc) have considerable disadvantages including impossibility of uniform distribution of the elements. Thus the idea of using open-cell metal foams as heat conducting elements seems to be very challenging. The aim of this work is to study the influence of characteristics (porosity, cell size) and chemical structure of the open-cell metal foams on energetic and ballistic properties of energetic materials. The results of this study can be used for the development of energetic materials with required characteristics.

9:30 AM

Microstructural Characterization of PORVAIR Metal Foams: *S. Raj*[1]; Jacob Kerr[2]; [1]NASA Glenn Research Center; [2]Pennsylvania State University

Metal foams are being studied for use in future aircraft engines to control environmental noise. The development of acoustic and structural models require an understanding of the complex foam microstructures and their role in noise reduction. Details of quantitative microstructural analyses of PORVAIR metal

foams are reported. The present results revealed that 57% of the cell faces were pentagonal in nature while the quadrilateral and hexagonal faces were 25 and 15%, respectively. These observations reveal that the Kelvin tetrakaidecahedron with six (43%) flat quadrilateral faces and eight (57%) curved hexagonal faces cannot adequately describe the PORVAIR cellular microstructure. Instead, it is suggested that the PORVAIR cells have 11 or 12 faces consisting of 3 quadrilateral, 2 hexagonal and either 6 or 7 pentagonal faces, respectively.

9:50 AM

Formation and Disappearance of Crack-Like Pores for Al Foams Made by PM Route: *Lei Wang*[1]; Guangchun Yao[1]; Xiaoming Zhang[1]; Yihan Liu[1]; Jia Ma[1]; [1]Northeastern University

Aluminum foams have been fabricated by powder metallurgy technology. The reason for formation of crack-like pores formed during early foaming stage and mechanism of their disappearance are analyzed systematically. The model for disappearance of crack-like pores has been put forward for the first time. Stress generated within compacted precursors is caused by particle interaction in the presence of high compaction pressure and is the main reason of formation of crack-like pores. The results show that pressure difference ΔP between initial round pores and crack-like pores is the drive force of disappearance of crack-like pores. The rapid reduction of ΔP is attributed to the decomposition characteristic of TiH2 powder.

10:10 AM Break

10:30 AM

Open Celled Bulk Metallic Glass Foam Using Eqaul Channel Angular Pressing: *Marie Cox*[1]; David Dunand[1]; Suveen Mathaudhu[2]; [1]Northwestern University; [2]U.S. Army Research Laboratory

Bulk metallic glasses (BMGs) have very high specific strength but lack room-temperature plasticity and fail catastrophically from highly localized shear bands [1]. Shear band arrest can be achieved by pores, leading to improved compressive ductility in porous BMGs [2]. Here, we present BMG foams with a relative densities of 30-55% produced by consolidating a powder mixture of $Zr_{58.5}Nb_{2.8}Cu_{15.6}Ni_{12.8}Al_{10.3}$ (Vitreloy 106a) and tungsten using different warm equal channel angular extrusion (ECAE) routes (1A, 2A, 2B 2C) followed by the dissolution of the tungsten fugitive phase. The foam morphology was investigated using a variety of imaging techniques and uniaxial compression testing was preform to determine the affect of porosity and ECAE route on the materials mechanical properties. [1] C.A. Schuh, T.C. Hufnagel, and U. Ramamurty: *Acta Materialia*, 2007, vol. 55, pp. 4067-4109.[2] A.H. Brothers and D.C. Dunand: *Advanced Materials*, 2005, vol. 17, pp. 484-846.

10:50 AM

Mechanical Damping Properties of Al-Si Closed-Cell Aluminum Foam: *Yong Liang Mu*[1]; Guang Chun Yao[1]; Hong Jie Luo[1]; [1]Northeasten University

The mechanical damping properties of Al-Si closed-cell aluminum foam with various porosities were measured using the cantilevered beam mount based on a modified sample geometry. The measured loss factor shows that Al-Si closed-cell aluminum foam have a damping capacity which is independent of frequency and increases with increasing porosity when the cell size is kept constant. There is a critical strain amplitude εe. When strain amplitude is less than εe, the loss factor does not change with strain amplitude. When strain amplitude exceeds εe, the loss factor increases remarkably with increasing strain amplitude. A Voigt two-parameter model was introduced to explain the damping behavior of Al-Si closed-cell aluminum foam.

11:10 AM

Sound Absorption of Closed-Cell Aluminum Foam Fabricated by Melt Foaming Route: Lisi Liang[1]; Yongliang Mu[1]; Lei Wang[1]; Zhongsheng Hua[1]; Jia Ma[1]; *Guangchun Yao*[1]; [1]Institute of Materials and Metallurgy, Northeastern University

Closed-cell aluminum foam was fabricated by molten body transitional foaming process. The sound absorption coefficient α was measured by standing-wave method in the frequency range up to 2000Hz. The relationship between α and pore structure of aluminum foam was studied. The results show that there is an optimal porosity with which aluminum gives the best sound absorption. Moreover, the influence of perforation rate and cavity thickness on α was investigated. The sound absorption coefficient increases remarkably when the perforation rate reaches 1.5%. However, when the perforation rate exceeds 2.5% α is lower than that of as received sample. Moreover, the highest sound absorption coefficient shows the trend of traveling to low-frequency region with increasing the cavity thickness.

11:30 AM

Study on Compressive Properties of Aluminum Foams Reinforced with Short Copper-Coated Carbon Fibers: *Jinjing Du*[1]; Guangchun Yao[1]; Zhuokun Cao[1]; [1]Northeasten University

The closed-cell aluminum foams reinforced with 1.7 vol.% copper-coated short carbon fibers (Al/Cf) were manufactured by melt foaming method using TiH2 as blowing agent. The distributions of the short fibers in the composite foams observed by scanning electron microscopy(SEM)and the quasi-static compressive properties of Al/Cf foams have been investigated. The compression test indicated that Al/Cf foams show better compressive properties than pure aluminum foams. The compressive curves of the composite foams exhibit a smooth and no dentate collapse plateau region; the compressive yield stress and energy absorption capacity of the composite foams increase more rapidly than those of pure aluminum foams with increasing density.

11:50 AM

The Influence of Cell Shape Anisotropy on the Compressive Property of Closed-Cell Al-Si Alloy Foam: *Yong Liang Mu*[1]; Guan Chun Yao[1]; [1]Northeastern University

Closed-cell Al-Si alloy foams have been prepared by melt route. The cell shape anisotropy ratio of Al-Si alloy foams specimens in relative density range of 0.11~0.39 were measured. The quasi-static compressive tests show that Al- Si alloy foams have higher plastic collapse stress in the longitudinal direction than in the transverse direction. The plastic collapse stress ratio increases with cell shape anisotropy ratio, which is basically in agreement with Gibson&Ashby model. Moreover, energy absorption capacity of Al-Si alloy foams was investigated. The results show that the energy absorption capacity in the longitudinal direction is higher than that in the transverse direction.

Alumina and Bauxite: Bayer Process Chemistry and Alumina Quality II

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee

Program Organizers: Carlos Suarez, Hatch Associates Inc; Everett Phillips, Nalco Company

Tuesday AM Room: 611
February 16, 2010 Location: Washington State Convention Center

Session Chair: Pat Clement, Alcoa - Point Comfort Operations

8:30 AM Introductory Comments

8:40 AM

Segregation of Alumina – The Challenge for the Aluminium Industry: *Andreas Wolf*[1]; Peter Hilgraf[1]; [1]Claudius Peters Projects GmbH

The possible segregation during filling of alumina into storage silos causes substantial problems in the further aluminium production chain. For example accumulation of finenesses < 45 µm leads to sludge formation inside the electrolysis cells which can result in anode effects. The present paper reports on the performance, functional and operational experiences with the Anti-Segregation Technology (ASS). The primary advantage of the air-locked ASS is feeding the alumina into the silos with an unchanged grain size distribution between inlet and outlet of the silo.This paper describes how to integrate ASS Technology and how to use the optimization potential of this technology in several installations up to 50000 tons storage capacity engineered by Claudius Peters.

9:10 AM

Unique High-Temperature Facility for Studying Organic Reactions in the Bayer Process: *Allan Costine*[1]; Joanne Loh[1]; Robbie McDonald[1]; Greg Power[1]; [1]CSIRO Minerals

CSIRO's new autoclave facility at Waterford features safe, continuous monitoring of oxygen and hydrogen concentrations in the headspace of a 2 L Parr reactor. The system can be operated up to 280°C and 1125 psi, enabling investigations over the full range of Bayer unit operations. The facility is

particularly suitable for the study of wet oxidation. Results from commissioning tests including detection response times, purge times and limits of detection for the O_2 and H_2 electrochemical sensors are provided. Preliminary results from the treatment of individual organics under Bayer digestion conditions are reported. The system will be used for both fundamental investigations and to solve specific problems for individual industry clients.

9:40 AM Break

10:00 AM

Technology Solutions to Increase Alumina Recovery from Aluminogoethitic Bauxites: *Andrey Panov*[1]; Alexander Suss[1]; Alexander Fedyaev[1]; [1]RUSAL VAMI

High grade lateritic bauxites from tropical regions have iron minerals largely represented by hematite and alumino-goethite. The latter can contain upto 33 molar % alumina subsituted in the goethite crystallic lattice, resulting 0.5 to 3.5% alumina being predominantly not recoverable even under high temperature digestion conditions. Finding the way of extracting this alumina can substantionally enhance economics of alumina refineries, specially processing imported bauxites. The paper describes technological solutions developed in RUSAL VAMI and enhancing extraction of alumina from alumino-goethite. Ways of industrial implementation of the technology are discussed.

10:30 AM

New Polymers for Improved Flocculation of High DSP-Containing Muds: *Matthew Davis*[1]; Qi Dai[1]; H.-L. Tony Chen[1]; Matthew Taylor[1]; [1]Cytec Industries

The production of alumina from bauxite requires effective solid-liquid separation in gravity thickeners to generate liquor containing low amounts of suspended solids. As global resources of high quality bauxite are depleted and efforts are made to exploit lower quality deposits which contain increased levels of reactive silica, this separation becomes more difficult. This is an expected consequence of increased levels of desilication product (DSP) and other silica-derived mineral species in the mud residue which are poorly flocculated by conventional polymeric flocculants. It has been discovered that polymers incorporating silane functionality show improved flocculation of suspended DSP solids when added to the slurry in combination with hydroxamated polyacrylamide (HXPAM). Recent developments will be presented to demonstrate the utility of these new molecules for improved clarification of red mud-containing slurries generated in the Bayer Process.

11:00 AM

Some Aspects of Tricalcium Aluminate Hexahydrate Formation on the Bayer Process: *Silvia Franca*[1]; Paulo Braga[1]; Jorge Aldi Lima[2]; Juarez Moraes[2]; Americo Borges[2]; [1]CETEM; [2]Alunorte

Tricalcium aluminate hexahydrate (TCA) - $Ca_3Al_3(OH)_{12}$ - is used in the Bayer process as a filter aid during the pregnant liquor polishing. The filtration efficiency depends on some TCA characteristics, such as particle size distribution and morphology and how these particles are formed by physical-chemical reactions that take place during the alumina production. To reach a high performance in the filtration step, it is important to understand the influence of variables like source of calcium - CaO or $Ca(OH)_2$ - residence time, stirring speed and temperature in the TCA formation. This study is being developed as partnership CETEM/ Alunorte – Alumina do Norte do Brasil S.A., using liquor from the plant streams, to evaluate the efficiency of their process. Preliminary results shown that the formation of TCA particles begins partially, in the first minutes of reaction, and that its morphology and particle size distribution can change, during the experiment, depending on the particle size distribution of lime and impurities, which play an important role in the lime reactivity and in the kinetic of the TCA formation.

Aluminum Alloys: Fabrication, Characterization and Applications: Processing and Texture

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee
Program Organizers: Subodh Das, Phinix LLC; Steven Long, Kaiser Aluminum Corporation; Tongguang Zhai, University of Kentucky

Tuesday AM Room: 615
February 16, 2010 Location: Washington State Convention Center

Session Chair: Tongguang Zhai, University of Kentucky

8:30 AM

Concurrent Precipitation in Continuous Cast Al Alloys: Q. Zeng[1]; *Tongguang Zhai*[1]; [1]University of Kentucky

Continuous cast AA3004 Al alloy hot band was annealed at 420°C for 4 hours followed by cold rolling of 70% in reduction and full recrystallization at 399°C. The alloy showed a strong P texture. EBSD and TEM were used to study nucleation and growth of the P oriented grains, and dislocation structures in the alloy. It was found that the P oriented grains were preferably nucleated around coarse particles, especially in the regions where plastic strain was over a critical value. P oriented grains grew faster than grains with other orientations. P orientation was likely to be formed by lattice rotation from brass orientation. The transition from brass to P orientations might be caused by sudden release of a large number of dislocations which were pinned by fine precipitates around particles.

8:50 AM

Characterization of Novel Microstructures in Al-Ag-Cu Ternary Eutectic: *Amber Genau*[1]; Lorenz Ratke[1]; Markus Köhler[1]; [1]German Aerospace Center

While binary eutectics have been extensively studied and are generally well understood, the dynamics of three phases developing from a liquid make ternary eutectics far more complex. Samples at the ternary eutectic composition in the AlAgCu system were directionally solidified with relatively low growth velocities between 0.2 to 4.0 microns/sec in a thermal gradient of 3 K/mm to insure fully coupled growth. The structures produced in these samples were extensively studied using SEM, EDX and EBSD. Cross sectional and longitudinal images will be used to demonstrate the types of microstructures which form and the varying degrees of organization they exhibit. Several attempts are made to quantify these non-trivial structures, such as the distribution of nearest neighbor phases and the distribution of interphase spacings. We also analyze the effects of solid state diffusion on the volume fraction and composition of the phases, and present information on the crystal orientation relationships.

9:10 AM

Evaluation of Process Speed Effect in Aluminium Extrusion by Experiment and Simulations: *Barbara Reggiani*[1]; Lorenzo Donati[1]; Luca Tomesani[1]; [1]University of Bologna

In order to analyse metal forming processes and to avoid expensive trial-and-error experiments, finite element simulations are commonly utilized to investigate the material flow, temperature distribution and many other process-related issues. Nevertheless, the crucial role of experimental tests is still recognized due to difficulties encountered in achieving accuracy in the simulation of complex processes like hot aluminium extrusion. With this aim a periodical meeting between extruders and developers of FE tools was organized and a special benchmark session was set. The extrusion of 4 L-shaped profiles made by AA6082 aluminium alloy were investigated at two ram speed (0.5 and 5 mm/s respectively) through a die designed with different concepts for each extrusion orifices. This work presents the comparison between experimental data recorded during the benchmark trials and two FE codes in terms of maximum process load, final profiles lengths, profile distortions and exit temperatures.

9:30 AM

Processing of Forgings with Fine Crystalline Structure out of Commercial Aluminum Alloys: *Vadim Trifonov*[1]; [1]Institute for Metals Superplasticity Problems, Russian Academy of Sciences

Processing of fine crystalline structure in metallic materials provides improved mechanical properties. However, hot die forgings with fine crystalline structures are not used widely. Fine crystalline structure processing of hot deformed

materials occurring at annealing from static recrystallization is connected with high strains at low temperatures and is very labor intensive. The advisability of fine crystalline structure processing in forgings out of commercial aluminum alloys in terms of labor intense has been analyzed and ways of its reduction are proposed. The paper considers various schemes of fine crystalline structure processing including a combined process of static and dynamic recrystallization, and also gives examples of practical use of hot die fine crystalline forgings for production of specific parts. The paper shows features of fine crystalline structure transformation occurring in forgings depending on temperature-strain rate regimes of their following hot processing.

9:50 AM Break

10:05 AM

Growth Morphology of Eutectic under Unidirectional Solidification in Al-13%Si Alloys Containing Strontium and Magnesium: Hengcheng Liao[1]; Wanru Huang[1]; Shengqing Wu[1]; Mingdong Cai[2]; *Qigui Wang*[3]; [1]Southeast University; [2]Exova - Houston Laboratory; [3]Advanced Materials Engineering, GM Powertrain

Effect of addition of Sr and Mg in the alloy, solidification velocity and thermal gradient on growth morphology of eutectic in Al-13%Si alloy under unidirectional solidification was discussed in this paper. It was found that growth morphology of eutectic changed from planar into celluar, celluar /columnar and afterwards equiaxed in nonfacet-facet Al-Si system with increase in addition of Sr and Mg in the alloy. Solidification velocity and thermal gradient have an important influence on evolution of eutectic growth morphology in Al-Si system. Under unidirectional solidification, different morphologies of eutectic growth, such as planar interface, celluar, celluar/columnar and equiaxed eutectic, could be obtained by tailoring Sr and Mg contents in the alloy, solidification velocity and thermal gradient. The third solute content (Sr and Mg), solidification velocity and thermal gradient together determine the stability of eutectic growth interface, consistent with the classical constitutional supercooling theory on planar interface stability of eutectic growth.

10:25 AM

Influence of Cold Rolling and Equal-Channel Angular Extrusion on the Microstructural and Mechanical Behaviors of Al-40Zn Alloy: *Gencaga Purcek*[1]; Onur Saray[1]; Ibrahim Karaman[2]; [1]Karadeniz Technical University; [2]Texas A&M University

Al-40Zn alloy was subjected to cold rolling, and equal-channel angular extrusion (ECAE) up to four passes via route-A at 200°C with and without successive cold-rolling. The microstructural evolution and mechanical properties of the alloy were investigated. Rolling of the as-cast alloy to 50% thickness reduction resulted in a banded microstructure with mainly elongated grains. ECAE processing alone led to the elimination of the as-cast microstructure and formed a structure consists of elongated, ribbon shaped alpha-phase. Additional rolling after ECAE to 75% thickness reduction brought about further reduction in microstructure. Rolling of the as-cast alloy increased the strength in the expense of ductility. ECAE alone decreased the yield strength (from 239MPa to 231MPa after 4A) of the alloy but resulted in an exceptionally high elongation to failure (from 13% to about %34 after 4A). Additional rolling after ECAE increased both strength and ductility of the alloy as compared to identical rolling.

10:45 AM

Experimental Investigations on Influence of Process Parameters on Weld Strength of Friction Stir Welded Aluminium Alloy: *Krishnaiah Arkanti*[1]; Syed Yousuful Haq[2]; [1]Osmania University; [2]Steel Authority of India Limited

The effect of the welding speed on the microstructure, mechanical properties of friction stir welded joints has been investigated in the IS 737 aluminium alloy.The functional behavior of weldments is substantially determined by the nature of the weld strength characterized by the tensile strength, metallurgical behaviour, surface roughness, weld hardness and micro hardness. This project attempts to determine and evaluate the influence of the process parameters of FSW on the weldments. The Tensile strength, Vicker hardness and microstructure are considered for investigation by varying tool speed, tool feed and maintaining constant depth of penetration of weld. Experiments were conducted on IS 737 Aluminium alloy in a Vertical axis CNC milling machine by programming for every experiment. Results show strong relation and robust comparison between the weldment strength and process parameters.

11:05 AM

Optimization of Baking Hardening of Al 5052 Sheet by a Response Surface Method: Atiye Nekahi[1]; Kamran Dehghani[1]; Mohammad Ali Mohammad Mirzaie[2]; *Niloofar Kamkar Zahmatkesh*[1]; [1]Amirkabir University of Technology; [2]Modares University

In this study, the bake-hardenability of Al5052 was investigated. The specimens were subjected to different baking treatments according to the design experiments of response surface method. A model based on response surface method was designed to predict the optimal combination of baking parameters. Then, the tests were carried out according to the levels predicted by the model. The optimal baking parameters predicted were baking at 285°C for 35 min with 9% pre-strain.maximum bake hardening was 92 MPa which is in close agreement with the predicted results. The counter plots were obtained to study the interaction of process variables. The elliptical counter plots indicate that the variables exhibit strong interaction during the baking of. The multiple correlation coefficient (R2) of the regression models was about 0.98.This implies that more than 98% of the variability in the response can be explained by the model confirming the excellent accuracy of the model.

11:25 AM

Orientation Distribution Plots in a Cold-Rolled Heat-Treated Specimen of Aluminum Alloy 6061: Samuel Adedokun[1]; *Victor Ojo*[2]; [1]University of Lagos, Akoka-Yaba, Lagos, Nigeria; [2]Ladoke Akintola University of Technology

Aluminum alloy 6061 is used for applications such as structural application. A 10-mm thick sample of aluminum alloy 6061 was given 85% deformation by cold rolling. The rolling was carried out through a multi-step process until the 85% deformation was achieved. Samples from the rolled specimen were cut and heat treated at 200°C, 310°C, 350°C, 400°C, 450°C and 500°C at 10, 30, 60 and 100 minutes for each of the temperatures. Pieces of heat treated samples were cut, cold mounted in epoxy resin and polished to 3micron surface finish. Thereafter, the samples were scanned by a x-ray diffractometer equipped with texture goniometer and texture measurements were carried on the samples. Orientation plots of the samples were generated after analysis of the results. Results indicated that texture variations were according to the changes in temperature and time of the heat treatment.

11:45 AM

Comparison of Textures and Microstructures between AA3XXX Hot Bands from Three Different Casting Processes: *Xiyu Wen*[1]; Yansheng Liu[2]; Ningileri Shridas[2]; Tongguang Zhai[1]; Zhong Li[3]; [1]University of Kentucky; [2]Secat, Inc.; [3]Aleris International Inc.

Textures in AA3xxx hotbands made from three different casting routes respectively (twin belt casting (CC), the proprietary pellet cast process (HPC) and direct chill (DC) casting) were measured using XRD and ESBD, separately. The microstructures of these hot bands were also studied using optical and scanning electronic microscopies. It was found that the differences in texture and microstructure. In the CC hot band, there is a typical rolling texture with a well-developed ß fiber. From surface to more ¼ depth of HPC hot band, it was found that there is a strong {001}<110> shear texture. In the DC hot band sample, there is a high volume fraction of cube texture component compared to other two processes.

Aluminum Reduction Technology: Hall-Héroult Cell: Technology

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee
Program Organizers: Charles Mark Read, Bechtel Corporation; Gilles Dufour, Aluminerie de Deschambault

Tuesday AM Room: 608
February 16, 2010 Location: Washington State Convention Center

Session Chair: Alton Tabereaux, Consultant

8:30 AM Introductory Comments

8:35 AM
Development of the AP39: The New Flagship of AP Technology: Olivier Martin[1]; Laurent Fiot[1]; David Munoz[1]; Xavier Berne[1]; Claude Ritter[1]; [1]Rio Tinto Alcan

A new milestone has been achieved with AP30 technology. During the summer of 2009, the AP39 cell has been fully demonstrated at Saint-Jean de Maurienne (France). This new cell combines very high productivity (at 385 kA) with a high level of process performance (anode effect frequency, current efficiency and energy consumption) and has the potential to go beyond 400 kA. After the modelling and design phase, three years of testing has enabled the development of a reliable cell with appropriate process control parameters. Several measurement campaigns have confirmed the cell thermal, electrical and MHD equilibrium. However, in order to achieve world class performance, a very efficient process from pre-feasibility study to start-up and operation is also required. Along with the AP39 technology results, this paper describes the overall support provided to smelter projects throughout the world and the latest smelter project performances obtained.

9:00 AM
DX Pot Technology Powers Green Field Expansion: *Ali Al Zarouni*[1]; Marc Zelicourt[1]; Maryam Al Jallaf[1]; Kamel Alaswad[1]; Arvind Kumar[1]; Abdulla Al Reyami[1]; Vijay Kumar[1]; Dinesh Bakshi[1]; Jose Blasques[1]; Ibrahim Baggash[1]; [1]DUBAL

Dubal's potline 8 was commissioned in the year 2008 with DX pot technology. The present potline amperage is 370 kA, current efficiency has exceeded 95.5% and energy consumption is less than 12.95 kWh/kg of aluminium. Anode effect frequency is well below 0.02. This has been achieved through a balanced electrical, thermal and magneto-hydrodynamic cell design, implementation of optimal controllers and sound operating practices. The design has low investment cost per tonne of aluminium produced, lower instrumentation and automation cost and better fume capture efficiencies. Dubal's in house developed pot controller unit, a web based monitoring system for pots and an ORACLE data base for historical data complement the DX technology. The technology has been at the forefront of the 1.5 million tonne green field smelter coming up in Abu Dhabi (U.A.E). It is expected to be commissioned in December 2009.

9:25 AM
New Logistic Concepts for 400 and 500 kA Smelters: *Maarten Meijer*[1]; [1]Hencon

With today's scenario tools it is possible to de-bottle operational issues and as a benefit lower the cost per ton. In this presentation key stone technology's that influences the success and performance of this new operational approaches are presented. With the introduction of cells that run above 300kA new challenges are introduced in the reduction logistic of modern smelters. So far the amperage increase showed a logical trend of up scaling in auxiliary equipment like transporters and cranes. The latest products used in High Amperage smelters shows the dawn of an area where alternatives come available. The implementation of this new products resulted in the need for better tools in order to predict how this new operation perform. High Amperage operations give room for Smart cost saving production if you re-engineer the production chain. This leads to surprising new and innovative solutions for every day problems.

9:50 AM
The Pot Technology Development in China: *Zhu Jia ming*[1]; Yang Xiaodong[1]; Sun Kangjian[1]; [1]SAMI

Starting from 1996, the Shenyang Aluminium & Magnesium Engineering & Research Institute (SAMI) developed a series of prebaked pot technologies ranging from 160kA, 190/200kA, 230/240kA 280kA, 300kA, 350kA to 400kA pots. All of them have been applied to the industry field, which gives rise to total capacity of 12Mt of aluminium. These pot technologies were developed through effective numerical simulation modelling and by learning the success and failure of the engineering and operation of previous pot technologies. Numerical modelling, pot design, pot measurements and process control will be described in this article .The SY series pot technology has been validated for its technical performance and economy. SAMI is launching a new campaign to reduce more investment and improve pot performance.

10:15 AM Break

10:20 AM
The Newly Advancement of SY400 Pot: *Sun Kangjian*[1]; [1]Shenyang Aluminum and Magnesium Engineering and Research Institute

The newly advancement of SY400 pot Kangjian Sun Hongwu Hu Shenyang Aluminum & Magnesium Engineering & Research Institute Keywords: pot technology, technical improvements, Energy consumption, SY400 potAbstract: Starting from 2002, the Shenyang Aluminum & Magnesium Engineering & Research Institute (SAMI) developed SY350 pot technologies, after optimize the SY350 pots and increase the current to the 400kA without increase the anode dimension. These pot technologies were developed through effective numerical simulation modeling and by learning the success and failure of the engineering and operation of these pot technologies. SAMI optimize the SY350/400 technology and have been applied to the industry field and there are 17 new 400kA potlines are designed or constructed, 2 potlines in operation. The SY 400 pot technology has been validated for its technical performance and economy and become one of best availability pot technology in the world.

10:45 AM
Successful Commercial Operation of NEUI400 Potline: *Xiquan Qi*[1]; [1]Northeastern University Engineering and Research Institute, Co., Ltd

Development of large aluminum reduction technology is a complex system engineering. While overcoming the core technologies, more attentions shall be paid to structure optimization, environmental protection and energy saving. As a professional engineering and research institute in light metals industry, NEUI has overcome successfully, by carrying out self-developing and joint collaboration, some bottle-neck technologies which affect the development of high amperage reduction cells, such as the simulation of MHD stability, 3-D thermoelectric field, stress and rigidity status both in superstructure and potshell, gas fluid dynamics inside the cell, etc. Simulated and optimized with multi-advanced software, NEUI400 (I) reduction cells have been put into normal operation rapidly after startup, and the technical parameters are close or over the design indices. Thus, all the simulation technologies of NEUI are proved to be mature and reliable. Here, the development process of NEUI400(I) is presented and comparison is done with measurement data.

11:10 AM
Continues Advancement in Lanzhou Smelter: Sun Kangjian[1]; *Jun Chen*[2]; [1]Lanzhou Branch CHALCO; [2]Shenyang Aluminum and Magnesium Engineering and Research Institute

Lanzhou smelter is one of the first primary aluminum producers in china which found in 1958, the development of Lanzhou smelter is the epitome of china alumina primary aluminum industry. Various china typical technologies were in operation last 51 Years. Now only the typical pots technology SY200 and SY350/SY400 pot were operation, this two potline were build in last ten years. Lanzhou has make technical improvements using optimum practice include optimization of operational, optimization of process control system and application of slotted anodes as well as improvement of anode quality.

11:35 AM
Baking Start-up and Operation Practices of 400kA Prebaked Anode Pots: *Yungang Ban*[1]; Xiquan Qi[1]; Dingxiong Lv[1]; Jihong Mao[1]; Yu Mao[1]; Zhaowen Wang[1]; Zhongning Shi[1]; Bingliang Gao[1]; Xianwei Hu[1]; [1]Northeastern University Engineering and Research Institute Co. Ltd

This paper introduces the important points concerned with baking start-up and operation management of 400kA pots in China. The smelter was designed by Northeastern University Engineering & Research Institute Co., Ltd (NEUI), and adopted in modernization project of ZhongFu Industry Co. Ltd. The performance of NEUI400 pots have been continuously improved since the strat-up in August of 2008. The pots are currently operating at 403kA and have been in operation successfully for more than 10 months. Good performance obtained from the NEUI400 pots with high current efficiency, low energy consumption and low anode effect frequency.

12:00 PM Concluding Comments

Biological Materials Science: Mechanical Behavior of Biological Materials I: Nature-inspired Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: John Nychka, University of Alberta; Jamie Kruzic, Oregon State University; Mehmet Sarikaya, University of Washington; Amit Bandyopadhyay, Washington State University

Tuesday AM Room: 205
February 16, 2010 Location: Washington State Convention Center

Session Chairs: John Nychka, University of Alberta; Jamie Kruzic, Oregon State University

8:30 AM IOM Mehl Award Winner Announcement for Rob Ritchie

8:35 AM Keynote
***Institute of Metals/Robert Franklin Mehl Award Winner*: Nature-Inspired Structural Materials**: *Robert Ritchie*[1]; [1]University of California Berkeley

The structure of materials invariably defines their mechanical behavior. However, in most materials, specific mechanical properties are controlled by structure at widely differing length scales. Nowhere is this more apparent than with natural materials. Bone and nacre, for example, are sophisticated composites whose unique combination of mechanical properties derives from an architectural design that spans nanoscale to near-macroscopic dimensions; few engineering materials have such hierarchy of structure and properties. Unlike engineering composites where properties are invariably governed by the "rule of mixtures", the mechanical properties of many natural composite materials are generally far greater than their constituent phases. However, actually making such materials synthetically has proved to be extremely difficult, particularly in bulk form. Here we describe an approach, involving processing by ice-templating, to developing bulk ceramic-polymer nacre/bone-like structural materials with unprecedented strength/toughness properties. Indeed, we believe that these materials represent the highest toughness ceramics reported to date.

9:15 AM
Mechanical Properties of Saxidomus Purpuratus Shells: *Yang Wen*[1]; Zhang Guangping[2]; Li Xiaowu[1]; Marc Andre Meyers[3]; [1]Northeastern University; [2]Institute of Metal Research Chinese Academy of Science; [3]University of California, San Diego

The strength and fracture behavior of Saxidomus purpuratus biological shells were investigated by means of three-point bending and compression tests. It was found that the bending strength and fracture mode are a function of lamella thickness and angles between lamellae. The flexure strength of the shell ranges from 51 to 128 MPa, whereas the compressive strength ranges from 42 to 151 MPa. Weibull statistical analysis yielded mean yielding moduli of 5.45 and 3.97, and the mean characteristic stresses of ~101 MPa and ~72.76 MPa for bending and compression, respectively. The lamellar configurations are correlated with the strength to understand the orientational and positional effects on the mechanical properties of the shell. The distribution of bending strengths and fracture modes as a function of the position of the specimens taken from the whole shell was also examined. The relation between mechanical properties and microstructures of the shell was elucidated.

9:35 AM
Structure and Mechanical Properties of Armadillo Armor: *Irene Chen*[1]; Y. S. Lin[1]; P.-Y. Chen[1]; Marc Meyers[1]; J. McKittrick[1]; [1]University of California, San Diego

The armadillo has a unique protective shell-like armor, called the osteoderm. It has a bony components to result in distinctive mechanical properties. The pectoral and pelvic shields of the osteoderm have polygonal (hexagonal) tiles with diameters of 5 mm; the banded and tail shields have rectangular shapes. Optical microscopy reveals that laterally oriented osteons are found in the papillary region within the skin layer of 2 mm in depth and are mainly collagen fibers. The surface layer of the epidermis is approximately 200 ìm and is composed of keratin. The collagen network within the layer provides mechanical toughness to the entire structure. The tough and highly mineralized polygonal tiles are bridged with a network of collagen fibers which give resilience to the whole structure. The mechanical properties (tensile, flexure, impact) will be discussed of the different shell components. Research support: NSF Biomaterials Program (Grant DMR0510138).

9:55 AM
Battle in the Amazon: Araipamas (Pirarucu) vs. Serrasalmus (Piranha): *Marc Meyers*[1]; Y. S. Lin[1]; P.-Y. Chen[1]; E. A. Olevsky[2]; J. McKittrick[1]; [1]University of California, San Diego; [2]San Diego State University

The Araipamas is a large fish (up to 200 kg) living primarily in Amazon basin lakes. It is covered with scales having up to 10 cm length that provide protection against the piranha, the principal predator. The scales are highly mineralized and are a laminate composite that can undergo significant non-elastic deformation prior to failure, providing significant toughness. We performed mechanical tests on the scales and teeth and characterized both. The piranha teeth form triangular arrays creating a guillotine action that is highly effective in slicing through muscle. The cutting and puncturing ability of the piranha teeth are evaluated and it is demonstrated that they cannot penetrate the Araipamas scales. Research funded by NSF DMR Biomaterials program (Grant DMR 0510138).

10:15 AM Break

10:25 AM Invited
Biological Materials, Biomaterials and Biomimetics: *Ulrike Wegst*[1]; [1]Drexel University

Biological materials have exceptional mechanical properties which still cannot easily be emulated by man-made materials. With an informed evaluation of their hierarchical structure, properties and function we wish to identify their principles of optimisation and to apply these in the development of novel and improved man-made materials. Illustrated will be how the mechanical efficiency of biological materials can be evaluated and compared with engineering materials. A variety of methods for the structural and mechanical characterisation of biological materials, ranging from synchrotron-based X-ray microtomography with phase-contrast to methods for in situ mechanical testing in SEM or FIB, will be presented. It will further be shown how, by collating principles of optimisation and function, a software-based design tool, the Biomimetic Design Guide, can be created to aid bio-inspired engineering and a systematic knowledge transfer from nature to technology. Finally, an example for biomimetic material design will be show to illustrate this approach.

10:55 AM
What it Takes to be Light as a Feather: *Sara Bodde*[1]; Joanna McKittrick[1]; Marc Meyers[1]; [1]University of California, San Diego

Feathers are the most distinguishable feature of all modern Aves, and they are considered to be the most complex of integumentary appendages of all vertebrata. We have observed structural hierarchy from mesoscale to nanoscale. The primary shaft, or rachis, and secondary structures, barbs, are foam-filled having cellular solid cores. Because flight feathers play a role in the generation of thrust and lift forces, we have focused on fitness of remiges, wing feathers, and retrices, tail feathers. We have conducted tensile tests of the rachis cortex, flexure tests of intact rachis sections, and compression tests of foam sections of rachis in order to assess optimization of structural adaptations for feeding ecology of selected species. Research support: N.S.F. Biomaterials Program (Grant DMR 0510138)

11:15 AM

Microstructural Features that Toughen Horn: *Katya Novitskaya*[1]; Ana Castro-Ceseña[2]; Luca Tombolato[1]; Po-Yu Chen[1]; Steven Lee[1]; Gustavo Hirata[3]; Joanna McKittrick[1]; [1]University of California, San Diego; [2]Centro de Investigación Científica y de Educación Superior de Ensenada; [3]Center for Nanoscience and Nanotechnology

Horns from the family bovidae are tough biological materials containing crystalline a-keratin nanofibers embedded in an amorphous keratin matrix. The horn structure is a laminated structure, with the laminates stacked from the dorsal to the ventral side of the horn. Optical microscopy images showed that the horn structure is composed of aligned empty tubules with diameter of 12-14 μm and 100-125 μm length. TEM analysis revealed the existence of a-keratin intermediate filaments, 12 nm in diameter, that bridge the laminates, which aids in resisting delamination. Deformation mechanisms such as microbuckling of the laminates, tubule closure, crack deflection and nanoscale filament bridging, provides the horn with its extraordinary toughness. Quasi-static and dynamic property behavior will be discussed. This research is supported by ARO Grant W911F-08-1-0461 and NSF Grant DMR 0510138.

11:35 AM

Structural Investigations on Demineralized and Deproteinated Bone: *Ana Castro-Ceseña*[1]; Po-Yu Chen[2]; Gustavo Hirata[3]; Damon Toroian[2]; Paul Price[2]; Joanna McKittrick[2]; [1]Centro de Investigación Científica y de Educación Superior de Ensenada; [2]University of California, San Diego; [3]Center for Nanoscience and Nanotechnology

Investigations of the microstructural and mechanical properties of elk antler and bovine femur bone were carried out on demineralized and deproteinated samples. Small pieces of compact and cancellous bone were demineralized or deproteinated at different degrees by aging in various solvents. Structural, chemical, and mechanical properties of demineralized and deproteinated antler and bone were studied by micro-CT, SEM, HR-TEM, IR, Raman spectroscopy and AFM. Both the demineralized and deproteinated bones appeared identical at the macro-scale. At the micro-scale, SEM images showed that the minerals are aligned in a coherent manner, forming a continuous network. The chemical composition change at different degrees of demineralization and deproteinization and their relation to mechanical properties were also examined. This research is supported by the National Science Foundation grant DMR 0510138.

11:55 AM

Arthropod Cuticle: A Biological Multifunctional Composite Used As Template for Multidisciplinary Nano-To-Macro-Scale Hierarchical Modeling: *Martin Friak*[1]; Michal Petrov[1]; Svetoslav Nikolov[1]; Christoph Sachs[1]; Helge Fabritius[1]; Pavlina Elstnerova[1]; Duancheng Ma[1]; Liverios Lymperakis[1]; Dierk Raabe[1]; Sabine Hild[2]; Andreas Zigler[3]; Joerg Neugebauer[1]; [1]Max Planck Institute for Iron Research; [2]Johannes Kepler University Linz; [3]University of Ulm

Biological structural materials receive increasing attention by material science because they have been optimized during evolution and they are therefore ideally suited to study the efficiency of nature's design principles. These materials differ fundamentally from most man-made structural materials in being structurally heterogeneous by combining different in/organic constituents into composites with hierarchical organization. We propose a hierarchical model for the prediction of the elastic properties of a mineralized arthropod cuticle using ab initio calculations to find the elastic properties at the nanoscale and hierarchical homogenization performed in a bottom-up order to find the cuticle properties at all hierarchy levels. Our results suggest that the mineral-protein matrix possesses a microstructure (so-called symmetric cell material) which exhibits extremal properties in terms of stiffness. We also discuss the role of chitin and the multifunctional optimization of the cuticle in terms of trade off between stiffness and transport capacity of the pore canal system.

Bulk Metallic Glasses VII: Alloy Development and Application I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee

Program Organizers: Peter Liaw, The University of Tennessee; Hahn Choo, The University of Tennessee; Yanfei Gao, The University of Tennessee; Gongyao Wang, University of Tennessee

Tuesday AM Room: 213
February 16, 2010 Location: Washington State Convention Center

Session Chairs: J. Eckert, IFW Dresden; Marios Demetriou, California Institute of Technology

8:30 AM Invited

Strengthening of Ti-Base Glass-Forming Alloys by Microstructure Design: *J. Eckert*[1]; [1]IFW Dresden

Ti-alloys have potential use for automotive, aerospace, and biomedical applications due to the low density of the Ti and high corrosion resistance but their strength only slightly exceeds 1000 MPa with plastic strain to failure of 10–15%. The strength of these conventional alloys is quite low compared to the strength (~2000MPa) of Ti–bulk glassy/nanostructured alloys. The amorphous/nanostructured alloys usually lack ductility, and an additional toughening phase is needed in the microstructure to improve plasticity. Therefore, it is of strong interest to develop composite microstructures.Using different Ti-base alloy systems as examples, we demonstrate the beneficial effect of composite microstructures on the mechanical performance of the alloys. We empha-size the possibilities to tailor different types of composite microstructures in favor of either strength or ductility, or a combination of both, and also discuss the ability to syn-thesize in situ composite microstructures in bulk form through inexpensive processing routes.

8:50 AM

Novel Semi-Solid Processing Techniques for Metallic Glass Matrix Composites: *Douglas Hofmann*[1]; Henry Kozachakov[2]; Hesham Khalifa[3]; Joseph Schramm[2]; Marios Demetriou[2]; Kenneth Vecchio[3]; William Johnson[2]; [1]Liquidmetal Technologies; [2]California Institute of Technology; [3]University of California, San Diego

Bulk metallic glasses (BMGs) are a unique class of materials known for their high strengths, large elastic limits and amorphous microstructure. As structural materials however, BMGs lack tensile ductility, exhibit low fracture toughness and have poor fatigue life, limiting their potential uses. Recently, it has been shown that semi-solidly processed BMG matrix composites can have mechanical properties equaling or surpassing the best crystalline materials. Unfortunately, due to the higher viscosity of semi-solid liquids, these alloys are difficult to die cast. In this work, we develop new processing techniques which allow us to create complex net-shapes from highly-toughened BMG matrix composites. We expect that this work will provide a foundation for future commercialization of structural BMG components.

9:00 AM Invited

High Performance Structures Made of Ductile-Phase Reinforced Metallic Glass: *Marios Demetriou*[1]; Joseph Schramm[1]; Douglas Hofmann[1]; William Johnson[1]; [1]California Institute of Technology

Ductile-phase reinforced metallic glasses with optimized microstructures are found to exhibit mechanical properties such as strength, ductility, and toughness on par with the highest-performance engineering materials like ferrous metals and titanium alloys. Unlike traditional engineering metals however, ductile-phase reinforced metallic glasses have a relatively low melting point which enables fabrication of high precision parts directly from the melt. In this presentation, periodic cellular (honeycomb) structures made of ductile-phase reinforced metallic glass will be introduced. Structures of different architectures and various relative densities will be presented, and structure-property relations will be discussed and compared to conventional steel honeycombs. For a given relative density and structure design, the present cellular structures outperform steel honeycombs in terms of strength and energy absorption by a factor of about 4, a consequence of the high yield strength and large process-zone size of

the ductile-phase reinforced metallic glass enabling large plastic deformability at high plateau stresses.

9:20 AM

Applications for Amorphous Metals in Reactive Materials: *Alan Brothers*[1]; [1]Mainstream Engineering

Amorphous metals have been considered for a wide variety of applications, ranging from solar wind catchers to cellular phone housings. This presentation will evaluate amorphous metals for a new application – reactive materials. Their effects on the key characteristics of reactive materials, such as energy density, energy release rate, processing and handling safety, and mechanical properties, will be discussed. The effects will then be illustrated using experimental data from a reactive Al-based amorphous metal matrix composite material, including calorimetric heats of reaction, flame speeds, sensitivity to electrostatic discharge and impact, and microhardness.

9:30 AM Invited

Thermodynamics and Stability of Nanoglasses with Tunable Atomic Structure: Basic Ideas and First Results: *Hans Fecht*[1]; [1]Ulm University

Nanoglasses can be considered as highly disordered non-crystalline materials with a tuneable atomic structure. They can be generated by introducing interfaces into metallic glasses on a nanometer scale. Interfaces in metallic glasses delocalize upon annealing close to Tg so that the free volume associated with these interfaces spreads throughout the entire volume of the glass. In fact, by controlling the spacing between the interfaces introduced as well as the degree of their delocalization the atomic structures and density (and hence all structure/density dependent properties) of nanoglasses may be controlled. First results using BMG type materials will be presented. The material here is produced either by (i) inert gas condensation and full compaction of non-crystalline clusters and (ii) by production of primary and secondary shear bands using extreme plastic deformation methods such as high pressure torsion.

9:50 AM

Nanofabrication with Metallic Glasses: *Golden Kumar*[1]; Shiyan Ding[1]; Jan Schroers[1]; [1]Yale University

Homogeneous and isotropic nature of metallic glasses makes them ideal candidates for nano-scale applications. The ability to form metallic glasses on the nanoscale (<100 nm) has been limited. This is because of enormous repulsive capillary forces which need to be overcome. We have recently shown that manipulation of metallic glasses on nanometer scale is controlled by the wetting of the metallic glass on the mold. Wetting can be controlled by the mold-metallic glass combination. In this study we evaluated the wetting behavior and capillary forces between different metallic glasses and mold materials. For some metallic glass mold combinations features as small as 13 nm with an aspect ratio exceeding 20 can be imprinted on the metallic glass. The unusual softening behavior of metallic glasses can be utilized for a versatile toolbox for nanofabrication. Applications of nanofabrication with metallic glasses are discussed and examples given.

10:00 AM Break

10:10 AM Invited

Development of Bulk Metallic Glasses with High Plasticity Using the Surface Nano-Crystallization: *Jian Lu*[1]; Ji Tang Fan[1]; Qing Wang[1]; Yuan Hao Huang[1]; Hao Jiang[1]; [1]The Hong Kong Polytech University

The enhancement of the plastic deformation capacity is a critical issue for the development of the bulk metallic glasses (BMG). This presentation summarizes the recent research works carried out in the field of high plasticity BMG using the surface nano-crystallization. A novel route to form the BMG with high plasticity property by subjecting the metallic glass to the surface mechanical attrition treatment (SMAT) will be presented. A structural gradient consisting of submicron-scale crystallization on the top surface to inside fully amorphous metallic glass matrix was obtained. Its compression plasticity was thus greatly improved compared to that of the as-cast metallic glasses. For example, the maximum compression strain of the treated BMG may reach a level of 10% for a Zr-Cu-Al based BMG without major sacrifice of the strength. The effect of the formation of nanograins and the compressive residual stresses on the compression plasticity will be discussed.

10:30 AM

Bulk Metallic Glasses and the Composites Fabricated by Microwave-Induced Heating and Sintering: *Guoqiang Xie*[1]; Song Li[1]; Dmitri V. Louzguine-Luzgin[1]; Motoyasu Sato[2]; Akihisa Inoue[1]; [1]Tohoku University; [2]National Institute for Fusion Science

Microwave (MW)-induced heating and sintering process has attracted increasing attention due to its significant advantages in material processing. MW radiation causes internal heating of the materials, and it is a volumetric heating, so that the lower temperatures and shorter times can be used compared to those applied at conventional heating processes. In this study, we investigated sintering behavior of Cu-, Ni-, and Fe-based glassy alloy powders, as well as their mixed powders blended with crystal or polymer powders, using MW-induced heating and sintering process in a single mode 915 MHz MW applicator in a separated electric (E) field and magnetic (H) field. These powders could be heated well in the H-field, but not heated enough in the E-field. The bulk sintered specimens with retention of a glassy phase were obtained. Addition of Sn particles reduced the sintering temperature, and promoted the densification of the sintered glassy alloy specimens.

10:40 AM Invited

Formation and Properties of New Au-Based Bulk Glassy Alloys with Ultralow Glass Transition Temperature: *Wei Zhang*[1]; Hai Guo[2]; Mingwei Chen[3]; Yasunori Saotome[1]; Chunling Qin[3]; Akihisa Inoue[4]; [1]Institute for Materials Research, Tohoku University; [2]Graduate School, Tohoku University; [3]WPI, Advanced Institute for Materials Research, Tohoku University; [4]Tohoku University

It has been reported that Au-based Au-Cu-Si-Ag-Pd bulk glassy alloys (BGAs) exhibited high glass-forming ability (GFA), low glass transition temperature (Tg) of about 130°C, large supercooled liquid region (ΔTx), and good processibility. Recently, we developed new Au-based BGAs with lower Au contents of 35-45 at.%, which had large ΔTx, low Tg, higher GFA with sample critical diameters (dc) of 2-6 mm, and high fracture strength over 1000 MPa. More recently, we investigated the thermal stability and GFA of the alloys with higher Au contents of 60-75 at.% in quaternary Au-Cu-Si-Ag (Pd) systems. It was found that the glassy alloys show ultralow Tg of 66-98°C, large ΔTx of 36-55°C, and high GFA with dc of 2-5 mm. The BGAs exhibited good mechanical properties, excellent corrosion resistance in 1N HCl and H2SO4 solutions, high stability at room temperature, strong oxidation resistance, and excellent thermoplastic formability at temperature below 100°C.

11:00 AM

Nanoglass Formation and Properties Studied by Molecular Dynamics Simulations: *Daniel Sopu*[1]; Karsten Albe[1]; Herbert Gleiter[2]; [1]TU-Darmstadt; [2]Research Center Karlsruhe

Nanoglasses are a new class of material which can be synthesized by consolidating glassy nanoparticles. Molecular dynamics simulations are presented which provide a detailed picture of nanoglass formation. The results proof the existence of interfaces in the nanoglass. By comparing simulations for covalently bonded Ge nanoglass and metallic CuZr nanoglass, the delocalization of the interfacial free volume is analyzed. Initially, the interface relaxation is driven by homogeneous plastic flow depending on the materials flow strain. Using pressure dependent diffusion coefficient calculated from MD simulations we further estimate the times scales needed for delocalization driven by thermally activated diffusion. In addition, we investigate the phonon density of states and the related thermal properties of nanoglasses and compare to bulk amorphous and nanocrystalline samples.Our results suggest that the density distribution within a nanoglasses can be adjusted by the initial particle size and chemical composition as well as by the annealing conditions.

11:10 AM

Fabrication and Mechanical Characterization of Zr-Based Bulk-Metallic-Glass-Matrix Composites: *Junwei Qiao*[1]; Yong Zhang[1]; [1]USTB

A series of Zr-Ti-Cu-Be-Nb bulk-metallic-glass-matrix composites were fabricated by the Bridgman solidification with different withdrawal velocities. By adjusting the withdrawal velocities, the scale and the volume fraction of dendrites dispersed in the glass matrix were optimized effectively. Under the withdrawal velocity of 1.0 mm/s, the composite materials exhibited the large compressive plasticity and the high fracture strength upon quasi-static loading. Additionally, the deformation and fracture behavior of the composites upon dynamic compressive loading were investigated.

11:20 AM

Synthesis of $Cu_{50}Zr_{50}$ Bulk Metallic Glasses Composites by Spark Plasma Sintering: *Zhihui Zhang*[1]; Troy Topping[1]; Ying Li[1]; Yizhang Zhou[1]; Enrique Lavernia[1]; [1]University of California, Davis

Annealing induced embrittlement often represents a challenge to synthesize bulk metallic glasses (BMGs) with enhanced ductility using powder metallurgy techniques. Spark plasma sintering (SPS) uniquely provides a fast heating rate (400 °C/min) and accurate temperature control to study the effects of structural relaxation and partial crystallization on the mechanical properties of BMGs. In this study, the crystallization behavior during SPS and the corresponding mechanical properties of $(Cu_{0.5}Zr_{0.5})_{100-x}Al_x$ (x=0, 2.5, 5, 7.5 and 10) BMGs were investigated. In the supercooled liquid region, the SPS consolidated $(Cu_{0.5}Zr_{0.5})_{100-x}Al_x$ alloys undergo partial crystallization. The $Cu_{50}Zr_{50}$ binary alloy crystallized into $CuZr_2$ and $Cu_{10}Zr_7$ while the Al-containing alloys crystallized into $CuZr_2$, CuZr and $AlCu_2Zr$. However, below the glass transition temperature, fcc-Cu nanocrystals formed in the SPS consolidated $Cu_{50}Zr_{50}$ alloy. The results showed that the fracture strength and ductility were enhanced due to the precipitation of fcc-Cu nanocrystals. The fracture behavior of the $(Cu_{0.5}Zr_{0.5})_{100-x}Al_x$ alloys was discussed.

11:30 AM

Synthesis of Amorphous/Amorphous and Amorphous/Crystalline Composites in Phase Separating Gd-Ti-Al-(Co/Cu) Alloys: *Sung Woo Sohn*[1]; Wan Yook[1]; Hye Jeong Chang[2]; Won Tae Kim[3]; Do Hyang Kim[1]; [1]Yonsei University; [2]Oak Ridge National Laboratory; [3]Cheongju University

In the multi-component systems exhibiting high glass forming ability, the difference in the heat of mixing between some binary combinations can be large, possibly leading to phase separation onto two glass phases. In the present study, results on the phase separation in Gd-Ti-Al-(Co/Cu) alloys where Gd have positive heat of mixing with Ti will be presented, showing that phase separating metallic glass system can offer a unique opportunity for designing composites with hierarchical microstructure. The existence of miscibility gap and spinodal decomposition curve was examined by thermodynamic calculation using CALPHAD method. Based on these thermodynamic calculation results, metastable pseudo-binary liquid phase diagram including miscibility gap and spinodal decomposition curve could be obtained. Considering the liquid phase diagram, we could predict and control the microstructure. Furthermore, the deformation behavior of interconnected-type phase separated alloys has been investigated, emphasizing that the different length-scale plays a role in the mechanical properties.

11:40 AM Invited

Air-Oxidation of a (Zr55Cu30Al10Ni5)98Er2 Bulk Metallic Glass at 350-500°C: *Wu Kai*[1]; P.C. Kao[1]; I.F. Ren[1]; P.C. Lin[1]; Z.H. Hsiao[1]; D.W. Xing[2]; P.K. Liaw[3]; [1]Institute of Materials Engineering, National Taiwan Ocean University; [2]Department of Materials Science, Harbin Institute of Technology; [3]Department of Materials Science and Engineering, The University of Tennessee, Knoxville

The oxidation behavior of a (Zr55Cu30Al10Ni5)98Zr2 bulk metallic glass (Zr-2Er BMG) was studied over the temperature range of 350-500°C in dry air. In general, the oxidation kinetics of the Zr-2Er BMG followed a two-to three-stage parabolic-rate law, and the parabolic-rate constants (kp values) fluctuated with increasing temperature. It was found that the oxidation rates of the Zr-2Er BMG are faster than those of the Er-free glassy alloy, indicative of a poor oxidation resistance. Very likely, the addition of 2% Er increased the concentration of oxygen vacancies in the defective ZrO2-lattice, which in turn enhanced inward diffusion of oxygen, thereby leading to the faster oxidation rates of the Zr-2Er BMG. The scales formed on the Zr-2Er BMG were strongly temperature-dependent, consisting exclusively of tetragonal ZrO2 (t-ZrO2) at 350°C, while minor amounts of monoclinic ZrO2 (m-ZrO2), CuO, Al2O3, and Er2O3 were detected at T > 400°C.

12:00 PM

Artificial Microstructures as a Tool Box to Study Shear Band Stabilization in BMG Composites: Golden Kumar[1]; *Jan Schroers*[1]; [1]Yale University

The current understanding in mechanical properties of BMG composites is that the interaction between shear bands and heterogeneities plays a critical role in stabilizing shear bands and preventing crack formation. The crack tip plastic zone size was suggested to be a crucial length scale influencing shear band interaction with the second phase. However, the properties of the heterogeneities—their size, distribution, and spacing—can also have an effect on this length scale and therefore on the resulting tensile ductility and toughness. With current BMG composite synthesis techniques, it is difficult to control size, shape, distribution, and spacing of heterogeneities independently. However, our recent progress in thermoplastic forming allows fabricating artificial microstructures from BMGs where the dimensional aspects of the heterogeneities can be varied—controllably and independently. The results from tensile tests on samples with a different size and distribution of second phase are reported.

Carbon Dioxide and Other Greenhouse Gas Reduction Metallurgy - 2010: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Energy Committee

Program Organizers: Neale Neelameggham, US Magnesium LLC; Ramana Reddy, The University of Alabama; Jiann-Yang Hwang, Michigan Technological University; Jean-Pierre Birat, Arcelor Mittal; Kotaro Ogura, Yamaguchi University

Tuesday AM Room: 310
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Mahesh Jha, US Dept of Energy; Lifeng Zhang, Missouri University

8:30 AM Introductory Comments

8:35 AM Invited

Overview of Removal Methods for CO2 and Other Greenhouse Gases and Details of the Method Using Non-Thermal Plasma: Marcela Morvová[1]; *Imrich Morva*[1]; Mario Janda[1]; [1]Comenius University

The possibilities of greenhouse gas mitigation using various methods are brought out. The special case of utilization of mixture of CO2 with other greenhouse gases in synergistic way inside non-thermal plasma based system is described in details. Measurements on pilot system comprising pyrolysis chamber as alternative energy system (production of hydrogen and liquid tar) were made for various biomass and waste as the input material. The exhaust from this system was treated with non-thermal plasma gap suitable for up to 650Nm3/hour of exhaust gas flow. Condensed portion of exhaust is liquefied in distillation unit, produced hydrogen is on-line stored inside nanosized carbon of our own production. The CO2 removal efficiency varies depending on exhaust composition between 45 to 98%. Other oxides as CO, NOx, SOx and several hydrocarbons and VOC are also removed. The main product connected with CO2 removal is solid powder of proteinoid nature.

9:15 AM

DOE's Industrial Energy Efficiency Grand Challenge Solicitation to Support Development of Technologies to Reduce Energy Intensity and Greenhouse Gas Emissions: *Mahesh Jha*[1]; Bhima Sastri[1]; [1]U. S. Department of Energy

U. S. industry consumes approximately 32 quadrillion Btu of energy, almost a third of all energy used in the country, and emits about 1.6 billion metric tons of CO2 per year. One of the mission of Energy Efficiency and Renewable Energy (EERE) division of U.S. Department of Energy (DOE) is to strengthen America's energy security, environmental quality and economic viability by enhancing the energy efficiency and productivity of the industrial sector. In support of this mission, the Industrial Technologies Program (ITP) issued "Industrial Energy Efficiency Grand Challenge" solicitation to seek, select and fund cost-shared development of transformational industrial processes and technologies that can reduce the energy intensity and greenhouse gas emissions from energy-intensive industries. This paper briefly describes the solicitation and selection process and presents a summary of the selected technologies to be developed.

9:40 AM

Sunshine to Petrol: A Metal Oxide-Based Thermochemical Route to Solar Fuels: *James Miller*[1]; Richard Diver[1]; Nathan Siegel[1]; Eric Coker[1]; Andrea Ambrosini[1]; Daniel Dedrick[1]; Mark Allendorf[1]; Gary Kellogg[1]; Roy Hogan[1]; Ellen Stechel[1]; Ken Chen[1]; [1]Sandia National Laboratories

Converting carbon dioxide and water to hydrocarbons is an attractive option for storing solar energy and impacting atmospheric CO_2 concentrations. Thermochemical approaches for this conversion are potentially highly efficient as they avoid the inherent limitations of photosynthesis and also sidestep the solar-to-electric conversion necessary to drive electrolytic reactions. Solar-driven two-step metal oxide-based thermochemical cycles for producing the components of syngas, CO and H_2, from CO_2 and H_2O are the basis of the "Sunshine to Petrol" project. Multi-cycle production of both H_2 and CO has been demonstrated over several iron- and cerium-based compositions fabricated into monolithic pieces both in the laboratory and at the National Solar Thermal Test Facility. Progress in advancing the chemistry, understanding, and fabrication of materials for this application will be reported. Additionally, systems analysis and progress towards further demonstrating the reactions in a unique and continuous solar-driven reactor, the counter-rotating-ring receiver reactor recuperator or CR5, will be reported.

10:05 AM Break

10:15 AM

Synthetic Fuel Production Utilizing CO2 Recycling as an Alternative to Sequestration: *Joseph Hartvigsen*[1]; S Elangovan[1]; Lyman Frost[1]; Carl Stoots[2]; James O'Brien[2]; J. S. Herring[2]; Manohar Sohal[2]; Grant Hawkes[2]; [1]Ceramatec Inc; [2]Idaho National Laboratory

Ceramatec, Inc and the Idaho National Laboratory are applying solid oxide fuel cell (SOFC) technology to syngas production by high temperature co-electrolysis of CO2 and steam. This technology utilizes SOFC stacks to electrochemically extract oxygen from steam and CO2, leaving hydrogen and carbon monoxide suitable for fuel synthesis. The product H2 to CO ratio of can be controlled to match the desired type of synthetic fuel product by varying the feed ratio. The resultant synthesis gas has been used to produce methane and Fischer-Tropsch liquid fuels.Concentrated industrial emitters of CO2, such as metallurgical reduction furnaces, cement kilns and fossil power plants can provide a valuable feedstock for a synthetic fuels industry, which incorporates energy from nuclear, solar, wind and hydropower sources. Widespread implementation of synfuel production will enable a greater reliance on intermittent renewable energy than can be accommodated by conventional electric demand profiles, while increasing our energy security.

10:40 AM

Development of Self-Reduction as the Future of Metals-Making Technology: *Jose Noldin*[1]; D'Abreu José[2]; [1]Tecno-Logos S/A; [2]Catholic University (PUC-Rio)

This paper will discuss the role of self-reduction as the technology of choice for metals production in the future. The main characteristics and fundamentals of this promising technological route will be presented, followed by a discussion on existing and developing technologies, potential impacts on raw materials requirements and economical and environmental benefits, with special focus on the reduction of CO2 emissions during metals production.

11:05 AM

Synthesis of Zeolitic Imidazolate Frameworks for Adsorbing Carbon Dioxide: Jinghua Zou[1]; *Huimin Lu*[1]; Min Li[1]; [1]Beihang University

Zeolitic imidazolate frameworks (ZIFs) are new type of porous materials, form inerratic polyhedral in three-dimensional with large cages and small apertures, and appear structural diversity. Their hole size can be adjusted. These materials have high selective adsorption capacity to carbon dioxide. In this paper, zeolitic imidazolate frameworks were synthesized by solvothermal synthesis method with dimethyformide(DMF) as solvent and structure-directing agent, divalent metallic ions as cation, and imidazole as ligand. The crystals were characterized with XRD, SEM for their crystal structures and physical properties, the adsorption of the crystals for carbon dioxide was also tested, and the result suggests that these porous materials have a capacity of 1.35ml/g for storing carbon dioxide at 273 Kelvin under ambient pressure.

11:30 AM

Photochemical and Photo Electrochemical Conversion of Carbon Dioxide to Methanol Using Nanotubular TiO2: *Manoranjan Misra*[1]; S. Mohapatra[1]; [1]University of Nevada

Global warming due to the emission of CO2 is a serious environmental concern. The atmospheric concentration of CO2 is about 384 ppm by volume and 3.0 □ 102 tonnes by weight. This paper discusses the potential of converting the CO2 into alternate fuels. CO2 feedstocks can be converted into fuels using band-gap engineered TiO2 nanotubes as a catalyst in the presence of solar light. Since no external energy is required, the energy saving would be in the order of 0.1-0.24 kwh/mole of CO2 as compared to electrolytic conversion.

Cast Shop for Aluminum Production: Grain Refinement, Alloying, Solidification and Shape Casting

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee

Program Organizers: John Grandfield, Grandfield Technology Pty Ltd; Pierre Le Brun, Alcan Voreppe Research Center

Tuesday AM Room: 609
February 16, 2010 Location: Washington State Convention Center

Session Chair: Rein Vainik, Swerea KIMAB

8:30 AM

On the Mechanism of Grain Refinement by Ultrasonic Melt Treatment in the Presence of Transition Metals: *Dmitry Eskin*[1]; Tetyana Atamanenko[2]; Liang Zhang[2]; Laurens Katgerman[2]; [1]Materials Innovation Institute; [2]Delft University of Technology

Ultrasonic melt treatment is known to induce grain refining in aluminum alloys. The degree of grain refinement is strongly linked to the stage of solidification when the treatment is applied and on the alloy compoistion. In the latter case the presence of grain refiners is important. In this paper it is shown that very strong grain refining can be achieved in aluminum and its commercial alloys when the ultrasonic treatment is combined with the introduction of Zr and Ti. Ultrasonic processing is performed in the temerature range of the primary solidification of an intermetallic phase, i.e. normal casting temperatures of aluminum alloys. Dual mechanism is discussed involving (1) the dipersion and refinement of primary intermetallic particles that act as solidification sites and (2) growth restriction by the transition metal(s) that are present in the liquid phase.

8:55 AM

Impurities in Al-5Ti-1B (wt.%) Grain Refiner Rod: Brian McKay[1]; Georg Nunner[1]; Georg Geier[2]; *Peter Schumacher*[1]; [1]University of Leoben; [2]Austrian Foundry Institute

A "standard" and a "poor" Al-5Ti-1B (wt.%) grain refiner rod have been examined using computed tomography (CT) and scanning electron microscope (SEM) equipped with an energy dispersive x-ray (EDX) spectrometer. The refining potency of each rod was checked using the TP1 test. CT and SEM results from the "poor" refiner rod revealed the presence of Al_3Ti agglomerates and residual unspent salts. These findings are indicative of an inefficient, non-optimal manufacturing process. Results from the TP1 test showed the "standard" rod to be more effective in promoting refinement, as expected. The quality of the grain refiner rod used in industrial practice is therefore vital as the grain size, feedability, cleanliness, uniformity of microstructure and integrity of the as-cast part may all be deleteriously affected. Moreover, a well controlled manufacturing process is important for the production of cleaner grain refiner as this can indirectly improve the quality of castings.

9:20 AM

Experience with Production Scale Usage of Optifine – A High Efficiency Grain Refiner: *John Courtenay*[1]; Rein Vainik[2]; [1]MQP Limited; [2]Swerea KIMAB

Optifine is a new grain refiner developed to give maximum grain refinement together with high consistency and good cleanliness. Laboratory tests, in combination with full scale trials, have shown that this grain refiner generally is at least twice as efficient than the standard grain refiners used today. This means

that at least two times more nuclei are active during the nucleation process and a fine grain can be obtained in the final cast with a much lower addition rate. This has now been demonstrated in full scale at Hulamin cast house in South Africa, where Optifine is used for all casts.

9:45 AM

Effects of Cooling Rate on Microstructure in En-Ac43000 Gravity Castings and Related T6 Mechanical Properties: *Ivan Todaro*[1]; Rosario Squatrito[1]; Alessandro Morri[1]; Luca Tomesani[1]; [1]University of Bologna

The aim of this work is to asses a correlation law between local cooling rate and SDAS for the EN AC43000 cast alloy. Bars 200 mm long with a double T cross section (suitable for tensile testing) were sand cast under controlled processing conditions. Chillers of different dimensions and materials, including water cooled ones, were placed at one side of the casting in order to obtain mono-dimensional heat flux and different cooling rates throughout the casting length. Thermocouples were used to acquire local cooling curves. A campaign of FE simulations was run to fine tune boundary conditions and to obtain a reliable cooling rate distribution throughout the whole casting. A correlation between experimental SDAS and simulated cooling rates was assessed. Finally, mechanical properties from extracted specimens in T6 condition were related to local SDAS and cooling rate.

10:10 AM

Evaluation of Transient Heat Transfer Coefficient Evolution in EN43000 Gravity Castings towards Steel Chills with Different Interface Conditions: Rosario Squatrito[1]; Ivan Todaro[1]; Luca Tomesani[1]; [1]University of Bologna

Heat exchange phenomena in foundry processes are governed by the heat resistance through contact interfaces between metal and casting tools. As known, physical and geometrical aspects play a fundamental role on the heat transfer conditions during the solidification process. This high dependence of heat transfer coefficient (HTC) by process variables determines the experimental method as the only way for a correct evaluation of heat fluxes during the cooling phase. The evolution of HTC between EN43000 aluminum alloy and H11 steel chills was investigated with different shapes of interface by means of an experimental gravity casting device and by developing an inverse method based on numerical analysis. The behavior of HTC as a function of time and casting temperature was evaluated with different treatment conditions of the chills surfaces (presence of coatings, knurling or smooth surface) to obtain correlations between HTC efficiency and manufacturing conditions.

10:35 AM

In Situ Synchrotron Quantification of Fe-Rich Intermetallic Formation in Al-Si-Cu-Fe Alloys: *Chedtha Puncreobutr*[1]; Junsheng Wang[1]; Peter Lee[1]; [1]Imperial College London

Iron uptake occurring during aluminum recycling can alter an alloy's castability while limiting the final component's fatigue life because of large Fe-rich intermetallic formation. To better understand these intermetallics, *in situ* synchrotron x-ray radiography was performed on an Al–7.5Si–3.5Cu–Fe(wt.%) alloy for two Fe levels and for different cooling rates. The intermetallic growth was quantified using image analysis. Nucleation temperatures were estimated by extrapolating growth back to zero size. The results illustrate that nucleation of the β-intermetallics is a function of both Fe level and cooling rate. The β-intermetallics nucleate between α-Al dendrites and grow rapidly until they impinge on surrounding α-Al dendrites. As the cooling rate increased, finer and closer packed α-Al grains form, limiting intermetallic plate growth. The results are compared to prior analytical and numerical models and discussed in terms of their influence on both alloy castability and component lifing.

Characterization of Minerals, Metals and Materials: Characterization of Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee
Program Organizers: Ann Hagni, Geoscience Consultant; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF; Jiann-Yang Hwang, Michigan Technological University

Tuesday AM Room: 307
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Sergio Monteiro, State University of the Northern Rio de Janeiro – UENF; Donato Firrao, Politecnico di Torino

8:30 AM

Detection of Creep Damage in a Nickel Base Super-Alloy by Nondestructive Means: *Hector Carreon*[1]; [1]UMSNH

By using eddy current images, it was shown that the eddy current technique could be used to monitor creep damage in a nickel base super-alloy. The eddy current results also show a significative variation in the electrical conductivity on the creep damage zone with respect to the background zone in nickel-base super-alloy samples. The absolute thermoelectric power coefficient show a strong dependence associated with creep damage in nickel base super-alloy samples using a copper hot tip reference. The eddy current and thermoelectric power techniques seems to show promise as nondestructive evaluation tools for monitoring the level of creep damage in metallic alloys.

8:50 AM

Microstructural, Mechanical and Fatigue Properties of Cobalt Alloys: Giorgio Scavino[1]; Paolo Matteis[1]; Giovanni Mortarino[1]; *Donato Firrao*[1]; [1]Politecnico di Torino

Cobalt alloy samples (wt.% composition: Cr 25, W 5, C 1.2, "stellite 6" type) were produced either by casting or by sintering (hot isostatic pressing) and subjected to microstructural examination and to fatigue tests performed at 250 or 500°C, with the staircase method, to determine the 2 million cycles fatigue limit. Cast samples exhibit coarse dendrites, with lamellar carbides in the interdendritic (eutectic) regions, whereas sintered samples exhibit spheroidal carbides dispersed in a fine-grained matrix; these microstructures are not detectably modified by the test temperatures. The cast material shows similar fatigue behaviors at 250 and 500°C. The sintered material (tested at 500°C only) exhibits comparatively better tensile and fatigue properties. The microscopic mechanisms for the fatigue crack nucleation and growth and for the overload fracture, as evidenced by fractographic investigations, are different in the two materials and are clearly dictated by their different microstructures and defect population.

9:10 AM

Reconstruction and Visualization of Multi-Phase Three-Dimensional Microstructure of a Cast Al-Si Base Alloy: *Arun Gokhale*[1]; Harpreet Singh[1]; Yuxiong Mao[1]; Asim Tewari[2]; Anil Sachdev[2]; [1]Georgia Institute of Technology; [2]General Motors Co.

This contribution reports reconstruction of the multi-phase multi-scale 3D microstructure of a permanent mold cast unmodified Al-12wt%Si-1wt%Ni base alloy containing eutectic Si platelets, coarse primary polyhedral Si particles, Fe-rich script intermetallic particles, and pores. These constituents are segmented, reconstructed, rendered, and characterized in three dimensions. The estimated 3D microstrucutral attributes include distribution of eutectic platelet thickness; mean volume, mean surface area, and mean thickness of the eutectic Si platelets; mean volume and mean surface area of the polyhedral primary Si particles; and mean number of faces, edges, and corners on the polyhedral primary Si particles.

9:30 AM

Phase Separation in Fe-20%Cr-6%Al-0.5%Ti ODS Alloy: *Carlos Capdevila-Montes*[1]; Michael Miller[2]; Felix Lopez[1]; Jesus Chao[1]; Kaye Russell[2]; [1]CENIM-CSIC; [2]ORNL

The temporal evolution of the microstructure resulting from phase separation of an Fe-20%Cr-6%Al-0.5%Ti ODS alloy at temperatures between 708 and

748 K has been analyzed by atom probe tomography (APT), thermoelectric power measurements (TEP), and differential scanning calorimetry (DSC). The roles of Al and Ti during the decomposition process have been investigated. Proximity histogram analysis revealed significant partitioning of Al and Ti, which is consistent with theoretical calculations. TEP measurements describe the macroscopic evolution of the process, which can be correlated with the kinetic equations obtained from atomic scale measurements obtained by APT. Finally, the activation energy of the process of phase separation is obtained by DSC. These results indicate that simultaneous phase separation into Fe-rich, Cr-rich and Fe-Ti-Al phases occurs.Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

9:50 AM

Development of a High-Temperature Micro-Indentation Technique for Material Mechanical Property Evaluation up to 1200°C: Jared Tannenbaum[1]; Brody Conklin[1]; *Bruce Kang*[1]; Mary Anne Alvin[2]; [1]West Virginia University; [2]National Energy Technology Lab

A depth-sensing micro-indentation technique is developed for evaluating material mechanical properties at elevated temperatures (to 1200°C). The depth-sensing micro-indentation system is capable of determining Young's modulus and creep strength of test materials with flat, tubular or curved architectures from room to 1200°C under either air or controlled gaseous environments. Calibration tests were conducted on a 1-in x 1-in 0.25-in H13 Tool Steel specimen from room to 600°C. Test resutls were in excellent agreement with literature values. Mechanical property evaluations of single crystal René N5 and Haynes 230 alloys were performed from room to 1100°C in air, inert gas, or in air with controlled moisture content. Post test microstructural analyses were conducted to correlate the effect of oxidation and moisture on the measured mechanical properties. Micro-indentation tests were conducted on MA 956 and PM 2000 ODS alloys at 1200°C. Preliminary test results will be presented and discussed.

10:10 AM

Characterization and Properties of a Stoichiometric NiTiPt High Temperature Shape Memory Alloy: *Fan Yang*[1]; Libor Kovarik[1]; Anita Garg[2]; Michael Kaufman[3]; Santo Padula[2]; Ronald Noebe[2]; Michael Mills[1]; [1]The Ohio State University; [2]NASA Glenn Research Center; [3]Colorado School of Mines

There has been a growing demand to achieve higher transformation temperatures than the commercial NiTi alloys can offer. Pt additions (greater than 10 at%) to the NiTi binary system is more potent in raising the Ms temperature than any other ternary element tested so far. For the stoichiometric alloy Ti50Ni29Pt21, the best shape memory response is obtained after aging at 500°C, during which a fine precipitate phase forms. Their unique structure and coherency have been characterized by conventional and high resolution scanning transmission electron microscopy. For the same alloy, aging at higher temperature introduces much coarser lath-like precipitates. The role of these phases in improving the work output and minimizing the permanent strain will be discussed.

10:30 AM

Estimation of Three-Dimensional Mean Dihedral Angle in a W-Ni-Fe Alloy Liquid-Phase Sintered in Microgravity: Maneel Bharadwaj[1]; Arun Gokhale[1]; *William Goodwin*[2]; [1]Georgia Institute of Technology; [2]University of Tennessee

The three-dimensional mean dihedral angle of impinging tungsten grains is estimated in a 50%W-35%Ni-15%Fe alloy liquid- phase sintered (LPS) in microgravity at 1500oC for times ranging from 1 to 600 minutes using unbiased stereology and automated image analysis. The estimated mean dihedral angles are in the range of 50 ± 3 degrees. The mean dihedral angle does not vary significantly with the liquid-phase sintering time although the mean intercept size of the tungsten grains increases almost by a factor of 5.

10:50 AM

Microscopic Analysis of Ni-Cr Alloy Produced by Single Roll Strip Casting: *Sanjeev Das*[1]; J. B. Seol[1]; Y.C. Kim[2]; C. G. Park[1]; [1]POSTECH; [2]Research Institute of Industrial Science and Technology

In the present investigation the microstructure and some mechanical properties of Ni-Cr alloy prepared by single roll strip casting (SRSC) were studied. The top surface (surface not in contact with the roll) of the as received sample was rough and lusterless. The grain size of the top surface was significantly larger compared to that of the bottom surface. Grain interior showed dendritic morphology. Etch pits, formed by dislocation were observed on the top and bottom surfaces of the sample. Scanning electron microstructure revealed continuous corroded region along the grain boundaries. X-ray diffraction (XRD) study confirms the formation of chromium carbide at the grain boundary, which depletes Cr near the grain boundary. SEM EBSD of the alloy in as-cast, homogenized, cold rolled, and annealed conditions were evaluated to observe the orientation of the grains. Twins were observed in cold rolled annealed sample.

11:10 AM

Influence of TCP Phase on Enduring Property of Single Crystal Nickle-Based Superalloys: *Tian Sugui*[1]; Qian Benjiang[1]; Li Tang[1]; Wang Minggang[1]; Xie Jun[1]; [1]Shenyang University of Technology

By means of the measurement of the enduring property and microstructure observation, an investigation has been made into the influences of the element Re and TCP phase on the enduring property of the single crystal nickel base superalloys. Results show that the strip-like TCP phase is precipitated along the <110> orientation on {111} planes in the containing/free Re superalloys during the aging, and the TCP phase is identified as μ phase, and the precipitated μ phase in 4.5%Re alloy is gradually spheroidized during aging. Thereinto,the strip-like μ phase may obviously decrease the enduring lifespen of the superalloys due to the consumption of the refractory elements and the effect of the stress concentration. But the stress concentration is not easily generated in the regions near the spheroidized μ phase, this is main reason of reducing to a small extent the creep lifespen of the 4.5%Re alloy.

11:30 AM

Structure and Properties of Melt-Spun Ni-Ti Shape Memory Alloy: *Walman Castro*[1]; Carlos Araújo[1]; George Anselmo[1]; [1]Universidade Federal de Campina Grande

Many physical properties of melt-spun ribbons as well as their microstructures sensitively depend on the values of the processing parameters such as the wheel speed, gas pressure, melt temperature and nozzle–wheel gap, etc. In the present study, the shape memory behavior of Ti–49 at.%Ni alloy ribbons fabricated at different cooling rates by the melt spinning and the influence of rapidly solidified on shape memory behavior was studied. When the ribbon is produced at a higher wheel velocity in melt spinning, the degree of undercooling becomes high because of its thinner thickness. Therefore, the amount of crystalline layer decreases with wheel velocity. The B2–B19 transformation occurs in ribbons fabricated at the wheel velocity of 30 m/s, while only B19 martensite transformation occurs in ribbons fabricated at the wheel velocity of 50 m/s.

11:50 AM

The Modeling and Processes Research of Titan Aluminides Structurization Received by SHS Technology: *Sereda Borys*[1]; Aleksandr Zherebtsov[1]; Yuriy Belokon'[1]; [1]ZSEA

As it is known the creation and development of new titanic alloys has been problematic up to now. They can be used in aircraft and vehicle construction, shipbuilding and as materials for keeping static loadings in high-temperature environments. In this work the synthesis of various titan aluminides is investigated. Mathematical model of structurization process was done. On the base of retrieved model the mechanisms of structurization in systems Ti + Al and Ti + 3Al was determined. The method of retrieving compacted products combusted in SHS conditions (SHS-compacting) was used to gain these intermetallic compounds.It is established that the SHS-compactizing technology is fully capable of supervising the process of structurizations with the whole complex of advantages and can be applied for receiving qualitative titan aluminides with the set of chemical-physical properties.

12:10 PM

Influence of Preparing Technologies on Microstructure and Creep Behavior of GH4169 Alloy: *Tian Sugui*[1]; Li Zhenrong[1]; Zhao Zhonggang[1]; Chen Liqing[2]; Liu Xianghua[2]; [1]Shenyang University of Technology; [2]Northeast University

Influence of the preparing technology on the microstructure and creep behaviour of the alloy is investigated by means of the creep curves measurement of the forged and hot continuous rolled (HCR) GH4169 alloy. Results show that the deformed features of the alloy during HCR are the twinning and dislocation activated within twinning, and HCR alloy displays a better creep resistance. During creep, the twinning occurs only the forged alloy, but the deformed features of HCR alloy is the twinning and dislocation activated within the twins. Thereinto, "pre-dislocation" in HCR alloy promote the multi-systems slipping of dislocation activated within the twins, and releasing the stress concentration,

which is thought to be a main reason of improving creep resistance of the alloy. In the later period of creep, the fact that the micro-cracks are formed and expanded along the boundaries is the fracture mechanism of the alloy during creep.

Coatings for Structural, Biological, and Electronic Applications: Metallic Coatings

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS: Thin Films and Interfaces Committee

Program Organizers: Nuggehalli Ravindra, New Jersey Institute of Technology; Gregory Krumdick, Argonne National Laboratory; Roger Narayan, Univ of North Carolina & North Carolina State Univ; Choong-un Kim, University of Texas at Arlington; Nancy Michael, University of Texas at Arlington

Tuesday AM Room: 309
February 16, 2010 Location: Washington State Convention Center

Session Chair: Roger Narayan, University of North Carolina

8:30 AM Introductory Comments

8:40 AM

Evaluation of Thermal and Intrinsic Stress in Copper and Tantalum Sputtered Films: *Anahita Navid*[1]; Eric Chason[2]; Andrea Hodge[1]; [1]University of Southern California; [2]Brown University

The thermal stress contribution to the total film's residual stress is presented for copper and tantalum films deposited by magnetron sputtering on Si substrates using a uniform power at various pressures. The in-situ stress was measured using a Multibeam Optical Stress Sensor (MOSS) system while the ex-situ stress was measured by stylus profilometry. Results show that the thermal stress contribution is quite significant and can be as large as the intrinsic stress and might change the stress state on the film after deposition. The deposition temperature was also examined as a function of sputtering pressure, in order to relate to the thermal stress. It was determined that as the pressure increases, the deposition temperature decreases; this was related to the calculated total energy of the sputtered atoms and gas neutrals which also decreased with increasing pressure.

9:05 AM

Surface Modification of Steel Substrate by Pulsed Laser Deposition Technique: *Shampa Aich*[1]; Saket Ahuja[1]; Lakpathi Banoth[1]; Indranil Manna[1]; [1]Indian Institute of Technology

A series of bi-layer and multi-layer coatings of Ti/TiB_2, Ti/TiN, Cr/CrN and Ti/TiN/Cr/CrN were successfully deposited over austenitic stainless steel substrate using pulsed laser deposition technique. The X-ray diffraction patterns reveal the coatings to be crystalline and compositional micro-analysis suggests that titanium atoms diffuse into the substrate to form strong bond between film and substrate. The Ti/TiB_2 coatings were developed in varying thicknesses from 55 nm to 550 nm, with the maximum thickness at 500ºC. For the nitride bi-layer and multi-layer coatings, the optimum coatings (~ 600 nm) were produced at 300ºC to obtain excellent surface properties. The multi-layered metal/nitride coatings improved resistance to scratching and helped in arresting the crack propagation. In terms of mechanical properties, double bi-layer (Ti/TiN/Cr/CrN) performs the best followed by Ti/TiN/Ti and then Cr/CrN/Cr. While the bi-layer of Ti/TiB_2 shows the best followed by single TiB_2 and then single Ti layers.

9:30 AM

A Nucleation and Growth Model for Pulse Plated Trivalent Chromium Deposition: *Yong Choi*[1]; Sik C. Kwon[2]; [1]Sunmoon University; [2]KIMS

eco-friendly trivalent chromium layers were prepared in modified chromium chloride and sulfate baths by pulse plating with ultrasonic agitation to replace hexavalent hard chromium coating in industrial fields. Deposition rates of the chromium layers by ultrasonic pulse plating was in the range of 300-350 nm/min. Micro-hardness of the ultrasonic pulse plating is lower than those of pulse plating and direct current plating. Wear resistance of the trivalent chromium layer prepared by ultrasonic pulse plating is higher than that of the chromium layer prepared by a direct current plating. The ultrasonic agitation during pulse plating resulted in increasing neutral salt fog spray life and wear resistance that is related to smaller crack size and more broad size distribution of the trivalent chromium. A model of nucleation and growth was proposed to describe deposition rate and microstructure of the pulse plating.

9:55 AM Break

10:10 AM

An Investigation on Phase Formations and Microstructures of Ni-Rich NiTi Shape Memory Alloy Thin Films: *B. Geetha Priyadarshini*[1]; Shampa Aich[1]; Madhusudan Chakraborty[1]; [1]Indian Institute of Technology

Ni-rich NiTi alloy thin film of 40 at. % Ti was fabricated by RF/DC magnetron sputtering using elemental Ni and Ti as sputter targets. Si (100) was chosen as substrate which was either held at room temperature or at 300 ºC during the deposition. The 380 nm thick films were characterized by Field Emission Scanning Electron Microscopy, Energy Dispersive Spectroscopy, Grazing Angle X-ray Diffraction, Atomic Force Microscopy and High-Resolution Transmission Electron Microscopy. The results showed that due to lack of surface mobility of the adatoms the room temperature deposited films were smooth, amorphous, with a grain size of 20-25 nm accompanied with porous microstructure. At higher substrate temperatures, increase in the surface diffusion leads to formation of partially crystalline, rougher films with denser, compact, fibrous grains without void boundaries. Formation of Ni-rich precipitates such as: Ni_4Ti_3, Ni_2Ti and Ni_3Ti along with small amount of the NiTi phase were also confirmed.

10:35 AM

Effect of Heat Treatment on the Microstructure and Mechanical Properties of Ti-Mo-N Coating Films: *Shoko Komiyama*[1]; Yuji Sutou[1]; Junichi Koike[1]; [1]Tohoku University

TiN coatings are widely used in cutting tools and medical implants. Recently, it has been reported that the addition of Mo is effective to enhance the wear resistance of TiN coatings [1]. In this study, the effects of heat treatment on the mechanical properties of (TiMo)1-xNx films were investigated. The (TiMo)1-xNx films were deposited on AISI304 steel by reactive RF sputtering using a Ti50Mo50 target. To deposit the nitrides, the mixture of Ar (7.5ccm) and N2 (0-2.0ccm) gases was introduced. The obtained films were heat treated at various temperatures in Ar atmosphere. It was found that the microhardness of the (TiMo)1-xNx films showed a maximum value of about 30GPa at N2=0.3ccm, which was increased further to 35 GPa after heat treatment at 1000° The maximum hardness was due to the formation of a fine grain structure. [1] Q. Yang et al. Wear 261 (2006) 119.

11:00 AM

PVD Coated Hot Work Tool Steels for Tooling Applications in Semi-Solid Processing of Steels: *Duygu Isler*[1]; Yucel Birol[2]; Mustafa Urgen[1]; [1]Istanbul Technical University; [2]TUBITAK

Semi-solid processing of metals combines the advantages of forging and casting while shaping of metallic components. Having already matured into an industrial practice for Al and Mg alloys, this technology could upgrade the market for forged steel parts. However, the surface-to-interior temperature differentials in steel thixoforming dies are much larger than with Al and Mg. Combined with the chemical, tribological and thermal interactions between die and formed material confer very specific requirements on tool materials. Hot work tool steel dies are inadequate due to the limited temper resistance of the commercially available grades. Physical Vapour Deposition (PVD) coatings, shown to have beneficial impact on the lifetime of pressure die casting tools, could offer a solution. X32CrMoV3-3 hot work tool steel was coated with AlTiN and AlTiON utilizing cathodic arc PVD technique in the present work. The performance of the coated samples was tested under thermal fatigue and erosive wear conditions.

Computational Thermodynamics and Kinetics: Wetting Phenomena I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Jeffrey Hoyt, McMaster University; Dallas Trinkle, Univeristy of Illinois at Urbana-Champaign

Tuesday AM Room: 308
February 16, 2010 Location: Washington State Convention Center

Session Chair: To Be Announced

8:30 AM Invited
Grain Boundary Premelting: Insights from Order Parameter Models: *Alain Karma*[1]; Ari Adland[1]; Robert Spatschek[2]; [1]Northeastern University; [2]Ruhr-University

The presence of liquid films at grain boundaries below the bulk melting point can alter macroscopic properties of polycrystalline solids and dramatically reduce their resistance to shear stresses. This talk will discuss recent progress made in understanding grain boundary premelting using both atomic-scale and coarse-grained order parameter models. New fundamental insights that will be reported stem from detailed numerical surveys of those models over a wide range of parameters, analyses in tractable limits, and critical quantitative comparisons with molecular dynamics simulations in bcc and fcc elemental systems.

9:00 AM Invited
Molecular Dynamics Simulations of Brazing: *Edmund Webb*[1]; Jeff Hoyt[2]; [1]Sandia National Laboratories; [2]McMaster University

Brazing is a relatively high temperature joining process important to a range of metallurgical technology. Brazing depends upon good wettability between constituent materials; furthermore, reactions between solid and liquid are often observed. Understanding fundamental phenomena in high temperature reactive wetting bears potential for impact on technology but also on theoretical descriptions of capillarity. Molecular dynamics (MD) simulations are a useful counterpart to experiment in describing fundamental wetting mechanisms. This talk will review MD simulations of high temperature wetting for metals in a braze geometry. Infiltration of molten metal into a solid pore is simulated for a system where the solid simultaneously dissolves into the liquid. Simulations reveal a regime where the dissolution reaction is aggressive enough to alter the kinetics of infiltration. MD simulations are coupled with Monte Carlo calculations to draw quantitative connections between the free energy of the dissolution reaction and the kinetics of pore infiltration.

9:30 AM
A Hybrid Phase-Field and ALE Model for Reactive Wetting in Metal/Metal Systems: *Shun Su*[1]; Bruce Murray[2]; Ying Sun[1]; [1]Drexel University; [2]Binghamton University

Reactive wetting refers to a liquid drop spreading on a substrate with reaction or dissolution. In order to model the dissolutive wetting process, fluid flow, species transport and phase change must be coupled. In this work, the phase-field method along with the Arbitrary Lagrangian-Eulerian (ALE) technique is used to simulate the evolution of an alloy drop on a metal substrate while the process is assumed to be isothermal. The liquid-vapor interface of the drop is tracked by a phase-field variable while the evolution of the solid-liquid interface is determined by the ALE method. The shape of the dissolution boundary and the extent of spreading are investigated as a function of initial solute concentration. The triple junction kinetics is studied in relation to the model assumptions. The simulations are performed on a millimeter scale and the results are compared with experimental data for a Sn-Bi system.

9:50 AM Break

10:00 AM Invited
Modeling Grain Boundaries: Wetting, Vacancies and Creep: *James Warren*[1]; William Boettinger[1]; [1]NIST

The challenges in developing a robust macroscopic model of grain boundaries that describes the majority of processes of interest to the materials processor are substantial. In this work I will describe the ongoing efforts to synthesize several different efforts in grain boundary modeling with classical models developed by Larche and Cahn into a coherent picture. It is hoped that such models will provide substantial insights into the mechanical properties of materials, and,in particular, insights into vacancy diffusion mediated creep (either in the bulk or grain boundaries) of materials with residual stress.The back-coupling of these phenomena to grain boundary wetting should also prove interesting.

10:30 AM Invited
Atomistic Behavior Driving High Temperature Contact Line Advancement: *Ying Sun*[1]; Edmund Webb[2]; [1]Drexel University; [2]Sandia National Laboratories

Atomic scale phenomena driving contact line advancement during wetting of a solid by a liquid are investigated via molecular dynamics simulations of Ag(l) drops spreading on Ni substrates. For homologous temperature ~5% above melting for Ag, essentially non-reactive wetting is observed with relatively high spreading velocity. Delivery of material to the contact line occurs preferentially along the L/V interface. New material transported into the drop edge near the L/V interface displaces existing edge material. Atoms forming the droplet edge at a given instant are preferentially constrained near the S/L interface as the droplet advances across the substrate. While contact line advancement appears dominated by rapid flow along the L/V interface, evidence emerges that molecular kinetic mechanisms play a role as some atoms move with the contact line, desorbing and re-adsorbing between neighboring sites at the S/L interface. Results illustrate specific mechanisms underlying wetting and spreading behavior at high temperature.

11:00 AM
Diffusivity in Al-Cu and Cu-Zr Liquids: *Shihuai Zhou*[1]; Ralph E. Napolitano[2]; [1]Ames Laboratory; [2]Department of Materials Science and Engineering, Iowa State University

A high-temperature thin tube liquid diffusion couple method is employed to determine the composition dependent diffusivity in binary Al-Cu and Cu-Zr alloys. Composition profiles are measured in quenched thin-tube specimens and analyzed in conjunction with assessed solution thermodynamic models. Experimental methods and results are presented, and composition-dependent atomic mobility and thermodynamic factors are discussed. This research is supported by the U.S. Dept. of Energy Office of Science through Ames Laboratory contract No.DE-AC02-07CH11358.

11:20 AM
A Study of the Te Melting Line and Solid-Liquid Transitions: *Chuck Henager*[1]; Fei Gao[1]; John Jaffe[1]; [1]PNNL

Te-particles that form during the growth of CdZnTe (CZT) single crystals are a macroscopic defect with apparent origin in the CZT melt. As the freezing point for CdTe is 1365 K and that of Te is 722 K it is important to understand the mechanisms by which presumably l-Te(Cd)-particles form within the CZT solid. A new interatomic potential (IAP) for Te has been developed based on a modified Stillinger-Weber form as was done previously for Se by others in order to study large systems more suited to crystal growth modeling. The IAP is used to study Te melting and the Te melting line via molecular dynamics. Results for density changes, enthalpy of melting, and Te liquid structures will be presented and compared to ab initio and literature results. The use of the new IAP for Te in developing an improved IAP for CdTe is discussed.

11:40 AM
Compositional Patterning and Morphological Evolutions in Binary and Ternary Alloys Driven by Irradiation: *Pascal Bellon*[1]; Anoop Damodaran[1]; Daniel Schwen[1]; Robert Averback[1]; [1]University of Illinois

The dynamical competition between irradiation-induced atomic mixing and thermally activated decomposition can induce the self-organization of the composition field into patterns. Analytical modeling and atomistic simulations works showed that a key requirement for this self-organization is that the forced mixing takes place at a finite range exceeding the characteristic range of diffusional jumps. We extend past kinetic Monte-Carlo approaches to

multi-component alloy systems, and we investigate the effects of composition-dependent atomic mobility. In the case of slow solute mobility, it is shown that precipitates stabilized by irradiation may develop a "cherry-pit" structure. In the case of ternary and quaternary alloys, we show that, compared to binary alloys, several new nanostructures can be stabilized, in particular core-shell precipitates. These results will be contrasted with available experimental results.

12:00 PM

Irradiation Induced Re-Solution and Growth of Xenon Nano-Bubbles Simulated by First Passage Monte Carlo: *Daniel Schwen*[1]; Robert Averback[1]; [1]University of Illinois

With the development of generation IV reactor technology, the impact of intragranular fission gas on the performance of UO_2 fuels has gained considerable concern. Gaseous fission products and their precipitation as nano-scale fission gas bubbles are known to adversely influence the thermal and mechanical properties of fuels.To study the gas diffusion and bubble population evolution we developed a first passage based fast Monte Carlo software. First passage Monte Carlo dramatically accelerates simulation times for dilute systems of diffusing particles, such as fission gas in fuel matrices, by analytically describing the temporal behavior of random walkers and thus removing the need to compute single atomic jumps. In contrast to mean field theories spatial correlations arising from re-solution distances and proximity to grain boundaries (sinks) can be readily incorporated. A variety of gas re-solution scenarios and diffusion models are explored and compared to existing data.

Cost-Affordable Titanium III: Powder Consolidation and Properties I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
Program Organizers: M. Ashraf Imam, Naval Research Lab; F. H. (Sam) Froes, University of Idaho; Kevin Dring, Norsk Titanium

Tuesday AM Room: 618
February 16, 2010 Location: Washington State Convention Center

Session Chairs: James Withers, Materials and Electrochemical Research Corporation; Derek Fray, University of Cambridge

8:30 AM

Equations for the Compaction of Titanium Powders: *Stephen Gerdemann*[1]; Paul Jablonski[1]; [1]NETL

Accurate modeling of powder densification has been an area of active research for more than 60 years. The earliest efforts were focused on linearization of the data since computers were not readily available which made curve fitting difficult. In this work several lots of titanium powders ranging in size, shape and chemistry were cold pressed in a single acting die instrumented to collect stress and deformation data during compaction. From this data the density of each button was calculated and then plotted as a function of pressure. The results show that densification of all the powders, regardless of particle size, shape or chemistry, can be very accurately modeled as the sum of an initial density plus the sum of a particle shape term and a powder densification term. These last two terms are found to be a function of applied pressure and take the form an exponential rise.

8:55 AM

Making Titanium Powder Metallurgy a Viable Alternative to Wrought for Manufacturing: *James Sears*[1]; [1]South Dakota School of Mines and Technology

An overview of the current and developing production sources for Titanium powder are given along with a review of the potential applications and new opportunities being pursued. It has long been known that titanium would enter many new markets if production and manufacturing costs could be reduced. It is also know that making components directly from Titanium Alloy powder circumvents many of the production costs associated with wrought Titanium manufacturing. The hurdle that prevents Titanium Powder Metallurgy (PM) for becoming a viable alternative to wrought manufacturing is the high costs associated with Titanium powder production. An evaluation of the processes that currently produce Titanium powder is presented along with the methods that are being employed to reduce the associated costs of Titanium powder production. An overview of the National Center of Excellence for Titanium Powder Metallurgy will be given.

9:20 AM

Development of an Affordable Supply Chain for Meltless Titanium Alloys: *Eric Ott*[1]; Andy Woodfield[1]; Jon Blank[1]; Michael Peretti[1]; David Linger[1]; [1]GE Aviation

The advent of new technologies for conversion of Ti-bearing raw materials to metal powder forms, and particularly the capability of some processes to produce titanium alloys directly provides a path to make titanium alloy products without incurring the expense, energy, and time associated with melting processes. This "meltless" titanium technology can substantially decrease the number of major processing steps and provide large improvements in product yield, energy utilization and emissions. Near-net-shape processing provides further material yield improvements for complex, machining intensive geometries. A disruptive, efficient and stable supply chain that takes advantage of these improvements is needed and new strategies must be developed to ensure sufficient production capacity as well as a competitive environment. This paper will establish technical, cost and capacity benefits of the meltless titanium technology, will propose criteria for development of a robust supply chain, and will discuss key requirements for aerospace and non-aerospace commercial implementation.

9:45 AM

Consolidation Process in Near Net Shape Manufacturing of Armstrong CP-Ti/Ti-6Al-4V Powders: *Yukinori Yamamoto*[1]; Jim Kiggans[1]; Michael Clark[1]; Stephan Nunn[1]; Adrian Sabau[1]; William Peter[1]; [1]Oak Ridge National Laboratory

This presentation summarizes our recent efforts to develop the manufacturing technologies of consolidated net-shape components by using new low-cost commercially pure titanium (CP-Ti) and Ti-6Al-4V alloy powders made by Armstrong process. Net-shape components have been fabricated by pressing and sintering, cold isostatic pressing (CIP), hot isostatic pressing (HIP), pneumatic isostatic forging (PIF), and/or adiabatic compaction. The press-and-sinter processing of the powders in the as-reduced condition were evaluated systematically in terms of theoretical density and microstructure as functions of time, pressure, and temperature. Up to 96.4% theoretical density has been achieved with the press-and-sinter technology. A consolidation modeling is also under development to interpret the powder deformation during processing. The detailed experimental results in conjunction with future plan will be discussed. This research was sponsored by the U.S. DOE, ORNL, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC. Additional funding and collaboration with the SHaRE User Facility at ORNL is also acknowledged.

10:10 AM Break

10:25 AM

Technical Challenges and Solutions for Cost-Efficient Manufacturing of Complex Shape Parts from Ti Alloys via PM HIP: *Victor Samarov*[1]; [1]Synertech PM Inc.

Innovative developments in HIP technology provide novel answers to the old challenges of processing complex shape parts from Ti alloys: -very low material yield during processing of complex shape parts; -dominating costs of machining to the final geometry; -high cost on initial materials, including alloyed powders; -increasing technical difficulties and cost with larger parts; -eternal fight for the appropriate micro-structure and properties in large forgings These new developments are based on the selectively net shape computer controlled HIP processing of alloyed powders atomized with high solidification rates and enable -to cut substantially the material losses and increase the "buy-to-fly" ratio several times; -to improve machinability of Ti parts; -to provide uniformity and homogeneity of complex shape parts -to build the material properties above the level of wrought. Various examples of developed parts and processes illustrate these novel solutions.

10:50 AM

Cost-Effective Production and Thermomechanical Consolidation of Titanium Alloy Powders: *Deliang Zhang*[1]; Stiliana Raynova[1]; Vijay Nadakuduru[1]; Peng Cao[1]; Brian Gabbitas[1]; Barry Robinson[2]; [1]The University of Waikato; [2]South Auckland Forging Engineering Ltd

This talk has two parts: (i) an introduction of the TiPro process which has been recently developed at the University of Waikato for cost-effectively producing titanium alloy powders from low cost TiO2 and Al powders and

other reactants such as calcium granules; and (ii) an overview of major recent findings from our research on thermomechanical consolidation of titanium and Ti-6wt%Al-4wt%V alloy powders. The TiPro process includes several steps: solid-liquid separation, crushing, powder purification and by-product treatment. The thermomechanical consolidation of titanium and titanium alloy powders involves open-die forging of powder compacts, extrusion of powder compacts, and rolling of forged disks and extruded billets. In the talk, the materials science principles underlying the powder production steps and the different thermomechanical powder consolidation processes will also be discussed based on the experimental results from microstructural characterisation of samples produced under different conditions.

11:15 AM

The Impact of Diffusion on Synthesis of High-Strength Titanium Alloys from Elemental Powder Blends: *Orest Ivasishin*[1]; Vadym Bondarchuk[1]; Dmytro Savvakin[1]; [1]G.V. Kurdyumov Institute for Metal Physics, NAS of Ukraine

High strength alpha plus beta and near-beta titanium alloys are being increasingly used in industry due to their excellent combination of properties. Blended elemental powder metallurgy (BEPM) allows producing the above alloys and parts from them in a cost-effective manner. However, the alloy synthesis is complicated by a big amount (up to 20 wt.%) of alloying elements which diffusional redistribution between alloying particles and titanium matrix has a strong impact on microstructure evolution. In this paper synthesis of the high-strength alloys from the powder blends based on either titanium or hydrogenated titanium was compared. It was found that hydrogen strongly affects diffusion controlled processes upon synthesis, such as chemical homogenization, densification and grain growth through its influence on phase composition and defect structure of the blends. Optimization of the processing parameters allowed to produce uniform, nearly-dense alloys with reduced grain size, which mechanical properties met the requirements of corresponding specifications.

11:40 AM

In-situ Compression and Sintering of CP-Ti Powder Made by Armstrong Process: *Wei Chen*[1]; Yukinori Yamamoto[2]; William Peter[2]; [1]Michigan State University; [2]Oak Ridge National Lab

This work used *in-situ* technique to investigate the pressing and sintering processes of the commercially pure (CP) Ti powder made by Armstrong process. Ti powders were uniaxially pre-pressed at designated pressures up to 100 ksi to form disk samples with different theoretical densities. Compression tests were performed in an SEM at different temperatures to obtain the mechanical properties and deformation behavior of these samples. *In-situ* sintering was also performed in an SEM to record the morphology change of the porosities on the sample surface during the sintering process. The results will provide valuable information for optimizing the manufacturing process of high-density near net shape Ti components. This research was sponsored by the U.S. Department of Energy, Oak Ridge National Laboratory, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC. Additional funding and collaboration with the SHaRE User Facility at ORNL is also acknowledged.

Electrode Technology for Aluminum Production: Non-Carbon Materials in Cathodes

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Ketil Rye, Alcoa Mosjøen; Morten Sorlie, Alcoa Norway; Barry Sadler, Net Carbon Consulting Pty Ltd

Tuesday AM Room: 616
February 16, 2010 Location: Washington State Convention Center

Session Chair: Arne Ratvik, NTNU

8:30 AM Introductory Comments

8:35 AM

Lower Aluminium Production Cost through Refractory Material Selection: *Ole-Jacob Siljan*[1]; Steinar Slagnes[2]; Anton Sekkingstad[2]; Egil Furu[1]; Sigurd Aaram[1]; Asbjørn Solheim[3]; [1]Hydro Aluminium AS; [2]North Cape Minerals; [3]SINTEF

Hydro Aluminium and North Cape Minerals have jointly developed olivine-based refractories for use in the cathode lining in aluminum electrolysis cells. This type of material has turned out to be thermally stable and cost efficient, and more than 400 cells have been started with olivine-based linings since the first installation at the SU3 prebake potline at Sunndalsøra in 1999. Operational data from SU3 indicate that cells with olivine-based lining have higher current efficiency than similar cells with traditional lining. This seems to be due to a combination of factors, as analyzed in the present paper. The introduction of olivine was accompanied by conversion to a much larger brick format, resulting in shorter pot turn-around time and reduced relining costs. Based on the positive experience from the Norwegian plants, the olivine-based refractory material Alubrick 2092 will now be installed in 60% of the cells in the Qatalum smelter.

9:00 AM

Chemical Degradation Map for Sodium Attack in Refractory Linings: *Kati Tschöpe*[1]; Tor Grande[1]; Jørn Rutlin[2]; [1]NTNU; [2]Hydro Aluminium AS

Here we show by autopsies of spent pot linings that the degradation of refractory linings in aluminum reduction cells is governed by sodium transport into the lining. The chemical reactions caused by sodium infiltration are qualitatively explained by the construction of a chemical degradation map. The degradation map corresponds to a predominance phase diagram showing the stable compounds present as a function of SiO2/Al2O3 ratio in the refractory lining and the amount of sodium infiltrated in the lining. We demonstrate that the degradation map is a useful tool for the evaluation of autopsies of spent pot linings and the prediction of the mineralogical composition of the spent pot lining.

9:25 AM

Reactions in the Bottom Lining of Aluminium Reduction Cells: *Asbjorn Solheim*[1]; Christian Schøning[1]; Egil Skybakmoen[1]; [1]SINTEF

The bottom lining in aluminium cells deteriorates due to chemical reactions with sodium vapour as well as with molten bath that penetrates the cathode carbon. The supposedly most important reactions in the system were studied, both theoretically and by experiments. The phase diagram for the system SiO2-Al2O3-Na2O was supplied with numbers for the equilibrium pressure of sodium as well as vectors showing the change in oxide composition during attack. It could be predicted that chamotte ends up as mainly nepheline. By exposing samples of firebricks to a fluoride melt at 950°C ("cup test") as well as to sodium vapour at 800°C, it was found that materials high in silica were less deteriorated than materials rich in alumina. The reason is probably that the silica-rich materials formed larger amounts of a viscous glass phase. Some observations concerning the "lens" formed in industrial cells are also reported.

9:50 AM

Sidewall Materials for the Hall-Heroult Process: Reiza Mukhlis[1]; Muhammad Rhamdhani[1]; *Geoffrey Brooks*[1]; [1]Swinburne University of Technology

The performance of current sidewall materials (such as carbon and silicon carbide-based materials) in an aluminum smelter in the Hall-Heroult process rely on the existence of a frozen electrolyte layer on the sidewall, which contributes to the high energy requirement of the process. If the overall heat required to be

dissipated with the process were lowered through improvements in anode and cathode design, it maybe possible to decrease the heat flux through the sidewalls and move to a low heat loss operating regime. This study will consider new sidewall materials that can resist the corrosive, reductive and oxidative nature of molten cryolite, molten aluminum and air, respectively, that would need to be developed in order to avoid the necessity of the frozen layer formation. This paper will review previously proposed and current sidewall materials, and details materials selection, design and process criteria for identifying viable alternative solutions to the problem.

10:15 AM Break

10:30 AM

Excellent Cryolite Resistance and High Thermal Conductivity SiC Sidewall Material for High-Amperage Aluminium Reduction Cells: *Zhigang Huang*[1]; [1]LIRR

With the development of the aluminium reduction cell towards high current and large-scale, the performance of the sidewall materials are expected to be even better. To meet the requirements of this technical trend, new sidewall material which contains SiC up to 95% has been developed. After testing cryolite corrosion resistance which was conducted by simulating the erosion environment of the aluminium reduction cell, the new material presents much better cryolite corrosion resistance nearly twofold of the traditional Si3N4-SiC material. In addition, the thermal conductivity of the new developed sidewall material is about 30% higher than that of traditional Si3N4-SiC material. High thermal conductivity is favorable to quick formation of the frozen ledge and can therefore improve the service life of the cells. The other properties are equivalent to those of Si3N4-SiC material. Therefore, the new developed material has been considered as a promising sidewall material for large-scale Aluminium reduction cell.

10:55 AM

Structure Design and Deformation Measurements of C/TiB2 Function Gradient Materials for Aluminum Reduction Cathode: *Jilai Xue*[1]; Baisong Li[1]; Jun Zhu[1]; [1]University of Science and Technology Beijing

Carbon/TiB2 Function Gradient Materials (FGM) were tested for use as aluminum reduction cathode materials, where a 10mm thick TiB2-rich layer was on the top as an Al-wettable surface, the C-TiB2 layers in the middle as a transition interface and the carbon on the bottom as a base. ANSYS simulations suggest that the C/TiB2 FGM cathode with the layers number n = 3 and the gradient coefficient = 0.6 can offer lower thermal strain and sodium expansion. Cathode deformations (creep and sodium expansion), were measured in a modified Rapoport apparatus. During aluminum electrolysis, the 3-layer FGM samples showed lower sodium expansion than the 2-layer one, while the later exhibited smaller creep deformation than the former. Both the creep and sodium expansion varied with changing in operating temperature, melt composition and current density. The obtained data can be used for cathode design and cell construction using the C/TiB2 FGM cathode materials.

11:20 AM

Electrolysis Expansion Performance of TiB_2-C Composite Cathode in $[K_3AlF_6/Na_3AlF_6]$-AlF_3-Al_2O_3 Melts: Fang Zhao[1]; *Lu Xiao-jun*[1]; Li Jie[1]; Lai Yan-qing[1]; Tian Zhong-liang[1]; [1]School of Metallurgical Science and Engineering, Central South University

Electrolysis expansion of pitch, furan, phenolic aldehyde and epoxy based TiB_2-C composite cathodes in $[K_3AlF_6/Na_3AlF_6]$-AlF_3-Al_2O_3 melts were tested, and the morphology and element distribution of cut sections of specimens after electrolysis were studied. The results show that pitch based TiB_2-C composite cathode has the maximal electrolysis expansion, while epoxy based TiB_2-C composite cathode has the minimal electrolysis expansion. Moreover, each binder has the optimum addition, corresponding to its own minimum electrolysis expansion. The optimum addition of pitch, furan, phenolic aldehyde and epoxy are 16%, 18%, 14% and 12% respectively. At this circumstance, electrolysis expansion of TiB_2-C composite cathodes are 1.49%, 1.26%, 1.18% and 0.92% severally. SEM and EDS analysis suggest that no matter what kind of binder used, TiB_2-C composite cathodes have good wettability by molten aluminum. K and Na penetrate into cathodes from exterior to interior gradually, and K has stronger penetrating ability than Na.

Failure of Small-Scale Structures: Deformation Events in Pillars, Films and Other Structures

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Marian Kennedy, Clemson Univeristy; Brad Boyce, Sandia National Laboratory; Reinhold Dauskardt, Stanford; Zhiwei Shan, Hysitron Inc

Tuesday AM Room: 206
February 16, 2010 Location: Washington State Convention Center

Session Chair: Zhiwei Shan, Hysitron Inc.

8:30 AM Invited

Prestraining and Annealing of Gold Micropillars: Strengthening and Weakening Turned Upside Down: *William Nix*[1]; Seok-Woo Lee[1]; [1]Stanford University

When soft metals are plastically deformed, they get stronger, mainly because the dislocation density increases dramatically. Correspondingly, annealing of strain-hardened metals commonly leads to softening because the annealing causes the dislocation density to decrease. Recent experiments with gold micropillars have shown that metals behave very differently at the sub-micrometer scale. At that scale plastic deformation leads to softening and annealing leads to hardening, just the opposite of what occurs in bulk metals. These results suggest that plasticity at the sub-micrometer and nanometer scale is controlled not by the elastic interactions of dislocations, as in bulk metals, but by the operation of dislocation sources. The evidence for source-controlled plasticity is reviewed and a model to describe the corresponding size effects and the unusual effects of prestraining/annealing is described. The model is inspired by Johnston and Gilman's dislocation dynamics approach to plasticity and celebrates the 50th anniversary of that classic work.

8:55 AM Invited

In-situ Micromechanical Testing: *Johann Michler*[1]; [1]EMPA, Swiss Federal Laboratories for Materials Testing and Research

The presentation will first review in-situ SEM micromechanical testing techniques (micro-compression, nanoindenter, atomic force microscope, nano-tensile MEMS device). In the second part different case studies will be presented: 1)The compressive fracture strength of micron-sized Si and GaAs pillars increases with decreasing pillar diameter, but submicrometer pillars were found to deform plastically in uni-axial compression. TEM observations suggest that single partial dislocations are the carriers of plasticity in GaAs and that the critical size for ductile – brittle transition can be predicted from the fracture mechanics of pillar splitting. 2) The yield strength of rectangular micro-pillars of monocrystalline tungsten is shown to scale with the smallest dimension of the structure, whereas micro-pillars of amorphous metals exhibit the macroscopic yield strength down to diameters of 300nm. 3) The strength of both monocrystalline silicon and Rhenium nanowires is found to be close to the theoretical strength, in contrast to nanocrystalline nanowires.

9:20 AM

Investigation of the Deformation Mechanism of Gum Metal by In Situ TEM Nanocompression Testing: *Elizabeth Withey*[1]; Andrew Minor[1]; Shigeru Kuramoto[2]; Daryl Chrzan[1]; John Morris[1]; [1]University of California, Berkeley; [2]Toyota Central R&D Laboratories, Inc.

Gum Metal is a set of ß-Ti alloys, which appear to deform near ideal strength. Recent work has suggested that Gum Metal deforms by a stress-induced martensitic transformation to the orthorhombic a" phase. In order to better investigate the roll of this martensitic deformation in the overall deformation behavior of Gum Metal, nanopillars of solution-treated and cold-worked Gum Metal were compressed in situ in a quantitative compression stage in a transmission electron microscope. By performing these tests in diffraction mode it is possible to follow the evolution of the crystal structure as deformation occurs and correlate it to a quantitative load-displacement curve. Results from these tests showed that the martensitic phase transformation sometimes occurred, but did not appear to be a significant cause of deformation.

9:35 AM Invited
A Combined Experimental and Simulation Study to Examine Lateral Constraint Effects in Ni Superalloy Microcrystals: Paul Shade[1]; Robert Wheeler[2]; Yoon-Suk Choi[2]; *Michael Uchic*[3]; Dennis Dimiduk[3]; Hamish Fraser[4]; [1]Universal Technology Corporation; [2]UES, Inc.; [3]Air Force Research Laboratory; [4]The Ohio State University

We have used a custom in-situ SEM mechanical testing system to study the deformation behavior of single-slip oriented Rene N5 microcrystals. Two different types of platens were used to explore the effect of lateral stiffness on the resultant mechanical response in both tension and compression, which approximated either a rigid or extremely compliant lateral constraint. The change in the lateral constraint of the test system had a demonstrable effect on many aspects or attributes of plastic flow: the yield stress and strain-hardening behavior, the intermittency of strain bursts, the spatial distribution of slip bands, and the development of internal crystal rotations. Finite element modeling of the experiments using an anisotropic crystal plasticity framework provided insight regarding changes in the internal stress field and resultant activity of slip systems. The experimental findings are rationalized based on these simulation results.

10:00 AM Break

10:15 AM Invited
Interface Fracture and Fatigue of Thin Metallic Films on Substrates: *Gerhard Dehm*[1]; Megan Cordill[1]; Walther Heinz[2]; Kurt Matoy[3]; F. Dieter Fischer[4]; [1]University of Leoben, Materials Physics; [2]Austrian Academy of Sciences, Erich Schmid Institute of Materials Science; [3]Kompetenzzentrum Automobil- und Industrie-Elektronik GmbH; [4]University of Leoben, Institute for Mechanics

In this overview two methods to obtain the interfacial energy release rates are discussed and an outlook on thermo-mechanical fatigue of metallic films on substrate is provided: (I) In order to study the critical energy release rate for crack initiation at silicon oxide/metal interfaces, miniaturized fracture mechanic specimens are employed. Bi-material cantilevers were fabricated by FIB milling which enables high spatial resolution mechanical characterization of buried interfaces. (II) For metallic films on polymers the energy release rate is estimated from a post-buckling analysis which considers the strain energy of the buckled configuration and the energy necessary for delamination. Based on a thermodynamical model the driving force for delamination is evaluated, which allows to calculate the delamination energy release rate. (III) Finally, the influence of film microstructure for thermal fatigue damage is elaborated by comparing epitaxial and polycrystalline Al films which were cycled up to 10.000 times between 373 and 723K.

10:40 AM
Buckle Driven Delamination in Thin Gold Film-Compliant Substrate Systems: *Neville Moody*[1]; John Yeager[2]; E. David Reedy[1]; Edmundo Corona[1]; Marian Kennedy[3]; Megan Cordill[4]; David Adams[1]; David Bahr[2]; [1]Sandia National Laboratories; [2]Washington State University; [3]Clemson University; [4]Erich Schmid Institute

Film durability is a primary factor governing the use of emerging thin film flexible substrate devices where compressive stresses can lead to delamination and buckling. It is of particular concern in gold film systems found in many submicron and nanoscale applications. We are therefore studying these effects in gold on PMMA systems using compressively stressed tungsten overlayers to force interfacial failure and simulations employing cohesive zone elements to model the fracture process. Delamination and buckling occurred spontaneously following deposition with buckle morphologies that differed significantly from existing model predictions. Moreover, use of thin adhesive interlayers had no discernable effect on performance. In this presentation we will use the observations and simulations to show how substrate compliance and yielding affects the susceptibility to buckling of gold films on compliant substrates. This work was supported by Sandia National Laboratories under USDOE Contract DE-AC04 94AL85000.

10:55 AM
Strain Rate Sensitivity Effects on the Failure of Metal Films on Compliant Substrates: *Megan Cordill*[1]; Gerhard Dehm[1]; [1]University of Leoben

Polymer substrates are used in a variety of new technically advanced flexible electronics and sensors where the devices flex and stretch. Mechanical properties and interfacial phenomena of thin films on compliant substrates are important to understand in order to design reliable flexible electronic devices. These responses are commonly measured with little discussion of the viscoelastic behavior and strain rate sensitivity of the polymer substrate. Using an in-situ tensile device inside a SEM the effects of strain rate on the mechanical and interfacial properties are determined. Thin films of Cr on PET and Cr on PI systems will be examined for their use as adhesion layers. The strain of initial failure and subsequent delamination are retarded when slow rates are used, thus increasing the lifetime of the components. Other factors of the polymer substrate, such as crystallite orientation effects of the PET, will also be discussed.

11:10 AM
Fracture Properties of Fuel Cell Membranes: *Ruiliang Jia*[1]; Kemal Levi[1]; Takuya Hasegawa[2]; Jiping Ye[3]; Reinhold Dauskardt[1]; [1]Stanford University; [2]Nissan Motor Co., Ltd; [3]Nissan ARC LTD.

Perfluorosulfonic acid (PFSA) polymer membranes are widely used as the electrolyte thin films to transport protons in fuel cells. These thin membranes (thickness ~ 100 μm) represent critical small-scale components in PEM fuel cells. The fracture of the membrane including pinhole formation and micro crack propagation, is a common failure mode that limits the operational life of the cells. In the present work, we adapt thin film testing methods to assess the fracture properties of PFSA membranes under mixed-mode loading conditions in simulated fuel cell operational environments. Moreover, the microstructure of PFSA polymers is studied, which provides us with the knowledge to examine the different mechanical and fracture behaviors of Nafion and its nanocomposite membranes. This study not only reveals some significant factors that influence the fracture properties of current Nafion membranes, but also investigates the methods to improve their thermo-mechanical reliability in fuel cells.

11:25 AM
Exploiting Delamination to Fabricate Microcontact-Printed MEMS: *Corinne Packard*[1]; Vladimir Bulovic[1]; [1]Massachusetts Institute of Technology

In standard silicon-based MEMS, delamination due to residual stresses from high temperature processing and stiction from surface tension of drying solvents are two common failure mechanisms limiting yield and reliability. We have developed a method for fabricating MEMS which avoids these limitations in a room-temperature, solvent-free process that relies on intentional, controlled delamination of thin films in a microcontact printing-based process. Release of metal membranes from a carrier substrate is assisted by a nanoscale molecular organic adhesion-reduction layer, allowing suspension of crack-free films over pre-patterned supports on a target substrate. This process is additionally capable of conformal coating of curved or flexible surfaces as well as large areas, since it is not limited by wafer-scale photolithography. Contrary to silicon-based MEMS fabrication practices where poor interfacial adhesion and thin film delamination are avoided, we demonstrate that these features can instead be exploited to fabricate functional MEMS sensors and actuators.

Global Innovations in Manufacturing of Aerospace Materials: The 11th MPMD Global Innovations Symposium: Innovations in Additive Manufacturing

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Shaping and Forming Committee, TMS: High Temperature Alloys Committee
Program Organizers: Deborah Whitis, General Electric Company; Thomas Bieler, Michigan State University; Michael Miles, BYU

Tuesday AM
February 16, 2010
Room: 306
Location: Washington State Convention Center

Session Chairs: Michael Peretti, GE Aviation; Patrick Martin, AFRL-RX

8:30 AM Invited
Additive Manufacturing's Role in Fabrication and Repair of Aerospace Components: *James Sears*[1]; [1]South Dakota School of Mines and Technology

Additive Manufacturing (AM) for component repair and manufacturing offers some unique solutions for Aerospace applications. Additive Manufacturing comprises technologies that add material to existing structures for function

or performance enhancement. AM is a CAD/CAM solid freeform fabrication technology that uses metal powder and fusion for repairing or manufacturing of components. Fusion can be invoked through laser, electron beam, plasma, or ultrasonic energy sources. Inherent to AM is the ability to add material for repair or manufacture of critical GTE components with minimal heat affect to the under lying material. Also, due to the nature of AM, hard coatings can be achieved without heat treatment allowing for repair of heat-treated steels. In some cases AM repair can be used to replace hard chrome or carburized surfaces. An overview of AM developments over the years will be provided along with details of repair and manufacturing several Aerospace components.

9:00 AM

Advanced Gas Atomization Processing for Ti and Ti Alloy Powder Manufacturing: *Iver Anderson*[1]; James Sears[2]; David Byrd[3]; Joel Rieken[4]; Andrew Heidloff[4]; Mike Glynn[3]; Mark Ward[3]; [1]Ames Laboratory; [2]South Dakota School of Mines and Technology; [3]n/a; [4]Iowa State University

The feasibility of a precision ceramic pouring tube has been demonstrated for efficient production of large quantities of fine spherical powders of pure Ti and Ti alloys by an advanced gas atomization method during initial trials of Ti alloy pouring and free-fall gas atomization. The experiments at University of Birmingham utilized a novel ceramic/metal composite tundish/pour tube and existing bottom pouring cold wall crucible induction melting capability, with pouring stream temperatures measured by a 2-color pyrometer. Minimal reaction/dissolution of both pour tubes was verified by microscopic and micro-analytical examination. The trials produced a chill cast ingot and spherical powder of Ti-6Al-4V (wt.%) and the composition and microstructure of both also were analyzed. Progress on close-coupled gas atomization studies at Iowa State University also will be reported. Work supported by Iowa State University Research Foundation and the Grow Iowa Values Fund and performed at Ames Lab under contract no. DE-AC02-07CH11358.

9:20 AM

Cold Spray Characteristics of Ti-6Al-4V Coating: *Ahmad Rezaeian*[1]; Eric Irissou[2]; Steve Yue[1]; [1]McGill University; [2]National Research Council Canada (NRC)

Cold spray process is a novel technique used to consolidate particles in order to manufacture complex parts or additive features with minimal waste. The advantages of this technology as compared with conventional thermal spraying techniques have been motivation for the recent investigations. Despite FCC structured materials such as Ni and Cu, aerospace materials such as Titanium alloys exhibited limited deformation upon impact which is key parameter to obtain dense coatings. In this work, Ti-6Al-4V powder was cold sprayed on steel and Ti-6Al-4V substrates using CGT 4000 gun. Gas temperature, pressure and stand-off distance were calibrated to achieve the maximum particle velocity. The gun traverses speed as well substrate thickness /material were varied in order to evaluate the effect of heat introduced during spray on the powder deformability and therefore the coating properties. Deposition efficiency, fracture features and microstructural changes were investigated by optical microscopy, XRD, SEM, and microhardness.

9:40 AM

Additive Manufacturing of Gamma Titanium Aluminide Parts by Electron Beam Melting: Silvia Sabbadini[1]; Oriana Tassa[2]; Paolo Gennaro[3]; *Ulf Ackelid*[4]; [1]Avio SpA; [2]Centro Sviluppo Materiali S.p.A; [3]ProtoCast S.r.l.; [4]Arcam AB

In recent years, Electron Beam Melting (EBM) has matured as a technology for additive manufacturing of dense metal parts. The parts are built by additive consolidation of thin layers of metal powder using an electron beam. With EBM, it is possible to create parts with geometries too complex to be fabricated by other methods, e.g. fine network structures and internal cavities. The process is run in vacuum, which makes it well suited for materials with a high affinity to oxygen, e.g. titanium compounds. We present material data from a recently developed EBM process for gamma titanium aluminide, Ti-48Al-2Cr-2Nb, including a microstructure investigation and a high temperature study of tensile and creep properties.

10:00 AM Break

10:20 AM

Properties of Ti-5Al-5Mo-5V-3Cr Samples Produced via Powder Hot-Isostatic-Pressing: *Nick Wain*[1]; Xinjiang Hao[1]; Ravi Swamy[1]; Xinhua Wu[1]; [1]The University of Birmingham

Hot-isostatic-pressing ("HIPping") of powder is an effective manufacturing technique: it allows the production of complex parts to very near net-shape and is therefore highly material-efficient. In this work, Ti-5Al-5Mo-5V-3Cr ("Ti-5553"), a high-strength metastable beta Ti alloy, was HIPped from powder to produce monolithic parts. The effects of powder production route, alloy composition and HIPping conditions on the microstructure and properties of the HIPped Ti-5553 were assessed. The difference between gas atomised powders and PREPed (plasma-rotating-electrode processed) powders has been studied, together with the influence of minor carbon addition on HIPped microstructure. Mechanical testing showed the effect of these different microstructures on tensile strength, fatigue and fracture toughness. Mechanical properties were often found to be comparable to those of Ti-5553 alloy produced from ingot. The significance of the results on the viability of HIPped beta Ti alloys is discussed.

10:40 AM

The Influence of Thermal History on the Microstructure of Laser-Additive-Manufactured Ti-6Al-4V Samples: *Xinhua Wu*[1]; Laura Qian[1]; Junfa Mei[1]; [1]The University of Birmingham

The microstructure and mechanical property of laser-additive manufactured Ti64 have been assessed. A 3D-transient finite element model has been developed for predicting the temperature history of laser-fabricated Ti6Al4V thin wall samples. The model has been validated by direct measurements of thermal history throughout the height of fabricated samples. The effects of location, laser scanning speed and laser power on the thermal profile have been modelled and compared with temperatures measured. The microstructures in direct laser fabricated Ti-6Al-4V under most conditions are dominated by columnar grains which contain martensites and this is consistent with those expected on the basis of the measured and predicted thermal histories of the different samples and for the different regions within samples.

11:00 AM

Fabrication and Characterization of Reticulated, Porous Mesh Arrays and Foams for Aerospace Applications by Additive Manufacturing Using Electron Beam Melting: *Sara Gaytan*[1]; L.E. Murr[1]; F. Medina[1]; E. Martinez[1]; L. Martinez[1]; R.B. Wicker[1]; [1]UTEP

Variously non-isotropic arrays of reticulated mesh geometries and isotropic foam structures of Ti-6Al-4V have been fabricated by additive manufacturing (AM) using electron beam melting (EBM). These arrays consist of complex, monolithic prototypes which include various mesh geometries and foam structures with a range of densities, including fully dense geometries. Density and stiffness variations have been fabricated for aeronautics/aerospace applications and these complex arrays provide unique energy or impact absorption features, thermal management, stiffness and strength in sandwich cores, and excellent corrosion resistance. These monolithic mesh arrays have been fabricated using geometrical unit cells whose bases and size variations can allow density, porosity, stiffness, and strength tailoring to fabricate multifunctional materials prototypes while foam components have been fabricated from CAD models based on CT-scans of common aluminum alloy foams. The microstructures of these prototypes have been characterized by optical and electron microscopy.

11:20 AM

Properties and Microstructure of Net Shape HIPped Ti6Al4V Components: Kun Zhang[1]; Junfa Mei[1]; *Xinhua Wu*[1]; [1]The University of Birmingham

The microstructures and mechanical properties of HIPped (Hot Isostatically Pressed) Ti6Al4V have been assessed on samples which have been manufactured from PREP powder. In preliminary experiments the HIPping conditions have been varied in order to identify the optimum HIPping parameters and a detailed study has been made on optimally HIPped samples. Test samples have been manufactured where the as-HIPped surface is retained and others where the as-HIPped surface has been removed by machining or electropolishing before testing. It has been shown that HIPped samples, manufactured using conventional HIPping, have rough surfaces on the scale of the powder diameter which leads to poor fatigue properties. A new HIPping procedure has been developed in which this surface roughness is eliminated and the fatigue properties correspondingly

improved. Details of the microstructure of the HIPped PREP powder will be presented and correlated with the tensile, fatigue and fracture toughness.

11:40 AM

Influence of Powder Particle Size on the Microstructure and Properties of HIPped Ti Alloy Powders: *Kun Zhang*[1]; Junfa Mei[1]; Xinhua Wu[1]; [1]The University of Birmingham

Two alloy powders(Ti-6Al-4V and Ti-25V-15Cr-2Al-0.2C(wt%)have been Hot Isostatically Pressed (HIPped) and the influence of particle size on the microstructure and mechanical properties assessed.The individual powders have very different microstructures with the Ti6Al4V being martensitic and the Ti-25V-15Cr-2Al-0.2C being retained beta and hence the response of these two different microstructures to HIPping is very different.The size range of the supplied powders was from 50–400µm.Powders were sieved to produce batches with sizes of 50~150µm,250~400µm and 50~400µm and these together with a batch using the whole size range were HIPped for both alloys.The properties of samples HIPped using the whole size range(50-400µm)powder were superior to those made using individual size fractions.For both alloys the smallest particle size fraction had lower ductility and smoother fracture surfaces than the large particle size. These observations are discussed in terms of the microstructures developed during HIPping and of the factors which influence the properties of HIPped powders.

Heterogeneous Nucleation and Initial Microstructure Evolution in Alloys and Colloids: Experiment I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS/ASM: Phase Transformations Committee

Program Organizers: Rainer Schmid-Fetzer, Clausthal University of Technology; Heike Emmerich, RWTH Aachen University; Frans Spaepen, Harvard University; Martin Glicksman, University of Florida; John Perepezko, University of Wisconsin, Madison

Tuesday AM Room: 614
February 16, 2010 Location: Washington State Convention Center

Session Chairs: David StJohn, University of Queensland; Heike Emmerich, RWTH Aachen

8:30 AM Invited

Heterogeneous Nucleation in Peritectic Systems: *Rohit Trivedi*[1]; Jongho Shin[1]; John Perepezko[2]; [1]Iowa State University; [2]University of Wisconsin-Madison

Heterogeneous nucleation is investigated through directional solidification experiments in the Sn-Cd peritectic system in which nucleation plays a key role in the formation of repeated bands of primary and peritectic phases. Nucleation undercooling for each phase is determined through the measurements of temperature and composition at the location where each band nucleates. Nucleation is found to occur at the ampoule wall-liquid-solid triple junction, and nucleation undercooling for each phase is found to be very small, from a fraction of a degree to about one degree. Relevant contact angles have been measured and it is shown that experimental nucleation undercooling values for the two phases are orders of magnitude smaller than those predicted by the classical nucleation theory. A mechanism of heterogeneous nucleation is proposed to explain the experimental results.

8:55 AM

Heterogeneous Nucleation in Charged Colloidal Model Systems Induced by Seeds of Various Shape and Structure: *Hans Joachim Schöpe*[1]; Patrick Wette[2]; Andreas Engelbrecht[1]; Markus Franke[1]; [1]University of Mainz; [2]Deutsches Zentrum für Luft- und Raumfahrt

Understanding the process that drives an undercooled fluid to the crystal state is still a challenging issue for condensed matter physics and plays a key role designing new materials. Crystallization undergoes crystal nucleation, growth and ripening. The crystallization kinetics and the resulting polycrystalline morphology are given by a complex interplay of these mechanisms. A great deal of progress has been made in recent years using colloidal suspensions as model system studying crystallization. Close analogies to atomic systems are observed which can be exploited to address questions not accessible in atomic solidification.We here present systematic measurements of the crystallization kinetics of a charged colloidal model system adding small amounts of seeds using time resolved scattering techniques. We report how the seeds size, structure and concentration affect crystal nucleation and growth as function of meta stability giving the possibility to control the crystallization process and the resulting microstructure of the polycrystal.

9:15 AM

Colloids as Model Systems for Undercooled Metallic Melts: *Ina Klassen*[1]; Patrick Wette[1]; Dirk Holland-Moritz[1]; Thomas Palberg[2]; Dieter M. Herlach[1]; [1]German Aerospace Center; [2]Johannes Gutenberg Universität Mainz

We present investigations of short-range order (SRO) of charged colloidal suspension by applying Ultra-Small-Angle X-ray Scattering using synchrotron radiation at DESY Hamburg. The colloidal system is shear melted, subsequently, it is in a liquid state far from equilibrium. The development of the structure factor is measured as a function of time after shear melting. The deviation from equilibrium is determined by the difference of the chemical potential between solid and liquid state. The structure factors as measured are analysed within a cluster model. The results reveal the preference of icosahedral SRO in the metastable liquid colloidal system. With decreasing deviation from equilibrium the icosahedral SRO is reduced and eventually vanishes at equilibrium. The SRO of the stable liquid state is determined to be fcc like. The results obtained from the colloidal suspensions are compared with previous investigations of SRO in undercooled melts of pure metals studied by elastic neutron diffraction.

9:35 AM

Competition between Heterogeneous and Homogeneous Nucleation near a Flat Wall: Patrick Wette[1]; Hans-Joachim Schöpe[2]; Ina Klassen[1]; *Dieter Herlach*[1]; [1]German Aerospace Center; [2]Johannes Gutenberg Universität Mainz

The competition between heterogeneous and homogeneous nucleation of a charged colloidal suspension is studied close to container walls. Colloidal crystals were shear melted and a metastable melt left to solidify afterwards. The crystallization kinetics was monitored using time resolved Ultra Small Angle X-ray Scattering (USAXS) at different degrees of metastability. Whereas at high metastability the homogeneous nucleation dominates the solidification scenario leading to a small amount of oriented wall based crystals. At lower metastability the homogeneous nucleated crystals do crystallize first while the crystallization of the wall crystal is delayed. When the amount of solid crystallized near the wall shows its fasted increase the amount of polycrystalline crystal decreases indicating that the randomly oriented polycrystal is converted into a wall crystal finally resulting in a large amount of oriented wall based material. Our experiments demonstrate the complexity of the crystallization process of colloidal suspension close to container walls.

9:55 AM

Preliminary Investigation of the Nucleation of Si in Entrained Droplets in High Purity Al Alloys: Muhammad Zarif[1]; Brian McKay[1]; *Peter Schumacher*[1]; [1]University of Leoben

An Al-5wt%Si master-alloy was produced using super purity Al 99.99 wt% and Si 99.999 wt% materials in an arc melter under a 200 mbar reduced Ar atmosphere. Using a meltspinner the master alloy was melt-spun by ejecting the melt with an ejection pressure of 100 mbar, at a temperature of 750 °C onto a Cu wheel, rotating at a circumferential wheel speed of 15 ms^{-1}. This resulted in the production of a ribbon ~3 mm in width and ~80 µm thick. Using optical microscopy entrained droplets of Al-Si eutectic within Al grains and at Al grain boundaries were observed. Differential scanning calorimetry and transmission electron microscopy techniques were subsequently employed to investigate the effect of Sr additions on eutectic undercoolings and to examine nucleation phenomenon, respectively.

10:15 AM Break

10:35 AM Invited

Study of Heterogeneous Nucleation in High Purity Al-Si Alloys with Sr Addition: *Peter Schumacher*[1]; [1]University of Leoebn

Metallic glasses give a unique opportunity to study nucleation as the glass forming ability of the alloys slows nucleation kinetics sufficiently down to observe individual nucleation events. These may occur above the glass transition temperature and are subsequently quenched in or alternatively

nucleation is triggered below the glass transition by e.g. annealing. In particular the deliberate addition of particles into glasses permit to study their effect on heterogeneous nucleation of crystals. Extensive studies of particles used in grain refining of Al alloys such as Al3Ti and TIB2 have been investigated in Al based glasses. Thereby, it was possible to distinguish between nucleation events occurring in both metallic glasses and conventional Al- alloys and growth effects dominating grain refinement in conventional alloys. This paper gives an overview on nucleation phenomena observed on added heterogeneous nucleation sites in Al based metallic glasses and their relevance to industrial casting practice.

11:00 AM
Embryonic Crystallization in Al-Based Marginal Glass Forming Alloys: *Eren Kalay*[1]; Matthew Kramer[1]; Scott Chumbley[1]; Iver Anderson[1]; Ralph Napolitano[1]; [1]Ames Laboratory

The Al-rich Al-RE and Al-TM-RE (TM: transition metals; RE: rare earth elements) marginal glass-forming alloys have attracted much attention due to unique devitrification products with a very high number density of nuclei on order of 10^20 to 10^24m^-3. A mechanism to explain such high nanocrystal densities has not been identified to date. In this study Al-Sm and Al-Tb systems were chosen to investigate this unusual nucleation phenomenon in marginal glass forming alloys. Both liquid and as-quenched structures were analyzed using a combined study of high energy X-ray diffraction (HEXRD), 3-D atom probe tomography and transmission electron microscopy (TEM). The transformation kinetics and microstructural evolution during initial crystallization was investigated using differential scanning calorimetry, and TEM. A new nucleation model based on the existence of the RE-rich clusters in the as-quenched state promoting high nucleation density of fcc-Al nanocrystals will be discussed. Research supported by U.S. DOE-OS, Ames Laboratory contract No.DE-AC02-07CH11358.

11:20 AM
Intrinsic Heterogeneous Nucleation in Eutectic Systems: *Joachim Bokeloh*[1]; Gerhard Wilde[1]; [1]Universität Münster

Experimental studies on nucleation kinetics in metallic systems are often impaired by the unknown identity of the nucleant phase and by the convoluting contributions of different nucleation mechanisms that might be active simultaneously in droplet ensembles. With respect to these issues, the solidification pathway of near-eutectic alloys offers the opportunity to study the kinetics of heterogeneous nucleation on intrinsic nucleant phases. If the melt is cooled below its liquidus, the first crystalline phase can form. Upon cooling the specimen below the solidus line, this first crystalline phase can then serve as pre-defined heterogeneous nucleant for the nucleation of the second crystalline phase. Moreover, analyzing repeated freezing cycles on single droplets prevents the convoluting contributions from different mechanisms in droplet populations. A statistically relevant data set for this reaction in the Ag-Cu system obtained by repeated differential thermal analysis cycles is presented and the kinetics of the nucleation reaction is evaluated.

Hume-Rothery Symposium: Configurational Thermodynamics of Materials: Session III

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Chris Wolverton, Northwestern University; Mark Asta, University of California, Davis; Gerbrand Ceder, Massachusetts Institute of Technology (MIT)

Tuesday AM Room: 212
February 16, 2010 Location: Washington State Convention Center

Session Chair: To Be Announced

8:30 AM Invited
Ab Initio Modeling of the Lattice Stability of W in the Presence of Interstitials: *Marcel Sluiter*[1]; [1]TU Delft

In recent years the ab initio study of alloy phase stability, as pioneered by Prof. de Fontaine, has found wide application. Especially in the area of phase diagram modeling ab initio studies have rapidly advanced. Here we examine the W-rich side of the W-O phase diagram. It has long been known that oxygen can stabilize the so-called beta-W structure, now better known as the Cr3Si prototype. So far it has remained unclear how oxygen affects the lattice stability of W. Here ab initio methods are used to examine the various ways in which oxygen can, or cannot be, present in W. A model for O in W is proposed which explains several phenomena observed in W thin films.

9:00 AM Invited
Alloy Thermodynamics without Lattice Stability?: *Axel van de Walle*[1]; [1]Caltech

Both the cluster expansion and the CALPHAD formalisms are implicitly based on the assumption that some well-defined lattice(s) (e.g. bcc, fcc, hcp) remain at least metastable throughout the whole composition range of an alloy system. However, this assumption is clearly violated in a number of systems (e.g. Ti-Al, Cu-Fe, etc.). This conundrum is generally addressed by viewing the free energies of unstable phases as merely convenient extrapolations without a physical meaning. We describe a more rigorous way to handle this issue, based on the idea that the (classical) partition function of the system can be cleanly factored as nested sums over lattices, over configurations and over displacive degrees of freedom. In this approach, a well-defined free energy can be assigned to mechanically unstable phases by constraining the domain of the integration traditionally employed to obtain free energies in lattice dynamics calculations, based on the geometrical features of the lattices involved.

9:30 AM Invited
DFT, CE, CALPHAD and Phase Field, Cooperative Phenomena Powered by DDF: *Suzana Fries*[1]; [1]ICAMS, Ruhr University Bochum

There are a series of methods for treating special features of materials which were independently developed in the last 60 years. A magnification of understanding is achieved by the combination of these methods. Because they extend from first-principles calculations to the processing of real alloys their link is not straightforward requiring a global vision of the studied event. This is possible only by an investigator carrying a certain scientific culture. Once this culture is transmitted the combination of these methods becomes more and more creative, and as a quality inherent to the culture, the development keeps tracing the subject as a whole.These phenomena will be exemplified by the achievements obtained by a large group of researchers which were touched by the DDF culture

10:00 AM Break

10:30 AM Invited
First-Principles Calculations of Free Energies of Unstable Phases: *Vidvuds Ozolins*[1]; [1]University of California, Los Angeles

Ab initio density-functional theory molecular dynamics simulations are used to solve the long-standing problem of calculating free energies of harmonically unstable phases, such as fcc W. We find that fcc W is mechanically unstable with respect to long-wavelength shear at all temperatures considered (T>2500 K), while the short-wavelength phonon modes are anharmonically stabilized. The calculated fcc/bcc enthalpy and entropy differences at T=3500 K (308 meV and 0.74 kB per atom, respectively) agree well with recent values derived from CALPHAD analysis of experimental data. Our results for Zr show that even harmonically stable phases (such as fcc and hcp Zr) become dynamically unstable at high temperatures. The proposed methods are expected to be useful for modeling the thermodynamic properties of solid phases and thermodynamic driving forces for structural transformations in pure elements and alloys using first-principles density-functional theory techniques.

11:00 AM
Another View of Phase Diagrams: *John Morral*[1]; Ximiao Pan[1]; [1]Ohio State University

With improved software and databases that predict phase diagrams there is need for new, user-friendly ways to view ternary and higher order phase diagrams. Two dimensional (2D) sections are well known ways to view information about phase stability, even when multiple components and phases are present. Three dimensional projections of ternaries are well known, too, especially liquidus projections. These are available for a variety of ternary systems. Liquidus and other projections in the literature are a series of lines that border single phase regions. On 2D phase fraction charts these are lines where the fraction equals one. However another type of diagram can be drawn using

lines where the phase fraction is zero. An example of such a diagram will be given in which the Q-phase stability in Al-Si-Cu-Mg alloys can be viewed as a function of three solute concentrations. The diagram is a polyisopleth projection at the solutionizing temperature.

11:20 AM Invited
The Kinetics of Diffusional and Structural Phase Transformations from First Principles: *Anton Van der Ven*[1]; [1]University of Michigan

While much progress has been made in the first-principles prediction of the thermodynamics of multi-component solids, predicting the kinetics of solid-state phase transformations remains a major challenge. A large class of phase transformations in multi-component solids involves a redistribution of its constituents, which requires atomic diffusion. Other phase transformations are characterized by structural changes at the crystallographic level. First-order phase transformations require the passage of interfaces separating new phases from old phases. One approach to simulate first-order phase transformations from first-principles is through parameter passing, whereby kinetic coefficients describing atomic diffusion and interface mobilities are implemented in a continuum description. In this talk I will describe first-principles approaches to predict coefficients of kinetic rate equations in multi-component alloys and intercalation compounds used in Li-ion batteries. Kubo Green expressions for rate coefficients are evaluated by applying kinetic Monte Carlo simulations to first-principles parameterized effective Hamiltonians.

11:50 AM Invited
Vibrational Thermodynamics at High Temperatures: *Brent Fultz*[1]; [1]California Institute of Technology

Differences between the vibrational entropies of alloy phases have become much better understood over the past decade. Our understanding is best for low temperatures, where it is often possible to use the harmonic approximation. Vibrational entropy at elevated temperatures is a bigger challenge. Only part of the story is provided by the quasiharmonic approximation, where vibrational frequencies are reduced as a crystal expands against its bulk modulus. Existing phonons alter the energy to excite other phonons (phonon-phonon interaction, PPI), and electronic excitations interact with vibrational excitations (electron-phonon interaction, EPI). It has been a surprise that the EPI in metals can be important thermodynamically at temperatures of 1000 K. One of the more prominent effects of the PPI is the lifetime broadening of phonon spectra, which highlights other risks of the quasiharmonic approximation. Nevertheless, systematic trends for the PPI in fcc and other structures will be reported.

International Symposium on High-Temperature Metallurgical Processing: Secondary Processing

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
Program Organizers: Jaroslaw Drelich, Michigan Technological University; Jiann-Yang Hwang, Michigan Technological University; Tao Jiang, Central South University; Jerome Downey, Montana Tech

Tuesday AM Room: 619
February 16, 2010 Location: Washington State Convention Center

Session Chair: Jiann-Yang Hwang, Michigan Technological University

8:30 AM Keynote
Overcoming the Final Challenges to Initiating Production Treatment of EBR-II Spent Fuel at Idaho National Laboratory: *Michael Simpson*[1]; [1]Idaho National Laboratory

Since its successful demonstration for DOE from 1996 to 1999, the Experimental Breeder Reactor-II Spent Fuel Treatment Process has been used to treat a total of 2.6 metric tons of fuel in addition to the 1.1 MT treated during the demonstration. And a number of evolutionary-type improvements have been made in the process to improve efficiency and minimize waste. Idaho National Laboratory is now on the cusp of initiating production operations at up to 3.4 MT/year. Key pre-requisites for such a production level include funding to support 24-hour operations. And key technology issues must be resolved including electrorefiner (ER) oxidant production, improving design of the ER anode-cathode module, developing a process for treating dross, scaling-up and qualifying the ceramic waste process, and renovating the mass tracking system that is currently used for material control and accountability. Each of these areas is currently being worked, and progress is rapidly being made.

9:10 AM
Recovery of Iron and Zinc from Electric Arc Furnace Dust Using a Microwave Processing Method: *Jiann-Yang Hwang*[1]; Xiang Sun[1]; Xiaodi Huang[1]; [1]Michigan Technological University

In the United States, there are approximately 700,000 to 800,000 tonnes of Electric Arc Furnace dusts generated each year. This material is rich in the oxides of iron, zinc, lead and chromium. It has been classified as a hazardous material and requires special disposal. A microwave processing method was investigated in this study. This method employs the mixtures of dust and powders of reductant under microwave for heating while the air is prevented from the system to cause the oxidation of the products. The products are the iron and zinc metals that can be easily recycled.

9:30 AM
Reduction Behavior of Chromium Oxide in Molten Stainless Steel Slag with Graphite: *Qiuju Li*[1]; [1]Shanghai University

To recover chromium in stainless steel slag, the direct smelting reduction of chromium oxide in molten slag was investigated by small furnace experiments. Graphite was used as reductant under conditions of 1823-1873K. The distribution ratio of chromium was addressed by investigating the thermodynamic equilibrium between the liquid iron and CaO-MgO-SiO2-Al2O3-CrOx-FetO stainless steel slag. Effects of temperature, slag basicity and composition on the reduction of chromium and iron oxides in the slag were investigated. The slag basicity had a significant effect on the reduction behavior. Increasing Al2O3 content from 0 to 10mass% in initial slag, reduction results showed that Al2O3 content in slag increased, the content of chromium and iron decreased in slag.

9:50 AM
A Novel Process for Preparing Ferronickel Powder from Laterite Ores: *Guanghui Li*[1]; Mingjun Rao[1]; Tao Jiang[1]; Yuanbo Zhang[1]; Qian Li[1]; [1](School of Minerals Processing and Bioengineering, Central South University

The traditional pyrometallurgical and hydrometallurgical processes for nickel recovery from laterite ores are characterized as either great energy consumption or low efficiency. In this paper, reduction roasting-magnetic separation process with addition of composite additive was developed to process the ores. Ferronickel powders with 6.5% to 8.0% Ni content, and 80% to 86% total iron grade were obtained from a sample of laterite ore with 1.58% Ni and 22.07% total iron grade; the recovery of nickel and iron is 80%~91% and 60%~74% respectively. The reduction behaviors and the function of composite additive were primarily involved. It was shown that composite additive not only promotes metallic iron grain growth but also reacts with gangues in the laterite to form non-magnetic or weak-magnetic matters, which are easily removed by low intensity magnetic separation.Key Words: Laterite, Composite additive, Direct reduction, Ferronickel

10:10 AM
Phase Equilibria in Ferrous Calcium Silicate Slags: Stanko Nikolic[1]; Hector Henao[2]; *Peter Hayes*[2]; Evgueni Jak[2]; [1]Xstrata Technology; [2]University of Queensland

Ferrous calcium silicate slags (described by the FeO-Fe2O3-CaO-SiO2 system) form the basis of many non ferrous slags used in primary and secondary metallurgical processes. Despite the industrial and scientific importance of this system, the phase equilibria have not been fully investigated over the range of process conditions encountered in industrial practice. This is on the one hand surprising but also understandable because of the inherent difficulties of undertaking accurate measurements of the system under controlled conditions. Characterization of this slag system is necessary to improve the optimization of existing metallurgical processes and to design new technologies.Sophisticated new experimental methods have been developed to investigate the phase equilibria of these slags. Using these techniques liquidus and solidus data can now be obtained for all primary phase fields in this system. Experimental data obtained to date have revealed interesting trends and some unexpected and important findings about the slag chemistry.

10:30 AM Break

10:45 AM

Decomposition/Volatilization of Enargite in Nitrogen-Oxygen Atmosphere: *Rafael Padilla*[1]; Alvaro Aracena[1]; Maria Ruiz[1]; [1]University of Concepcion

Enargite (Cu_3AsS_4) occurs as impurity in most Chilean copper sulfide minerals. However, when enargite content in copper concentrates is high (>1.5%), smelting of such a concentrate produces pollution problems and contamination of the final copper with arsenic. Thus understanding the behavior of enargite at high temperatures is crucial. In this paper, research data is discussed on the decomposition/volatilization of natural enargite mineral in nitrogen and oxygen-containing atmospheres by thermogravimetry and DTA analyses. The results indicated that enargite decomposes in nitrogen through intermediate tenantite to form Cu_2S in the range 600-900°C. However, in the presence of oxygen, enargite reacts forming CuO as final product. Temperature affects significantly the arsenic volatilization rate in inert and oxidizing atmospheres. At 900°C in pure nitrogen, 95% arsenic volatilized in 15 min, while in the presence of 1% oxygen, 95% of arsenic volatilized in 6 min. At higher oxygen concentration, the rate is even faster.

11:05 AM

Influence of Additives on Dephosphorization of Oolitic Hematite by Direct Reduction Process: *Guanghui Li*[1]; Chaoming Xie[1]; Yuanbo Zhang[1]; Qian Li[1]; Tao Jiang[1]; [1]School of Minerals Processing and Bioengineering, Central South University

With the rapid development of iron and steel industry in the world, the demand and consumption of iron ores increase continuously. Large reserves of high phosphorus content hematite resources have been found in China, but traditional separation methods are found invalid to treat the ore due to high phosphorus content(0.4%~1.0%),low total iron grade(35%~50%) and fine dissemination size of iron-oxide grain. Based on the investigation of mineralogy of oolitic hematite ore, additive aided direct reduction-magnetic separation process is developed to process the ore in this study. The effects of additives, parameters of reduction and magnetic separation are tested. The metallic iron concentrate with total iron grade of 93.84% and phosphorous content of 0.083% has been obtained in laboratory for an ore sample with total iron grade of 48.96%, phosphorous content of 1.61%; the iron recovery is 93.0%, and dephosphorization reaches 96.74%.

11:25 AM

Thermodynamic Analysis and Experimental Test of primary Al-Si Alloy Prepared by Carbothermal Reduction of Bauxite Tailings: Yang Dong[1]; *Di Yuezhong*[1]; [1]Northeastern University

The thermodynamic analysis on the carbothermal reduction of bauxite tailings was carried out. The carbothermal reduction process, effect of Fe2O3 addition on the reduction reaction and the composition of products were studied by various methods such as TG/DTA, XRD, XRF, SEM-EDS. It was found that Fe2O3 could not only reduce the reaction temperature and the yield of SiC, but also improve the reduction ratio of Al2O3.The proper temperature was 1900° in the carbothermal reduction of bauxite tailings. The carbothermal reduction process could be divided into 4 stages, and the key stage was the formation and decomposition of carbides stage. Besides Al-Si-Fe phases, there were residual Al2O3 and carbides phases in the products.

11:45 AM

Thermodynamic Study on the Recovery of Vanadium from Low-Vanadium Hot Metal: *Xuemei Qing*[1]; Bing Xie[1]; Qingyun Huang[1]; Jianping Xiao[1]; [1]Chongqing University

Thermodynamic calculation software FactSage was used to calculate the thermodynamics of the oxidation reaction of carbon/silicon/vanadium etc in low-vanadium hot metal, the oxidation behavior of the elements in hot metal and the valencies of vanadium and iron in vanadium slag were analyzed. The vanadium slag which was obtained by simulating the reaction of vanadium recovery in laboratory were analyzed by x-ray diffraction technique and spectral analysis technique to verify the theoretical derivation results, and the effects of ferrous oxide contents on the grade of vanadium slag were also studied. Meanwhile, the effects of temperature on the equilibrium distribution ratios of carbon/silicon/vanadium etc were studied by the thermodynamic equilibrium experiments.

12:05 PM

Effect of Damp Grinding on Preparation of Oxidized Pellet from Pyrite Cinders Concentrates: Jianchen Li[1]; Guohua Bai[1]; Guanghui Li[1]; Xiaoqing Zhou[1]; [1]School of Minerals Processing and Bioengineering; Central South University

Pyrite cinders (PyCs) are by-products of sulphuric acid production, and they usually contain variable iron oxides; therefore, PyCs are regarded as a kind of iron bearing resources. Many investigations have been carried out to produce oxidized pellet with PyCs concentrates after beneficiation in lab, but its industrial application encounters embarrassment due to inferior ballability and low induration strength of fired pellet. In this study, damp grinding was adopted to enhance the pelletization of PyCs concentrates. It is shown that strength of green ball is able to be increased obviously when PyCs concentrates are ground for 4min at 10% moisture. The roasting experiments in small scale and pilot scale indicate that the compression strength of fired pellet is also able to be increased after damp grinding, their strength go up to 3332N/P, which meets requirements of blast furnace.

Jim Evans Honorary Symposium: Flow, Solidification, and Inclusion Behavior in Casting Processes

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division
Program Organizers: Ben Li, University of Michigan; Brian G. Thomas, University of Illinois at Urbana-Champaign; Lifeng Zhang, Missouri University of Science and Technology; Fiona Doyle, University of California, Berkeley; Andrew Campbell, WorleyParsons

Tuesday AM Room: 620
February 16, 2010 Location: Washington State Convention Center

Session Chair: Vaughan Voller, University of Minnesota

8:30 AM Introductory Comments

8:40 AM

Inclusion Motion and Removal in a Steel Slab Continuous Casting Strands under EMBr: *Lifeng Zhang*[1]; Yufeng Wang[1]; [1]Missouri University of Science and Technology

In current study, a 3-D numerical model is built to investigate the effect of a local type ElectroMagnetic Brake (EMBr) on the fluid flow, heat transfer and inclusion motion in slab continuous casting strands. The results indicate that the magnetic force affects the jet characteristics, including jet angle, turbulent kinetic energy and its dissipation rate. In order to reduce the top surface velocity and stabilized the top surface, the magnetic flux intensity should larger than a critical value. With a 0.39 T magnetic flux intensity, the top surface velocity and its fluctuation can be well controlled, and less slag is entrained. The motion of argon bubbles is also studied. More bubbles, especially >2.0 mm bubbles, escape from the top surface between the mold Submerged Entry Nozzle (SEN) and ¼ width for the case with a 0.39 T EMBr. This may push the top slag away and create an open "eye" on the top slag. Small bubbles (=1mm) tend to escape from one side of wide face no matter with or without EMBr, which is induced by the swirl flow from the SEN outport. EMBr has a little effect on the overall removal fraction of inclusions; however, it affects the local distribution of inclusion in the slab. With EMBr, more inclusions accumulate the region just below the surface, thus a worse subsurface quality, while the inner quality of the slab is better than that without EMBr. For heat transfer in the mold, the heat flux on the narrow face and the area of possible break-out zones can be reduced by using EMBr. Prevention of bias flow and/or asymmetrical flow in mold by EMBr is also concluded.

9:05 AM

A General Enthalpy Method for Molding Solidification Phenomena: *Vaughan Voller*[1]; [1]University of Minnesota

The development of fast, accurate, and robust computation methods is crucial to the modeling of fluid flow and solidification in continuous casting. A basic solidification modeling approach, nearing 60 years of use, is to the enthalpy formulation; an approach that is able to provide numerical solutions on fixed grids. Enthalpy methods work well when the solid/liquid interface

is at a constant phase-change temperature. In a general solidification system, however, the phase change temperature may be a function of (i) the curvature of the interface, (ii) the speed of the interface, and (iii) the redistribution of solute phases. In this paper it is first shown that the modest enthalpy method can be easily and straightforwardly modified to model all of three of these situations. This modified approach is then verified by comparison with available analytical solutions and alternative more sophisticated front tracking approaches.

9:30 AM

Modelling of Mould Filling in Open Mould Conveyor Ingot Casting: *Vu Nguyen*[1]; Patrick Rohan[1]; John Grandfield[2]; Kevin Naidoo[3]; Kurt Oswald[3]; [1]CSIRO; [2]Grandfield Technology; [3]o.d.t. Engineering

This paper describes the modelling procedure and compares the predicted flow patterns during mould filling with those obtained from casting trials for CASTfill - a patented low dross filling system used in open mould chain conveyor ingot casting machines. The results showed the predicted flow patterns were in good agreement with those observed during casting trials. The effects of flow patterns and the predicted aluminium oxide generation on ingot appearance are also discussed.

9:55 AM

Influence of a Plunging Liquid Jet on a Dual Alloy Casting: *Autumn Fjeld*[1]; Abdellah Kharicha[1]; Andreas Ludwig[1]; [1]University of Leoben

In casting processes with an impinging jet pouring stream high levels of turbulence, mixing and entrained phases are introduced into the molten metal. For very large scale castings the impinging jet approaches 6 m long, reaching high velocities with high impact force. The impact of such a pouring stream creates highly turbulent zone at depths up to 1.5 m below the surface of the molten metal. The intensity of this impact driven flow is of particular interest in the case of dual alloy castings in which one alloy is cast into a precast shape of another alloy. This results in a remelting of the precast alloy and subsequent solidification of a mixed bonding zone between the two alloys. Investigations with a numerical model have shown that understanding the interaction between the impinging jet, flow patterns and melting is critical to ensuring optimal amount of remelting and bonding between the alloys.

10:20 AM Break

10:35 AM

Centrifugal Casting of Complex Geometries: Computational Modelling and Validation Experiments: *Diane McBride*[1]; Nick Croft[1]; D. Shevchenko[2]; N. Humphreys[2]; P. Withey[3]; N. Green[2]; Mark Cross[1]; [1]Swansea University; [2]The University of Birmingham; [3]Roll Royce Plc

Centrifugal casting offers one route through to high quality products in difficult to cast high temperature low superheat alloys. The coupling of free surface flows and complex rotating geometries, results in significant centrifugal forces; combined with rapid heat transfer and solidification this yields a significant computational modelling challenge. The objective of the work reported here is to develop a comprehensive computational model of centrifugal casting that can reliably predict the macro-defects that arise from the process. In this contribution we describe: • The development of the computational model yielding simulations which involve of the order of a million elements with thousands of time steps on large parallel clusters • Experimental data to validate the model, and • The configuration of a full scale computational model capturing all the important macro-phenomena.

11:00 AM

Integration of CFD Simulation and Virtual Reality Visualization for Iron and Steel Making: *Chenn Zhou*[1]; [1]Purdue University Calumet

Computational Fluid Dynamics (CFD) has become a powerful simulation technology used in many industrial applications for process design and optimization to save energy, improve environment, and reduce costs. In order to better understand CFD results and more easily communicate with non-CFD experts, advanced virtual reality (VR) visualization is desired for CFD post-processing. Efforts have recently been made at Purdue University Calumet to integrate VR with CFD to visualize complex data in three dimensions in an interactive, virtual environment. The virtual engineering environment greatly enhances the value of CFD simulations and allows engineers to gain much needed process insights for the design and optimization of industrial processes. Examples of a number of applications to iron and steel making will be presented.

11:25 AM

Surface Tension and Temperature Effect on Ar Bubbles Behavior at the Solid/Liquid Interface of the Steel: *Sang-min Lee*[1]; Sang-joon Kim[1]; Hae-geon Lee[1]; [1]GIFT POSTECH

In order to clarify the entrapment behavior of bubbles onto the solidifying front of molten steel, the effect of surface tension gradient induced force was investigated. During the solidification of steel, the surface tension gradient due to the concentration gradient is developed in addition to the temperature gradient by the mold cooling. In view of the bubble movement in the vicinity of the solidification front, therefore, the effect of the gradient of solutal surface tension must be taken into consideration together with the thermal gradient effect.The order of magnitude analysis shows that the thermal Marangoni force is comparable to the solutal Marangoni force especially for the larger size of bubbles. The temperature dependency of surface tension(ds/dT) of Fe-S binary system is also considered with varying concentration of sulfur. At the critical concentration the sign of ds/dT is changed and hence the thermal Marangoni effect so generated may act as either pushing or pulling force to the bubble in the vicinity of solid/liquid interface.

11:50 AM

Water Model Experiments for Hydrodynamic Forces Acting on Inclusion Particles in Molten Metal under Turbulent Condition: *Takuya Kato*[1]; Shin-ichi Shimasaki[1]; Shoji Taniguchi[1]; [1]Tohoku University

On the metal manufacturing processes, the inclusion particles in molten metal cause the crucial problems to metal products. In recent years, the inclusion removal processes are improved based on numerical simulations of inclusion particles behavior. However, it sometimes provides an inaccurate result because the hydrodynamic forces acting on particle in turbulent condition, especially lift force, has not been clarified yet. Among various hydrodynamic forces, the drag force always influences largely to particle trajectory. On the other hand, the lift force acts an important role only in high shear rate condition such as wall vicinity. Its prediction model, however, is needed for numerical simulations about inclusion particle behaviors of adhesion to wall and entrapment to interface. Thus, we aimed to evaluate the drag and the lift force acting on small particle in turbulent shear flow by means of water model experiments and numerical simulations.

Magnesium Technology 2010: Coatings and Corrosion

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Sean Agnew, University of Virginia; Eric Nyberg, Pacific Northwest National Laboratory; Wim Sillekens, TNO; Neale Neelameggham, US Magnesium LLC

Tuesday AM Room: 612
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Susan Slade, US Magnesium LLC; Robert C. McCune, Robert C McCune & Associates LLC

8:30 AM

Galvanic Corrosion and Stress Corrosion Cracking of Steel and Aluminum Bolts in Magnesium Die Cast Alloy AZ91: *Gerhard Gerstmayr*[1]; Gregor Mori[2]; Wilfried Eichlseder[1]; [1]Chair of Mechanical Engineering, University of Leoben; [2]University of Leoben

This paper discusses the applicability of selected steel and high-strength aluminum bolts AW 6056 and AW 7075 in magnesium die cast alloy AZ91 regarding corrosion. All aluminum bolts in magnesium parts reveal superior corrosion properties compared to galvanized steel bolts with respect to galvanic corrosion. No aluminum bolt AW 7075 failed in magnesium due to stress corrosion cracking (SCC). Even aluminum bolts in T6 condition can be used for automotive applications when compressive residual stresses introduced by thread rolling are present.

8:50 AM

Characterization of Multilayer Coating Prepared by Combining Plasma Electrolyte Deposition and Electroless Copper and BTA Passivity on Magnesium Alloy: *Yongfeng Jiang*[1]; Yefeng Bao[1]; [1]Hohai University

A novel multilayer coating was prepared to produce pore-free copper coatings on AZ91 magnesium alloy combining the methods of plasma electrolytic oxidation (PEO) pre-treatment, electroless copper plating and benzotriazole(BTA), which are examined using scanning electron microscopy (SEM) equipped with energy dispersive analysis of X-rays (EDX). Electrochemical characterizations methods are employed to evaluate corrosion protection of the coating to substrate in 5% NaCl solution. It is indicated that electroless copper process produces a rough interface between the electroless copper layer and the ceramic layer. And the corrosion potential shifts to the positive direction significantly and the current density decreases by more than one order of magnitude. There is no noticeable galvanic corrosion pits on the surface of the duplex coating combination PEO and electroless copper after 168 h neutral salt spray testing. The color of copper after BTA immersion could be hold above 60 days.

9:10 AM

Enhanced Corrosion Resistance of AZ91 Mg Alloy by Plasma Electrolytic Oxidation with KMnO4: *Dong H. Shin*[1]; In J. Hwang[1]; Ki R. Shin[1]; Kang M. Lee[1]; Bongyoung Yoo[1]; [1]Hanyang University

The plasma electrolytic oxidation(PEO) process in AZ91 alloy was studied with a electrolyte containing potassium permanganate(KMnO4). Potasium permanganate in the electrolyte affected film thickness, surface morphology and microstructure of oxide layers obtained by the PEO process. Oxide layers on AZ91 Mg alloy by the PEO process was confirmed as a mixture of MgO, MgF2 and Mn2O3. The corrosion resistance of this sample was superior to that of the sample processed in the bath without KMnO4 due to the presence of manganese oxide in the oxide layer.

9:30 AM

Laser Surface Alloying of a Creep Resistant Magnesium Alloy MRI 230D with Al and Al2O3: G. Rapheal[1]; *S. Kumar*[1]; C. Blawert[2]; Narendra B. Dahotre[3]; [1]Indian Institute of Science; [2]GKSS Research Centre; [3]The University of Tennessee

A creep resistant magnesium alloy MRI 230D was laser surface alloyed (LSA) with Al and (Al+Al2O3) using Nd:YAG laser at four different scan speeds. The coating comprised of fine dendritic and cellular structure, and interfacial bonding was good. A few solidification cracks reaching down to substrate were observed. The LSA alloy exhibited an improvement in wear resistance due to the presence of ultra-hard Al2O3 particles and increased solid solubility of Al in the coating owing to rapid solidification. The LSA alloy with Al exhibited a poorer corrosion resistance, while a two step coating with Al and (Al+Al2O3) exhibited a slightly better corrosion resistance, than the substrate. This is attributed to the increased coating thickness and closure of solidification cracks in second coating. The corroded surface of the LSA alloy revealed a highly localized corrosion. The laser scan speed did not exhibit a monotonic trend either in wear or corrosion resistance.

9:50 AM

Corrosion Phenomenon Evaluation of Mg Alloys Using Surface Potential Difference Measured by SKPFM: *Rei Takei*[1]; Hiroyuki Fukuda[1]; Hisashi Imai[1]; Junko Umeda[1]; Katsuyoshi Kondoh[1]; [1]Osaka University

One of weak points of magnesium alloys used as the structural materials is lack of corrosion resistance. That is because magnesium is low in standard electrode potential, and results in the galvanic corrosion when contacting the other metals. The electro-chemical evaluation of Mg alloys is often applied to investigate the corrosion phenomena. However, it is essential to clarify the Volta potential at the interface between α-Mg and dispersoids or precipitates in the discussion of the corrosion behavior. This study suggests the quantitative evaluation of the local surface potential difference (SPD) at the interface by using Scanning Kelvin Probe Force Microscope (SKPFM) system. This equipment serves the contact potential difference (CPD) between sample metal and the needle (PtIr5) of cantilever. Furthermore, the effects of additives such as iron (Fe), copper (Cu) and carbon nanotube (CNT) of Mg alloys, strongly causing the corrosion phenomena, on the CPD are discussed in detail.

10:10 AM Break

10:30 AM

A Study of Corrosion Film Growth on Pure Magnesium and a Creep-Resistant Magnesium Alloy in an Automotive Engine Coolant: Zhiming Shi[1]; Pankaj Mallick[1]; *Robert McCune*[2]; S. Simko[3]; F. Naab[4]; [1]University of Michigan-Dearborn; [2]Robert C. McCune and Associates; [3]Ford Research Laboratory, Ford Motor Co.; [4]Ion Beam Laboratory, University of Michigan

In recent years, several creep resistant magnesium alloys have been developed which has created a great interest in using magnesium in automotive engine components, such as the engine block. One major technical concern in using magnesium for engine blocks is its corrosion performance in the presence of engine coolant that flows through its cooling passages. The principal objective of this study was to determine the effects of engine coolant parameters, such as the engine coolant composition, presence of sodium chloride, time and temperature, on the corrosion film growth on pure magnesium and a creep resistant magnesium alloy. The film thickness and composition were determined using both Auger Electron Spectroscopy (AES) and Rutherford Backscattering Spectrometry (RBS). A design of experiments approach was used to determine the main effects of these parameters and their interactions on the corrosion film growth.

10:50 AM

Electroless Nickel Phosphorus Plating on AZ31: *Georges Kipouros*[1]; Khalid Shartal[1]; [1]Dalhousie University

A nickel – boron coating placed on a nickel – phosphorus coating which in turn is placed on a phosphate-permanganate conversion coating layer produced on the magnesium alloy AZ31. This work reports on the determination of optimum kinetic parameters to produce a coherent nickel – phosphorus coating using an electroless procedure phosphate-permanganate conversion coating layer. Measurements of the plating rate as a function of the experimental variables such as the compositions of the plating bath constituents, the temperature and the pH using the weight gain method were implemented and the phosphorus content of the electroless nickel-phosphorus coatings was measured using EDS analysis. The surface morphology of the coating was examined using SEM, and XRD. The deposition rate of electroless nickel-phosphorus coating increases by increasing the deposition temperature, the concentration of free nickel ions, and the concentration of hypophosphite ions in the plating bath.

11:10 AM

Improving Corrosion Performance of AZ31B Mg Alloy Sheet by Surface Polishing: *Guang-Ling Song*[1]; Zhenqing Xu[2]; [1]GM R&D; [2]Meda Limited Engineering and Technical Service

AZ31B Mg alloy sheets in the as-received and polished conditions were compared in terms of their corrosion behavior. It was found that the as-received surface has significantly poorer corrosion performance than the polished surface. It is strongly suggested that a surface cleaning treatment should be conducted for AZ31B parts in production. A preliminary analysis of the improvement by polishing was carried out. Iron contamination of the as-received surface is thought to be responsible for its poor corrosion performance. However, the detailed mechanism of the detrimental effect of the iron-contaminant on corrosion is unclear according to a theoretical analysis of the polarization behavior of steel in the same corrosion environment. It appears that in addition to metallic iron particles, iron oxides and carbonates on the surface could also deteriorate the corrosion performance of the AZ31B sheet.

11:30 AM

Microstructure and Corrosion of AZ91D with Small Amounts of Cerium: *Daniela Zander*[1]; Meredith Heilig[2]; Norbert Hort[3]; Gerald Klaus[4]; Andreas Buehrig-Polaczek[4]; Joachim Gröbner[5]; Rainer Schmid-Fetzer[5]; [1]TU Dortmund; [2]Colorado School of Mines; [3]GKSS Research Centre; [4]Foundry-Institute of RWTH Aachen; [5]TU Clausthal

Recent investigations of magnesium-aluminum alloys revealed a strong influence of mainly four microstructural parameters on corrosion: porosity, volume fraction, distribution and Al-content of Al12Mg17. Further an improved corrosion resistance was observed by the addition of rare earth elements. The influence of cerium on casting skin, inner microstructure and corrosion of sand- and die-cast AZ91D with 0.5-2.0 wt.% cerium was investigated by SEM and TEM before and after potentiodynamic measurements or electrochemical impedance spectroscopy in pH8 and pH11. Cerium significantly improved the corrosion resistance by the formation of a passive layer. A strong influence

of cerium on the average grain size, changes in morphology and/or volume fraction of Al12Mg17 and formation of a cerium-rich intermetallic phase was observed. Therefore, not only the chemical composition but also the change of microstructure influences the local corrosion mechanism and passive layer formation of AZ91D with cerium. This was supported by DFG (Za 426/3-1).

11:50 AM

Effect of Neodymium Addition on Corrosion Resistance of Mg-Li Alloy: *Min Li*[1]; Guang Chun Yao[1]; Yi Han Liu[1]; Hai Bin Ji[1]; [1]Northeastern University

Effect of neodymium addition on corrosion resistance of Mg-Li alloy was investigated using the metallographical observation, scanning electron microscope (SEM),X-ray diffraction (XRD) and polarization curve method. The results indicate that 0.5-2 wt. % neodymium in Mg-Li alloy can significantly reduce the corrosion current density, and Mg-Li-2 wt. % Nd alloy has better corrosion resistant performance, the corrosion current density reached 36μA/cm2, which is lower than that of traditional AZ91D (54.50μA/cm2) obviously. The reason for increasing corrosion resistance is attributed to grain refinement due to the addition of neodymium, which lead to the weak cathodic polarization behavior and the decrease of corrosion current density. Moreover, the denser corrosion film of Mg-Li alloy was formatted due to the addition of neodymium, which can increase the corrosion potential alloy.

Magnesium Technology 2010: Creep, Relaxation, Recovery, and Recrystallization

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Sean Agnew, University of Virginia; Eric Nyberg, Pacific Northwest National Laboratory; Wim Sillekens, TNO; Neale Neelameggham, US Magnesium LLC

Tuesday AM
February 16, 2010
Room: 613
Location: Washington State Convention Center

Session Chairs: Xiaoqin Zeng, Shanghai Jiao Tong University; Eric Nyberg, Pacific Northwest National Laboratory

8:30 AM

Effect of Aluminum Addition on the Strengthening and High Temperature Deformation Behavior of Mg-3Sn-2CaAlloy: *Pitcheswara Kamineni*[1]; Y.V.R.K. Prasad[1]; Norbert Hort[2]; Karl Kainer[2]; [1]City University of Hong Kong; [2]GKSS Research Centre

The hot working behavior of Mg-3Sn-2Ca-0.4Al alloy has been investigated in compression in the temperature range 300–500°C and strain rate range 0.0003–10 s^{-1}, with a view to evaluate the strengthening effect of aluminum. The stress-strain curves exhibited steady state behavior at strain rates lower than 0.01 s^{-1} and at temperatures higher than 350°C and flow softening occurred at higher strain rates. There has been some strengthening due to aluminum addition particularly at lower temperatures compared to that of the base alloy Mg-3Sn-2Ca. Standard kinetic rate equation is obeyed in two different temperature and strain rate regimes and an analysis yielded apparent activation energy values of 175 and 195 kJ/mole in the two regimes. These are higher than that for self-diffusion in magnesium suggesting that the strengthening due to aluminum and the large volume fraction of intermetallic particles CaMgSn present in the matrix generate considerable back stress.

8:50 AM

Atomistic Simulation of Grain Boundary Sliding in Mg during High Temperature Deformation: *Hao Zhang*[1]; [1]University of Alberta

A better understanding of superplasitc deformation mechanisms in wrought magnesium alloys at high temperature will allow us to better control the processes so that material properties can be optimized. In this work, a series of molecular dynamics simulations were performed to study grain boundary sliding of three types of [10-10] tilt grain boundaries in a magnesium bicrystal at elevated temperature. Simulations showed that grain boundary sliding did not occur over the stress range applied; instead, coupled shear motion (grain boundary sliding induced boundary migration) was dominant. While the measured coupling coefficient, the ratio of boundary tangential displacement to boundary normal displacement, was in good agreement with theoretical prediction, the detailed shear behavior was different, depending on types of grain boundary, magnitude of applied shear stress and temperature. It was also noted that grain boundary twining was the predominant mechanism that allowed the coupled shear motion to occur in HCP magnesium.

9:10 AM

Grain Size Effect on the Dome-Forming Limit and Deformation Mechanism of AZ31B Magnesium Alloy Sheets: *HyungLae Kim*[1]; WonKyu Bang[2]; YoungWon Chang[1]; [1]POSTECH; [2]RIST

The deformation behaviour of AZ31 Mg alloy has been studied in relation to temperature. The rolled sheet with 50mm thickness was homogenized at 400°C for 4 hrs. The specimens were then prepared in the directions of the rolling direction (RD). A series of tensile tests was then carried out under the strain rate of 10-2 /s at RT, 100°C, 200°C and 300°C together with the load relaxation tests to obtain flow curves in terms of stress and strain rate. The flow curves were found to consist of the usual GMD (grain matrix deformation) curve, GBS (grain boundary sliding) and an additional term due to friction stress as was prescribed by an internal variable theory. Especially, friction stress term in low homologous temperature and GBS term in high homologous temperature was shown.

9:30 AM

Approaching Bolt Load Retention Behaviour of AS41 through Compliance and Creep Deformation: *Okechukwu Anopuo*[1]; Yuanding Huang[1]; Norbert Hort[1]; Hajo Dieringa[1]; Karl Kainer[1]; [1]GKSS Research Centre

Bolt load retention (BLR) test is a practical way to quantify the degree of fastener clamp load retained at bolted joint in an engineering assembly. This is especially important for automotive power train applications working at elevated temperatures above 100°C. BLR behaviour, creep and microstructure are important to achieve more heat resistant Mg-alloys for automotive power train application. In this study, these methods of investigation were used to analyse the elevated temperature properties of permanent mould cast AS41. Investigations were carried out at temperatures between 100°C and 175°C and stresses of 40-90 MPa. Stress exponent values of ~ 2 at low stress regions of 40 and 55 MPa, and between 4-6 at high stress regions of 70 and 90 MPa suggest grain boundary sliding and dislocation climb. The prediction of the BLR behaviour and experimental results are in good agreement.

9:50 AM

Grain Boundary Sliding Characteristics of Az31 Alloy Sheet: *Yong-Nam Kwon*[1]; [1]Korea Institute of Materials Science

In the present study, commercially rolled AZ31 alloy sheets were used to investigate grain boundary sliding characteristics with temperature. Microstructural observation showed that grain boundary sliding occurs in the case of smaller grained alloy while dislocation slip is dominantly operating for larger grains. Deformation assisted grain growth becomes evident at superplastic temperature of AZ31 alloy. Also, cavitation started at the very low level of strain under superplastic deformation condition. Initial strong basal texture remained after deformation irrespective of temperature. Therefore, it might be concluded that poor accommodation for grain boundary sliding of AZ31 alloy results from a limited slip systems even at the high temperature where grain boundary sliding becomes very active. Also, grain growth and compatibility problems during grain boundary sliding are discussed.

10:10 AM Break

10:30 AM

Elevated Temperature Tensile Behavior of Extruded Magnesium Sheets: *Paul Krajewski*[1]; Adi Ben-Artzy[2]; [1]General Motors; [2]Rotem Industries Ltd

The elevated temperature tensile behavior of AZ31, AM50, ZM21, ZK10, and ZK30 magnesium alloys was investigated using direct extruded sheets. Uniaxial tensile testing to failure and step strain rate testing were performed at 400C and 450C to determine the effect of alloy composition and microstructure on elevated temperature ductility, strain rate sensitivity, cavitation and fracture behavior. Ductilities of almost 300% were observed in the as-extruded material with the ZK alloys giving the highest ductility at temperature. The high ductility of the ZK materials was due, in part, to their ability to maintain a relatively fine grain size throughout tensile testing. The stress exponent, n, for dislocation creep was affected by aluminum content as was the amount of grain boundary sliding. These results will be discussed in the context of designing improved alloys for elevated temperature forming processes.

10:50 AM
Elevated-Temperature Tensile Behavior of a Rapidly Solidified and Reverse Extruded Mg-Zn-Y-Ce-Zr Alloy: *Jon Carter*[1]; Paul Krajewski[1]; Dan Shechtman[2]; [1]General Motors R&D; [2]Technion-Israel Institute of Technology

The elevated-temperature tensile behavior of a rapidly solidified and reverse extruded Mg-6% Zn-4.2%Y-1.2%Ce-0.66%Zr alloy was studied. The material was prepared as plates, from which sheet-like tensile bars were extracted using wire EDM. The grain size was 0.7 microns. Testing was performed at 300C, 400C, and 450C using both constant strain rate (0.001, 0.01, 0.1 s^{-1}) tests to failure, and step strain rate tests. The material showed very high elongations at 400C and 450C, with a maximum of 800%. The peak in ductility occurred at the intermediate strain rate of at both temperatures. The effect of grain size on tensile behavior was investigated by annealing some samples to coarsen the grains to two microns prior to testing, and by testing AZ31 sheet having a grain size of 10 microns.

11:10 AM
Microstructure, Tensile Properties and Creep Resistance of Binary Mg-Rare Earth Alloys: *Mark Gibson*[1]; Suming Zhu[1]; Mark Easton[1]; Jian-Feng Nie[1]; [1]CAST CRC

This paper investigates the microstructure and its relationship to tensile properties and creep resistance of binary Mg-rare earth (RE) alloys. Alloys with 0.5-5wt.% additions of La, Ce or Nd were produced by high pressure die casting (HPDC). The intermetallic phase formed in the eutectic was identified to be Mg12La, Mg12Ce and Mg3Nd in the Mg-La, Mg-Ce and Mg-Nd alloy systems, respectively. In tensile tests, the alloys showed an increase in yield strength but a decrease in elongation with an increase in the addition of RE element. The creep resistance was found to be significantly influenced by the choice and the amount of the RE element, with the Mg-La alloys being the least creep-resistant and the Mg-Nd alloys the most creep-resistant. The tensile properties and creep resistance are analysed based on microstructural observations.

11:30 AM
The Role of Strain on the Recrystallization Behaviour of Hot Worked and Annealed Magnesium Alloy Mg-3Al-1Zn: *Aiden Beer*[1]; [1]Deakin University

The present work examines the microstructure that evolves during the hot deformation and subsequent annealing of magnesium alloy AZ31. In particular, the role of strain on the progression of dynamic recrystallization (DRX) and post-deformation recrystallization is investigated. It is found that for the smallest strain examined, the percentage of DRX is low and that, upon subsequent annealing, post-deformation recrystallization results in a slightly larger grain size. With larger deformation strains, the percentage of DRX is increased and finer annealed grain sizes are obtained. However, when the percentage of DRX is above approximately 40%, strain appears to have no influence on both the kinetics of post-deformation recrystallization and the fully annealed grain size developed. Increasing strain alters the texture of the hot deformed microstructure (for the deformation mode examined) and the texture developed during post-deformation recrystallization is found to be similar to that of the dynamically recrystallised microstructure prior to annealing.

11:50 AM
The Relationships between Grain Boundary Sliding and Grain Orientation in AZ31 Magnesium Alloys at Room Temperature: *Daisuke Ando*[1]; Yuji Sutou[1]; Junichi Koike[1]; [1]Tohoku University

Recently we reported the occurrence of grain boundary sliding (GBS) at room temperature in AZ31 Mg alloy, and proposed that GBS was induced by plastic anisotropy. Since then, additional papers on GBS have been published by others, but the detailed mechanism remains unclear. The purpose of this study is to find a quantitative correlation between GBS and plastic anisotropy. On the chemically polished surface of rolled AZ31 samples, a grid pattern was drawn by a focus ion beam system. The same area was examined by EBSD to obtain crystallographic information. These samples were tensile-tested along the rolling direction at room temperature to a 10% elongation. The deformation of the grid pattern was compared with the expected deformability from the EBSD data. The obtained results provide qualitative understanding of the origin of the GBS at room temperature.

12:10 PM
Texture Change in Pure Mg and Mg-1.5wt%Mn Casting Alloy during Compressive Creep-Deformation: Mert Celikin[1]; D. Sediako[2]; *Mihriban Pekguleryuz*[1]; [1]McGill University; [2]Canadian Neutron Beam Centre, NRC

Most Mg alloys undergo creep under the service conditions of the engine block (50-100MPa, 175-200ºC). Texture studies on creep-tested pure Mg and Mg-1.5wt%Mn casting alloys were performed via Neutron Diffraction (ND) and XRD. Analysis of cast pure Mg, before creep testing, indicated that preferential orientation exists with the HCP prismatic planes normal to the growth direction of columnar grains due to directional solidification. Following compressive creep testing (15MPa, 175ºC, 150hrs.), the maximum intensity of the texture in pure Mg dropped notably, indicating a slight rotation of basal-plane normals. No significant change in the maximum intensity of texture was observed in Mg-1.5%Mn samples creep tested at 15 MPa at temperatures up to 175°C. Creep tests conducted at higher loads (above the yield strength - 50MPa, 150ºC) on both pure Mg and Mg-1.5%Mn samples resulted in the rotation of the basal plane normals towards the compression axis.

Materials in Clean Power Systems V: Clean Coal-, Hydrogen Based-Technologies, Fuel Cells, and Materials for Energy Storage: Materials for Hydrogen Production, Storage, and Distribution

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Conversion and Storage Committee
Program Organizers: Xingbo Liu, West Virginia University; Zhenguo Yang, Pacific Northwest National Lab; K. Weil, Pacific Northwest National Lab; Mike Brady, Oak Ridge National Lab; Jay Whitacre, Carnegie Mellon University; Ayyakkannu Manivannan, National Energy Technology Laboratory; Zi-Kui Liu, Penn State University

Tuesday AM Room: 211
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Jason P. Trembly, Center for Energy Technology, RTI International; Richard Ricker, NIST

8:30 AM Invited
Co-Production of Pure Hydrogen and Electricity from Coal Syngas via the Steam-Iron Process Using Promoted Iron-Based Catalysts Sub-Pilot Plant Based Studies: *Jason P. Trembly*[1]; Brian S. Turk[1]; Raghubir P. Gupta[1]; [1]Center for Energy Technology, RTI International

RTI with funding from the U.S. Department of Energy has successfully developed a novel iron-based catalyst for using the iron redox cycle to produce high purity high pressure hydrogen from syngas. This novel iron-based catalyst was based on nanostructured crystalline phases which allowed significantly higher hydrogen production at significantly lower temperatures compared to the state-of-art iron-based redox systems. A preliminary techno-economic analysis of RTI's steam-iron process using this catalyst for co-production of hydrogen and power in an IGCC plant showed cost benefits over a commercial pressure swing adsorption process for hydrogen production. RTI is continuing the development of this steam-iron process technology focusing on scaling up production of the novel iron-based catalyst and design and fabrication of a circulating dual fluidized bed reactor system. Catalyst and process scale-up activities have been initiated producing catalyst batches up to 100 lbs and fabricating a 50kwth system capable of operating at 550°C.

9:10 AM
High Permeability Ternary Palladium Alloy Membranes with Improved Sulfur and Halide Tolerances: *Kent Coulter*[1]; J. Douglas Way[2]; David Sholl[3]; Bill Pledger[4]; Gokhan Alptekin[5]; [1]Southwest Research Institute; [2]Colorado School of Mines; [3]Georgia Institute of Technology; [4]IdaTech LLC; [5]TDA Research

A three year Department of Energy (DOE) funded project has focused on developing robust, poison-tolerant, hydrogen selective free standing membranes. Extensive DFT calculations have been performed for H in the bulk of Pd, Pd96Ag4, Pd96Au4, Pd96Cu4, Pd96Ni4, Pd96Pt4, Pd96Rh4, Pd96Y4, Pd74Cu26, Pd70Cu30, Pd70Cu26Ag4, Pd70Cu26Au4, Pd70Cu26Ni4,

Pd70Cu26Pt4, and Pd70Cu26Y4. In the last two years, over 50 freestanding foils of binary and ternary Pd alloys have been deposited. Pure gas permeation tests with the binary and ternary alloy membranes have been completed and to date PdCuAu and PdCuPt membranes have exhibited equivalent performance at temperatures in the range of 423-773°C with their performance correlating well with the theoretical predictions. Multiple membranes have been tested under the National Energy Technology Laboratory (NETL) Test Protocol with initial promising results from Au containing foils. In this presentation the modeling, fabrication and performance data for these novel membranes will be presented and the challenges discussed.

9:30 AM

Palladium-Hydrogen Interaction in Dislocations: Trapping and Diffusion: *Hadley Lawler*[1]; Dallas Trinkle[1]; [1]University of Illinois, Urbana-Champaign

Palladium has a high hydrogen solubility, and a high diffusivity due to low binding energy in the bulk. However, experiments have shown that additional binding sites are available in single-crystal Pd with much higher binding energy, effectively storing residual H in the crystal after removal from high pressure H. The storage of H is believed to occur in dislocation cores, which act as nanoscale H traps. Electronic-structure calculations of an isolated Pd dislocation core using flexible boundary conditions, to accurately couple to the long-range elasticity solution, determine the binding energy of H to a dislocation core, the changes in local geometry and electronic structure. Local vibrational modes of H give information about dynamics and compare with neutron scattering measurements; together with energy barrier calculations, H pipe diffusion is compared with bulk diffusivity. These calculations help elucidate the physical ingredients to design more energetically favorable hydrogen storage traps in materials.

9:50 AM

Materials Metrology for a Hydrogen Distribution Infrastructure: *Richard Ricker*[1]; Thomas Siewert[1]; Andrew Slifka[1]; David McColskey[1]; David Pitchure[1]; [1]NIST

Hydrogen is a very promising energy transfer and storage medium. However, hydrogen can be absorbed into and alter the fracture resistance of metals and alloys. An infrastructure for the safe and economical use of hydrogen requires codes and standards for the design and construction of pressure chambers, equipment, and pipelines that will not fail and endanger the public. The ability of these codes and standards to economically protect public safety will be depend on the quality of the available data and the ability of the measurement methods to accurately represent service conditions. This paper will present and discuss the NIST program to meet these needs through the construction of a high-pressure hydrogen gas testing facility and the development and evaluation of testing methods. The prioritization of the research and the coordination with other national metrology institutes and agencies will be discussed.

10:10 AM Break

10:20 AM

Influence of Activation Process on the Hydrogen Storage Properties of Carbon Materials: *Vinay Bhat*[1]; Cristian Contescu[1]; Nidia Gallego[1]; Frederic Baker[1]; [1]Oak Ridge National Lab

Storing large amount of hydrogen near ambient temperature in safe, economic and portable manner is the challenge that poses obstacles in path of commercializing the fuel cell vehicles. Till now, none of the explored materials or technologies meets the requirements set by DoE. Activated carbon materials can adsorb and store large amount of hydrogen, but at cryogenic temperatures. Recent investigations demonstrate that, even at room temperature, a significant amount of hydrogen could be stored on activated carbons if the pores are tuned between 5 to 7 Å. Here, we present how the method of activation influences the hydrogen storage properties of carbon materials. The results support that creation of nano-pores using chemical activation is more effective than physical activation in enhancing the hydrogen storage capacity of carbon materials. In addition, we also explore how catalyst addition further influences the storage capacity and kinetics of activated carbon materials.

10:40 AM

Hydrogen Storage Using Electric Field Enhanced Adsorption: *Jiann-Yang Hwang*[1]; Shangzhao Shi[1]; Xiang Sun[1]; Stephen Hackney[1]; Xuan Li[2]; [1]Michigan Technological University; [2]University of Science and Technology Beijing

Hydrogen adsorption capacity has been low for the effective use of hydrogen fuel cell in automobile applications. Efforts have been attempted by many researchers to develop the material that can meet the target of the Department of Energy. The weak van der Waals force between the hydrogen molecule and the sorbent is a fundamental barrier for high adsorption capacity. In an effort to increase the attractive force between the hydrogen molecule and the sorbent, an electric field was applied. The results are presented in this paper.

11:00 AM

Effect of Hydrogen on the Mechanical Behavior of AISI 4340 and SA372 Steels: Anil Saigal[1]; Junior Aguaze[2]; Gary Leisk[1]; Chris San Marchi[3]; *Douglas Matson*[1]; [1]Tufts University; [2]Boeing Co.; [3]Sandia National Laboratory

This work addresses selection of candidate materials for use as pipelines and storage tanks at distribution systems to support developing the infrastructure necessary to promote sustainable energy management via the hydrogen economy. Air and vacuum-melted 4340 and SA372 Grade J Class 70 steels, which were first copper plated and annealed, were analyzed to study the effects of hydrogen charging on its mechanical properties. Hydrogen charging increases the stiffness of 4340 steels by 3.4 to 7.8% and decreases the stiffness of SA372 alloy steel by 5.2%. Hydrogen charging results in decrease in strength of air-melted 4340 steel, increase in strength of vacuum-melted 4340 steel, and no significant change in the strength of SA372 steel; while it always leads to reduced ductility. Copper-plating has the ability to retain hydrogen concentration within the specimen. Finally, air-melted 4340 steel was found to hold maximum amounts of hydrogen followed by vacuum-melted 4340 and SA372 steels.

11:20 AM

Ni_3Al Foil Catalysts for Hydrogen Production from Methanol: *Ya Xu*[1]; Dong Hyun Chun[2]; Jun Hyuk Jang[3]; Masahiko Demura[1]; Dang Moon Wee[3]; Toshiyuki Hirano[1]; [1]National Institute for Materials Science; [2]Korea Institute of Energy Research; [3]Korea Advanced Institute of Science and Technology

Hydrogen is attracting much attention as a clean and efficient energy source. It is highly necessary to develop efficient, low-cost catalysts for hydrogen production. Ni_3Al intermetallic compound has been known as a promising high-temperature structural material because of its excellent high temperature strength and corrosion/oxidation resistance. Recently, we have successfully fabricated Ni_3Al cold-rolled thin foil. In this study, we investigated the catalytic properties of the Ni_3Al foil for hydrogen production from methanol decomposition in the temperature range of 713-793 K. The Ni_3Al foil showed high catalytic activity and selectivity for methanol decomposition by spontaneous formation of fine Ni particles during the reaction. The results indicate that the Ni_3Al foil can be used both as catalyst and as structural material of microreators for hydrogen production.

11:40 AM

Interfacial Facture Toughness of α-Al2O3(0001)/β-NiAl(110): *Kuiying Chen*[1]; I. Ofzidani[2]; L. Zhao[3]; [1]National Research Council Canada ; [2]University of Ottawa; [3]National Research Council Canada

Interfacial fracture toughness K_{IC}^{int} of thermally growth oxide (TGO) α-Al2O3(0001)/β-NiAl(110) bond coat is one of key parameters in assessing the lifing of thermal barrier coating (TBC) in aerospace gas turbine components. This research reports on a systematic study of interfacial fracture toughness of α-Al2O3(0001)/β-NiAl(110) using a combination of ab initio density functional theory (DFT) calculations with fracture mechanics. The interfacial fracture toughness model considers the contribution of both elastic and plastic deformations in the crack tip energy rate formulate. By means of the calculated surface energies γs, the work of adhesion Wad, Peierls-Nabarro (P-N) barrier energy UP-N and a generalized fault energy γF, the interfacial fracture toughness K_{IC}^{int}has been evaluated. The effect of interfacial embrittlement sulphur and potential interfacial enhancer alloying elements such as Pt and Hf on K_{IC}^{int}has been examined, and a comparison was made on interfacial fracture toughness between the clean and doped interfaces with a purpose to reveal a possible atomic mechanism that may suppress sulphur detrimental effect in terms of electronic structures.

Materials Processing Fundamentals: Process Modeling 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: 601
February 16, 2010 Location: Washington State Convention Center

Session Chair: Prince Anyalebechi, Grand Valley State University

8:30 AM
Finite Element Modeling of the Twin-Roll Strip Casting Process: Application to an AZ31 Magnesium Alloy: *Youliang He*[1]; Elhachmi Essadiqi[1]; [1]Natural Resources Canada

Twin-roll casting is an integrated casting and rolling process that involves the continuous solidification of a melt and the plastic deformation of the solidified metal between two rotating rolls. Computer modeling is a valuable tool to help engineers understand the metallurgical process during twin-roll casting. However, commercially available software can only model either the fluid flow/heat transfer of the melt or the plastic deformation of the solid, but not both. In this study, an object-oriented finite element program was developed to simulate the steady-state fluid flow, solidification and plastic deformation of an AZ31 alloy in a horizontal twin-roll caster. The melt, the mush zone and the solid were treated as a generalized Newtonian flow in an Eulerian frame and different constitutive equations were employed to describe the material behaviours. The effect of operational parameters on the solidification process was discussed based on the simulation results.

8:50 AM
A Thermodynamic Model and Database for Gaseous Species Dissolved in Molten Multicomponent Slags: *Youn-Bae Kang*[1]; Arthur Pelton[1]; [1]Ecole Polytechnique de Montreal

A thermodynamic model has been developed in the framework of the Modified Quasichemical Model in the quadruplet approximation in order to permit the calculation of solubilities of various gaseous species (sulfide, sulfate, nitride, carbide, water, etc.) in molten oxide slags.The model calculates the solubilities solely from existing databases of the thermodynamic activities of the component oxides and the Gibbs energies of the pure components (oxide, sulfide, sulfates etc.).In particular, solubilities of sulfide in the Al2O3-CaO-FeO-Fe2O3-MgO-MnO-Na2O-SiO2-TiO2-Ti2O3 multi-component slag, predicted with no additional adjustable model parameters, were found to be in good agreement with all available experimental data.The model applies at all compositions from pure oxides to pure sulfides, sulfates, etc., and from basic slags to acidic slags. By coupling this database with other evaluated databases for molten metal and gas phases, practically important slag/metal/gas equilibria can be computed such as the S-distribution ratio and gas impurity pick-up levels in molten steel.

9:10 AM
Stress Analysis and Deformation Prediction of a Heavy Hydraulic Turbine Blade Casting in Heat Treatment Process: *Jinwu Kang*[1]; [1]Tsinghua University

The hydraulic turbine blade of the matensitic stainless steel (Cr13%,Ni5%,Mo1%) is a kind of large and curved shape, which is susceptible to deformation in both casting and heat treatment processes. The coupling analysis of heat transfer, phase transformation and stress in heat treatment process of the turbine blade for Three Gorges Project is carried out by using Deform-3D software. Its temperature fields, phase fields and stress fields in heat treatment process are obtained. And its deformation is predicted. In the cooling process of normalizing, the blade exhibits relatively large deformation. Based on the stress analysis and deformation prediction in heat treatment process, an algorithm for the determination of reverse deformation of the blade casting in heat treatment process is presented. And the reverse deformation distribution of blade is obtained.

9:30 AM
Discreate Particle Simulation of Solid Flow in a Blast Furnace: *Hun-je Jung*[1]; Jong-in Park[1]; [1]Inha University

Burden distribution control is an important technology by which iron is efficiently reduced from iron-bearing particles. This paper reports a numerical study of solid flow in blast furnace by discreate element model(DEM). The results demonstrate that the DEM approach show trajectory of the particle and stock line profile without global assumptions. The applicability of the propoesd DEM approach is validated from its good agreement with the experiment in term of solid flow patterns.

9:50 AM
Ultrasound Removing Oxygen Gas Bubbles on Anode and Reducing Cell Voltage during Pb Electrodeposition: *Jilai Xue*[1]; Yifang Zheng[1]; Jiegang Li[1]; [1]University of Science and Technology Beijing

Oxygen gas bubbles generate on anode during Pb electro-deposition process in PbSiF6-H2SiF6 aqueous solutions at room temperature. Cell voltage varies as the bubbles formed on the anodes surface. The bubble layer on the anode surface makes a voltage drop that consumes energy in the Pb process and can decrease by applying ultrasound on the anode. The effects of ultrasound frequency and anodic current density were investigated with ultrasound on and off, alternatively during the Pb electrode-position process. The removing of the gas bubbles were recorded with a drop in the cell voltage. The cell voltage can be lowered by 5% with ultrasound, which demonstrate potential energy saving in Pb production.

10:10 AM Break

10:20 AM
Lorentz Force Velocimetry: Fundamentals and Application: *Christian Karcher*[1]; Yuri Kolesnikov[1]; Vitaly Minchenya[1]; Andre Thess[1]; [1]Ilmenau University of Technology

Lorentz force velocimetry (LFV) is a non-contact measuring technique to determine the flow-rate in high-temperature liquid metals. Physically, this technique exploits the basic principles of magnetofluiddynamcis. Here, by Ohm's law, eddy currents are generated within an electrically conducting fluid that moves through an externally applied magnetic field. The interaction of these currents and the magnetic field induces Lorentz forces in the melt that break the flow. In turn, by Newton's 3rd law, the Lorentz forces exert an accelerating force on the magnet system. These reaction force is proportional to the flow-rate and can be measured precisely by high-performance digital strain gages. At Ilmenau University of Technology we have designed and constructed various prototypes of such Lorentz force flowmeters. Moreover, have tested the prototypes in industrial environment during the production of secondary aluminum. The paper presents the theory of LFV as well as results of the industrial test measurements.

10:40 AM
The Studying of Nonstoichiometric Pyrrhotites Heat Capacity: *Tatyana Chepushtanova*[1]; Vladimir Luganov[1]; Brajenda Mishra[2]; [1]Kazakh National Technical University; [2]CSM

Basing on literary data complicated phase relations in pyrrhotites area of Fe-S system throw with different thin properties of high-temperature solid solution structure. To present day the heat capacity of pyrrhotites from Fe0,885S until Fe0,90S in reference books is absent. In this work represent the results of studying phase transitions at Neel temperature in pyrrhotites, the dependence of heat capacity from temperature at 320 – 303ºC, 600 – 783ºC. Was established that heat capacity of pyrrhotites Fe0,855S – Fe0,888S practically is not depend from structure and equal at 550ºC 73,30 J/mole•ºC, at 740, 750, 783ºC – 71,50 J/mole•ºC.

11:00 AM
Multi-Scale Solidification Model for Laser Engineered Net Shaping (LENS) Process: *Hebi Yin*[1]; Sergio Felicelli[1]; [1]Mississippi State University

A two-dimensional multi-scale solidification model was developed to study the heat transfer, fluid flow, and grain growth occurring in the molten pool during the LENS process. This model coupled the finite element method to calculate mass, energy and momentum transport in the macroscale with the cellular automaton technique to calculate liquid/solid phase change at the microscale. The validation of the model was performed by comparing the simulation results with experimental data from previously published works, showing

good agreement in temperature distribution and the dendrite morphology. The simulation results showed that Marangoni convection played a critical role in determining the flow pattern in the molten pool and exerted influence on the pool size and shape. It was also found that columnar dendrites can be formed, with dendrite arm spacings as fine as a few microns. In addition, the influence of fluid flow on the dendrite growth in the pool was discussed.

11:20 AM

Multitechnique Characterization and Prediction of Phase Diagram Topology: *Marcelle Gaune-Escard*[1]; [1]Ecole Polytechnique, CNRS UMR 6595

Complex phase equilibria in lanthanide halide-alkali metal halide systems are characterized by the existence of several stoichiometric compounds. Their stability depends both on the nature of cations (lanthanide, alkali) and of the halide. A simple scheme for classifying phase diagram topology, with predictive capability, is presented.

11:40 AM

High-Strength Spring Steel: *S.A.J. Forsik*[1]; Sangwoo Choi[2]; P.E.J. Rivera-Diaz-del-Castillo[3]; Sybrand van der Zwaag[1]; [1]Delft University of Technology; [2]POSCO Lab.; [3]University of Cambridge

Latest developments in automobile spring steels demand an ultimate tensile strength (UTS) exceeding 2400 MPa along with an area reduction (AR) in excess of 25 %. This requirement was achieved via austenitisation followed by oil-quenching and tempering at various temperatures (ranging from 250 to 450°C). The present work shows the influence of both heat treatment parameters in achieving ≈ 2450 MPa UTS and ≈ 27 % AR. These properties are achieved via a fine lath martensite microstructure on which vanadium, ε and θ carbides precipitate in various proportions at the tempering stage. These are identified via X-ray and transmission electron microscopy. The experimental evidence suggests ε carbides to be responsible for such properties, as they display dimensions in the range of 30 nm and a spacing lower than 60 nm. Our attempts to model the precipitation sequence so as to further improve the mechanical properties are presented.

12:00 PM

High Temperature Oxidation of Fe-Cu-Sn Alloys for Surface Hot Shortness: *Lan Yin*[1]; Sridhar Seetharaman[1]; [1]Carnegie Mellon University

Steel produced in an electric arc furnace (EAF) contains a high amount of copper that causes a surface cracking phenomenon called surface hot shortness. It is known that tin can exacerbate hot shortness problem but the precise causes are yet to be elucidated. A series of Fe-0.3 wt%Cu-x wt%Sn alloy samples with Sn contents ranging from 0.03 to 0.15 wt% were oxidized in air at 1150 °C for 5 minute and 10 minutes using Thermogravimetry (TG). Scanning electron microscopy (SEM) investigations show that: (i) The oxide/metal interface is planar; (ii) Sn leads severe penetration of liquid-Cu into grain boundaries; (iii) cracks and iron oxides were found beneath the oxide/metal interface. A numerical model was developed to explain the planar interface morphology in the Fe-Cu-Sn ternary system. Focused ion beam (FIB) serial sectioning technique was used to reveal 3-D structure of the penetrated liquid-Cu and cracks beneath the oxide/metal interface.

Mechanical Performance for Current and Next-Generation Nuclear Reactors: Advances in Modeling for Reactor Conditions

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Nuclear Materials Committee

Program Organizers: Dylan Morris, NIST; Greg Oberson, Nuclear Regulatory Commission; Nicholas Barbosa, National Institute of Standards & Tech; Wolfgang Hoffelner, Paul Scherrer Institute

Tuesday AM
February 16, 2010
Room: 201
Location: Washington State Convention Center

Session Chairs: Ioannis Mastorakos, Washington State University; Dylan Morris, NIST

8:30 AM Invited

Atomic-Scale Modeling of the Dislocation - Radiation Obstacle Interactions Responsible for Mechanical Property Changes in Irradiated Metals: *Brian Wirth*[1]; Hyon-Jee Lee[1]; [1]University of California, Berkeley

In the early stages of deformation of irradiated materials, dislocations emitted from grain boundaries and localized regions of stress concentration interact with and destroy radiation-induced defects, creating defect-free channels. Although the presence of channels in a wide range of materials is well documented, the atomistic processes responsible for defect annihilation are not yet well established. In this presentation, we describe our recent atomic-scale molecular dynamics simulation results to investigate the motion and interaction of screw, edge and mixed dislocations in fcc Cu and bcc Fe-Cu and Fe-Cr alloys. The simulations have been performed to investigate the dislocation interaction and detachment mechanisms with a variety of obstacles, including dislocation loops, stacking fault tetrahedron, voids and helium bubbles, as well as coherent precipitates. These observations provide insight into irradiation hardening and defect interaction and annihilation mechanisms, as well as interaction rules for larger- scale modeling, and are compared with available experimental results.

9:00 AM

Multiscale Modeling of Amorphous-Fe and Fe-Ni Systems Used in Extreme Environments such as Nuclear Reactors: *Ioannis Mastorakos*[1]; N. Le[1]; H.M. Zbib[1]; M. Khaleel[2]; [1]Washington State University; [2]Pacific Northwest National Laboratory

The development of fusion as a viable energy source depends on ensuring structural materials integrity.Structural materials in fusion reactors operate in harsh radiation conditions coupled with high levels of hydrogen and helium production,thus experiencing severe degradation of mechanical properties.The development of structural materials for use in such a hostile environment is predicated on understanding the underlying physical mechanisms responsible for microstructural evolution along with corresponding dimensional instabilities and mechanical property changes. The purpose of this work is to study the behavior of a-Fe and Fe-Ni systems under irradiation using both Molecular Dynamics(MD)and Dislocation Dynamics(DD)simulations.The approach is to pass critical information from the atomistic scale(MD)to the microscale(DD)in order to study the behavior of the material at higher scales.In particular, information pertaining the dislocation-defects(such as voids, helium bubbles and prismatic loops)interactions is obtained from MD simulations. Then this information is used by DD to simulate large systems with high dislocation,and defect densities.

9:20 AM

Influence of Hydrostatic Stress on Primary Defect Generation during Displacement Cascade in a-Fe: *Kevin Boyle*[1]; Ronald Miller[2]; [1]CANMET-MTL; [2]Carleton University

Materials for next generation nuclear reactors must be able to withstand more severe operating conditions as compared to current reactors. Advanced steels are being considered for various reactor components due to their thermo-mechanical stability. Although there have been numerous computational studies of primary defect generation in a-iron, few have considered the influence of

stress. The current study investigates the influence of hydrostatic pressure on the statistics of primary displacement cascade defect generation in a-iron using molecular dynamics. Both compressive and tensile hydrostatic pressures (0-1000 MPa) are investigated for a range of primary-recoil energies (1-20 keV). Although hydrostatic pressure is found to have a relatively small influence on defect formation energy, it is found to have a relatively large influence on the total number of defects produced. The results are rationalized in terms of the influence of hydrostatic pressure on defect formation energy and mobility and the molten cascade core.

9:40 AM

Phase-Field Simulation of Void and Fission-Gas Bubble Evolution in Irradiated Polycrystalline Materials: *Paul Millett*[1]; Anter El-Azab[2]; Michael Tonks[1]; Srujan Rokkam[2]; Dieter Wolf[1]; [1]Idaho National Laboratory; [2]Florida State University

The interactive evolution of both polycrystalline microstructure and irradiation-induced defects such as voids and fission gas-filled bubbles in nuclear fuels and structural alloys is complex and critically important to the long-term performance of fission reactors. Here, the phase-field technique is used to model the evolution of multiple point-defect species (vacancies, self-interstitials, and gas atoms), generated randomly in space and time to represent collision cascade events, thus allowing spatially-resolved simulations of void and gas bubble nucleation and growth both within grain interiors and at grain boundary interfaces (which are shown to be heterogeneous nucleation sites). Illustrative results including the formation of void denuded zones and void peak zones adjacent to grain boundaries, the interlinkage of intergranular gas bubbles leading to fission gas release, and the effects of temperature and stress gradients will be presented. This work was supported by the DOE-BES Computational Materials Science Network (CMSN).

10:00 AM

Modeling the Effect of Stress on Defect Migration and Void Formation Using the Phase Field Method: *Michael Tonks*[1]; Anter El-Azab[2]; Paul Millett[1]; Dieter Wolf[1]; [1]Idaho National Laboratory; [2]Florida State University

Microstructural defects play an important role in determining the internal stress in materials. They can also alter the applied stress distribution, e.g. stress concentrations due to voids. The internal stress, in turn, affects the defect dynamic within the material. In this work, we model the internal stresses in irradiated materials, taking into consideration the intrinsic defect stress as well as their interactions with each other and with the applied stress, all within the framework of phase field simulation. We then apply the combined approach to the problem of point defect diffusion and microstructure evolution in irradiated materials. The stress models are first verified against analytical expressions. They are then used to investigate the effect of an applied load on point defect migration and void formation in irradiated materials.

10:20 AM Break

10:35 AM

Modelling Steels Used in Nuclear Energy Applications: Maria Samaras[1]; Maximo Victoria[1]; *Wolfgang Hoffelner*[1]; [1]Paul Scherrer Institute

Materials under the extreme conditions present in nuclear energy facilities are degraded by their exposure to conditions such as high temperatures, irradiation and a corrosive environment. Life-time assessments still follow simple concepts. To go beyond the current state-of-the-art methodologies, an understanding of the materials' physical phenomena on a scale ranging from the microscopic level all the way up to macroscopic effects is necessary. Multiscale modelling is emerging as a complement to experimental work and is intended to enhance and speed up the assessments of the expected life-time of materials. Model validation through experiments needs to be included in modelling schemes to accurately describe materials phenomena and to enable extrapolation to longer time frames. It is hoped that such paths of research will eventually lead to a predictive methodology, which will not only provide lifetime predictions but also tools for alloy development. Such modelling strategies will be presented in this talk.

10:55 AM

Universal Scaling of Work Hardening Parameters in Type 316L(N): *Isaac Edwin*[1]; B. K. Choudhary[2]; [1]Pohang University of Science and Technology; [2]Indira Gandhi Centre for Atomic Research

The tensile flow behaviour of Type 316L(N) austenitic steel had been well described in terms of Hollomon and Ludwigson relations over the wide range of test temperatures and strain rates. The work hardening parameters obtained from the Hollomon and Ludwigson relations yielded interesting inter-relations: all the strength coefficients and the transition stress values exhibited a linear variation with the respective strength exponents and transition strains. The observed linear relation was found to extend over the temperature and strain regions where the flow behaviour was dominated by various micro mechanisms such as cross slip, dynamic strain ageing, recovery etc. The inter-relation exhibited could be correlated with the linear relation reported between tensile strength and uniform strain for the alloy. This suggests that there exists a universal scaling of all the strength parameters and strain parameters with the tensile strength and uniform strain respectively.

11:15 AM

Experimental Analysis and Computational Modeling of Temperature Dependent Cyclic Plastic Hardening and Strain Controlled Ratcheting: *Koen Janssens*[1]; [1]Paul Scherrer Institute

Cyclic temperature shock induced low cycle fatigue can play a role in the safety of the primary cooling system of nuclear reactor power plants. As the yield stress varies with temperature, a thermal shock leads to a sudden local change in the mechanical properties, which may induce unrealistic strain localization and local ratcheting in an elasto-plastic finite element model. We present an experimental analysis of the temperature dependent cyclic hardening of AISI 316L stainless steel, and report on the influence of ratcheting as observed in strain-controlled fatigue experiments. An evaluation of the performance of an implementation of the experimental data into a temperature-dependent, cyclic plastic hardening model is given in the context of a case study of notched ring specimens submitted to cyclic thermal shocks.

11:35 AM

Intergranular Thermal Residual Strain in Rolled and Texture-Free a-Uranium: *Don Brown*[1]; James Wollmershauser[2]; Bjørn Clausen[1]; Thomas Sisneros[1]; [1]Los Alamos National Laboratory; [2]University of Virginia

Uranium is known to undergo pronounced thermal ratcheting due to very anisotropic and strongly temperature dependent coefficients of thermal expansion and elastic stiffness. This intrinsic phenomenon can cause unpredictable mechanical behaviors during thermo-mechanical processing and industry utilization, such as in nuclear reactors, effectively wasting resources and/or endangering process stability or efficiency. In this study, the intergranular thermal residual strains are determined from crystallographic lattice parameters in solid polycrystalline rolled and texture-free a-uranium measured by neutron diffraction during cooling. Elasto-plastic self-consistent modeling serves to assist in the identification of the initiation and degree of activity of plastic deformation, such as slip and twinning, during the cooling process. Temperature dependence of single crystal elastic constants and coefficients of thermal expansion specific to a-uranium are uniquely allowed in the employed model. The measured and predicted internal strains and their relationship with the micro-mechanics of deformation and the development of back stresses are discussed.

11:55 AM

Evolution of the Thermo-Mechanical Response of Nitride and Oxide Nuclear Fuels through Microstructurally Explicit Models: Manuel Parra Garcia[1]; Kirk Wheeler[1]; Kenneth McClellan[2]; *Pedro Peralta*[1]; [1]Arizona State University; [2]Los Alamos National Laboratory

A two-dimensional (2D) thermo-mechanical finite element model of a cylindrical fuel pellet, seen from the longitudinal plane, that includes degradation of its mechanical and thermal properties with temperature and burnup has been used to investigate variability of the thermo-mechanical response (stress and strain fields, grain boundary interactions, temperature distributions, creep and swelling responses) due to microstructure heterogeneity within a Representative Volume Element (RVE). Microstructural information was obtained from sintered ZrN, as a surrogate for (Pu,Zr)N, processed under conditions similar to those used in actinide bearing fuels. The 2-D RVE obtained from microstructural characterization, which includes pore and grain

geometry as well as grain orientation, is surrounded by "effective material" and located at different positions in the model to evaluate the effect of stress, strain, and temperature gradients on local fields. The same techniques are used to analyze UO2 fuels to establish a comparison.Work supported under DOE/NE Agreement # DE-FC07-05ID14654

12:15 PM

First Principles Study of Defects in Uranium.: *Nikolas Antolin*[1]; Oscar Restrepo[1]; John Morral[1]; Wolfgang Windl[1]; [1]Ohio State University

With renewed interest in development of nuclear power, studies of nuclear materials are increasingly important. In particular, previously unstudied properties of uranium metal are relevant in development of metallic fuels, which are being examined by the Global Nuclear Energy Partnership as a potential replacement for oxide fuels. Ab-initio calculations have been successful in providing the necessary input for continuum-level diffusion-reaction models such as parameters and processes to include, and thus are primary candidates to build models for metallic-fuel performance. However, application of ab-initio methods is problematic in modeling the body-centered cubic phase of uranium, because it is mechanically unstable, similar to bcc phases of other metals including Zr and Hf. We use first principles calculations to examine defect energies of uranium while addressing problems of phase instability. Using defect energies, favorable defects in uranium metal can be determined, allowing subsequent calculation of diffusion mechanisms relevant in design of nuclear fuels.

Modeling, Simulation, and Theory of Nanomechanical Materials Behavior: Plasticity and Strength of Nanostructured and Nanoscale Materials III

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: Thomas Buchheit, Sandia National Laboratories; Sergey Medyanik, Washington State Univ.; Douglas Spearot, University of Arkansas; Lawerence Friedman, Penn State University; Edmund Webb, Sandia National Laboratories

Tuesday AM Room: 304
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Thomas Buchheit, Sandia National Laboratories; Sergey Medyanik, Washington State University

8:30 AM

Effects of Vacancies on Dislocation Nucleation in Metals – An Atomistic Simulation Study: *Sergey Medyanik*[1]; Iman Salehinia[1]; [1]WSU

As strength of crystalline materials strongly depends on their ability to nucleate new dislocations or move the existing ones, a proper knowledge of factors that play major role at the first stages of plastic deformation is highly desirable. However, fundamental understanding of the role of point defects, such as vacancies or inclusions, on the onset of plastic deformation in otherwise defect free solids is still lacking. In this work, atomistic simulations are applied to study the role of vacancies in the inception of plasticity in metals. In particular, the effect of one vacancy and its position on dislocation nucleation in nickel during nano-indentation is explored. Furthermore, the effect of the vacancies density on the onset of plastic deformation is also investigated.

9:00 AM

On Homogeneous Nucleation of Dislocation Loops in Nanocrystalline Materials: *Yuri Estrin*[1]; Vincent Lemiale[2]; Rob O'Donnell[2]; Laszlo Toth[3]; [1]Monash University; [2]CSIRO; [3]Universite de Metz

Dislocation glide as a mechanism of plasticity of nanocrystalline materials has been debated for quite some time. While the occurrence of dislocations in nanosized grains has been often questioned, molecular dynamics simulations suggest that dislocations can be generated at a grain boundary, traverse the grain and get absorbed at the opposite grain boundary. Further work has suggested that dislocations can be nucleated within nanograins. In this talk we consider homogeneous nucleation of dislocation loops in individual nanoparticles and also in nanosized grains in a polycrystal. An emphasis is put on the role of image forces, which in this situation act to promote thermally activated nucleation of dislocation loops. On the basis of estimates of the activation energy for this process, it will be shown that image-force assisted homogeneous nucleation is possible in polycrystalline nanomaterials, albeit for a special case of very small grain size.

9:20 AM

Real Space Dislocation Dynamics Model Using the Phase Field Approach: Siu Sin Quek[1]; *Rajeev Ahluwalia*[1]; David Srolovitz[2]; [1]Institute of High Performance Computing Singapore; [2]Yeshiva University

We describe a dislocation dynamics model in an elastically anisotropic crystal using the phase-field approach and solve it in real space so that realistic boundary conditions can be prescribed readily. This model allows us to study size and boundary effects on mechanical properties and serves as a basic framework to couple dislocation plasticity with phase transformation in materials (e.g. spinodal decomposition, martensitic transformation etc.). We apply the model to study the interplay between binary alloy phase separation and dislocation dynamics. The lattice mismatch that results from the phase separation can influence the dynamics of defects like dislocations, while the long range elastic fields induced by the dislocations can in turn affect the phase separation. The coupling between the phase transformation and the dislocation dynamics is simulated in a single phase-field framework and the resulting microstructure evolution in the system is analyzed.

9:40 AM Break

10:00 AM Invited

Dislocation Dynamics Simulations of Thin Film Nanoimprinting: Yunhe Zhang[1]; Erik Van der Giessen[2]; *Lucia Nicola*[1]; [1]Delft University of Technology; [2]University of Groningen

Metal nanoimprinting is of great technological interest due to its current as well as its potential applications in miniaturized systems. The objective of this study is to investigate numerically the capability of metal films to retain imprints when indented by an array of rigid bodies of various shapes, size and spacings. The challenge originates from the size dependent plastic properties at sub-micron size scales, which causes a non-trivial interaction of the plastic zones underneath the indenters. The approach used in this study is 2D discrete dislocation plasticity, where plasticity in the metal film originates from the collective motion of discrete dislocations. The simulations track the evolution of the dislocation structure during loading, unloading and relaxation and provide an detailed descriptionof the metal surface profile and the residual stresses.

10:30 AM

Microstructural Aspects of Material Strength in Small Volumes: *Amine Benzerga*[1]; P. J. Guruprasad[1]; [1]Texas A&M University

There is abundant experimental evidence that the plastic behavior of crystals changes at micro-scales in a way that is not necessarily captured by state-of-the-art models. In this paper, plasticity length scale effects are analyzed by means of direct numerical simulations that resolve the scale of the carriers of plasticity, i.e., the dislocations. A computationally efficient, atomistically informed dislocation dynamics framework which has the capability of reaching high dislocation densities and large strains at moderately low strain rates in finite volumes is recalled. Using this framework, a new type of size effect in the hardening of crystals under compression is discovered. In light of such findings, behavior transitions in the space of meaningful structural parameters, from forest-hardening dominated regime to an exhaustion hardening dominated regime are discussed. Various scalings of the flow stress with crystal size emerge in the simulations, which are compared with experimental data onmicro- and nano-pillars.

10:50 AM

Temporal Statistics in the Framework of Kinetic Theory of Crystal Dislocations: *Jie Deng*[1]; Mamdouh Mohamed[1]; Anter El-Azab[1]; [1]Florida State University

Theoretical modeling and numerical simulation of temporal statistics of dislocation ensembles is presented. A hierarchy of kinetic equations is established to describe the evolution of dislocation density, in which the source terms are governed by the rates of dislocation cross slip, annihilation and junction reactions. Stochastic point process and time series theories are used to model the spatial and temporal dependence of these processes and to model the associated rate terms in the kinetic equation. The statistical properties of these

processes, in both time and frequency domain, are analyzed in conjunction with dislocation dynamics simulations. The moving average technique is applied to remove the trend and keep all the processes stationary. Numerical simulation of the autocorrelation function and spectrum provides the preferred frequencies of different types of processes, which, together with their dependence of dislocation density, provide better understanding of the temporal nature of those processes.

11:10 AM
Multiscale Simulation of Crystals, Defects and Deformation Using the Phase Field Crystal Model: *Zhi Huang*[1]; Jonathan Dantzig[1]; [1]University of Illinois at Urbana-Champaign

Bridging atomic and continuum length and time scales is an important computational challenge. The phase field crystal (PFC) model approaches this problem by minimizing a continuum-based free energy functional to describe processes at the atomic length scale and diffusive time scale. Previous work demonstrated that the combination of an amplitude formulation of the PFC equation with adaptive mesh refinement enables efficient simulation of systems up to micron length scale. However, the amplitude formulation does not represent accurately the behavior near high-angle grain boundaries and other defects. We present a hybrid algorithm that couples the amplitude equations in the crystal bulk to the original PFC equation near defects. A wavelet decomposition scheme is used to couple the two descriptions. Several examples are given to demonstrate the approach, and we also examine the role of deformation in the movement of dislocations and grain boundaries.

Neutron and X-Ray Studies of Advanced Materials III: Diffuse Scattering I

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Titanium Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Jaimie Tiley, Air Force Research Laboratory; Erica Lilleodden, GKSS Research Center; Peter Liaw, University of Tennessee; Yandong Wang, Northeastern University

Tuesday AM Room: 303
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Zahirul Islam, Argonne National Laboratory; Jan Ilavsky, Argonne National Laboratory

8:30 AM Keynote
X-Ray and Neutron Scattering for the Examination of Micro- and Nanostructured Materials: *Gernot Kostorz*[1]; [1]ETH Zurich

Diffraction and scattering experiments, possibly complemented by electron microscopy, are often decisive for the understanding and control of materials properties. X-rays and neutrons have served as probes for the study of microstructures in materials for a long time. The availability and continuous improvement of suitable facilities at neutron and synchrotron radiation sources have been very useful to materials scientists and favored the realization of increasingly sophisticated experiments. Some recent applications of neutron and X-ray scattering – diffraction, diffuse and small-angle scattering – will be discussed where the limits of spatial and temporal resolution of both types of radiation or their unique or complementary properties were fully exploited. With the combination of appropriate tools, the structure, shape and size characteristics of spatial inhomogeneities and their evolution can be studied with great accuracy, in the bulk and at or near surfaces, and even on single objects.

9:00 AM Invited
X-Ray Scattering Investigation of Semiconductor Magnetic Composite Materials: *Vaclav Holy*[1]; Guenther Bauer[2]; Rainer Lechner[2]; [1]Charles University in Prague; [2]J. Kepler University

Semiconductor magnetic composites represent a promising route for fabrication of room-temperature magnetic semiconductors. The magnetic properties of these materials substantially depend on the sizes, positions, and on the atomic structure of magnetic inclusions embedded in a non-magnetic semiconductor matrix. For the characterization of composite GeMn and GaFeN systems we used both x-ray diffraction and x-ray spectroscopy methods. The former class of x-ray methods gives information on the size, and crystallographic nature of the inclusions as well as on elastic strains in the semiconductor matrix around the inclusions. Anomalous x-ray diffraction performed for the photon energies close to the absorption edge of the magnetic ions makes it possible to study the positions of the magnetic ions dissolved in the semiconductor lattice. X-ray spectroscopy methods like EXAFS or DAFS are used for the determination of the magnetic ion positions in magnetic inclusions.

9:20 AM Invited
Depth Dependent Ordering, Two Length Scale Phenomena and Crossover Behavior in a Defective Skin Layer of V_2H: *Kevin Bassler*[1]; Charo Del Genio[1]; Johann Trenkler[2]; Aleksandr Korzhenevskii[3]; Rozaliya Barabash[4]; Simon Moss[1]; [1]University of Houston; [2]Carl Zeiss SMT AG; [3]Institute of Problems of Mechanical Engineering; [4]Oak Ridge National Laboratory

Structural defects in a crystal can alter its ordering behavior. Such defects are responsible for the "two length scale" phenomena in which a sharp central peak is superimposed over a broad peak in critical diffuse X-ray scattering. In a defective skin layer crystal of V_2H, X-ray diffuse scattering shows unusual two length scale phenomena. Defect-free bulk V_2H has a strong first-order structural transition, but the defective skin has a weak first-order transition and a continuous transition with critical properties that crossover from one universality class to another. We explain this with a theory in which order preferentially occurs in cylindrically shaped regions near edge dislocations. As temperature is reduced, the crossover occurs as order spreads from individual cylinders through a network of cylinders until the correlation length in the network diverges. Reducing the temperature further causes a weak first-order transition in the remaining portions of the crystal.

9:40 AM Invited
Using X-Ray Correlation Spectroscopy to Test Dynamical Scaling: *Mark Sutton*[1]; [1]McGill University

Many of the properties of a material depend more on its microstructure than its atomic structure. X-ray photon correlation spectroscopy (XPCS) is an ideal way to study temporal fluctuations of these structures. By extending PCS to x-rays, allows one to study opaque materials and to probe much shorter length scales, as required,for example, by binary alloys. Furthermore, XPCS has been extended to study fluctuations in non-equilibrium systems where it measures a two-time correlation function. This ability to measure two-time correlation functions has been used to study dynamical scaling in first order phase transitions of both conserved and non-conserved order parameter systems. Since XPCS measurements reflect fluctuations of the order around its instantaneous value, IFS results in a new kind of test of the general concept of dynamical scaling. The presented measurements are compared to a theory of dynamical scaling and agree remarkably well with its predictions.

10:00 AM Invited
Pair Distribution Function of Relaxed Se-Clusters inside a Zeolite Structure: *M. Castro-Colin*[1]; T. Baruah[1]; R. Zope[1]; A. Abeykoon[2]; W. Donner[3]; M. Brunelli[4]; S. Moss[2]; A. Jacobson[2]; [1]University of Texas at El Paso; [2]University of Houston; [3]Technische University Darmstadt; [4]ESRF

Zeolites have a rigid alumino-silicate porous structure that present an excellent template for allocation of nanoclusters, and due to their morphologies, provide the means to vary their bulk counterpart properties. Selenium was introduced into the pores of a faujasite, where Sodium was removed by ionic exchange to increase its adsorption capability. Selenium atoms are then constrained to form structures within the nanometer-sized sodalite cages, on the one hand integrating themselves to the zeolite structure and contributing to the Bragg intensities, which can be studied via Rietveld refinement, but on the other hand giving rise to diffuse scattering where the pair-distribution function technique is most useful. To understand the structural origin of the diffuse signal a comparison was made between structures generated using ab-inition molecular dynamics and those produced by further relaxation, through a Monte Carlo procedure. A comparison is made of those results.

10:20 AM Invited

Quantification of Void Network Architectures of Suspension Plasma Sprayed Yttria-Stabilized Zirconia Coatings Using Ultra-Small Angle Scattering (USAXS): *Jan Ilavsky*[1]; Ghislain Montavon[2]; Alain Denoirjean[2]; Stéphane Valette[2]; Pierre Fauchais[2]; [1]APS, Argonne National Laboratory; [2]Université de Limoges

Suspension plasma spraying is novel process to form 20 to 100 µm thick ceramic coatings from a suspension of nanometer-sized (50 nm diameter) feedstock. The resulting unique void (pore) network architecture is challenge to characterize and quantify using most techniques due to small sizes involved. Nevertheless, the discrimination of these pore architectures in terms of size and shape distribution, anisotropy, specific surface area, etc., is critical for the understanding of processing, microstructure, and properties relationships. Number of different microstructures, as deposited and annealed, was studied. Total void content of varied between 13 to 20% with most pores being smaller than 30 nm. Only one-tenth to one-third of voids volume was found to be accessible by intrusion. Pore sizes changed during annealing even at temperatures as low than 850°C. Combination of USAXS and Helium pycnometry, combined with scanning electron microscopy was found to provide sufficient detail for these challenging microstructures.

10:40 AM Invited

Near-Surface and Bulk Microstructure: A Comparative Study for Ni-Pt and Pt-Rh: *Bernd Schoenfeld*[1]; [1]ETH Zurich

Diffuse scattering of alloys offers access to short-range order and, if states of thermal equilibrium are present, to effective pair interaction parameters. Differences in the local atomic arrangement between the bulk and the near-surface microstructure might occur due to relaxation, segregation profile and reconstruction, but they were barely investigated till now in a quantitative way. Here, results of such a comparative study of Ni-23 at.% Pt and Pt-47 at.% Rh solid solutions will be reported, employing synchrotron radiation under grazing incidence for the near-surface microstructure. Differences in local order were found for Pt-Rh between the (111) and (110) surfaces where only the latter surface gave diffuse maxima at 1½0 positions as in the bulk. For Ni-Pt(111), Ni-Pt(110) and the bulk sample, diffuse maxima are always located at the 100 positions of the $L1_2$ superstructure. The presence of the segregation profile is repeatedly seen in the near-surface microstructure.

11:00 AM Invited

Complex Intermetallics – The Decisive Role of Weak Reflections: *Walter Steurer*[1]; Thomas Weber[1]; Miroslav Kobas[2]; [1]ETH Zurich; [2]Dectris Ltd.

Increasing structural complexity is reflected in an increasing amount of weak and very weak diffraction intensities. Without including them in a structure analysis the results would poorly reflect the actual complex ordering phenomena, if at all. The decisive role of weak reflections will be discussed on two examples. The first example deals with the Bragg reflection density as a function of synchrotron radiation exposure time of a high-quality icosahedral quasicrystal in order to see how perfect a quasicrystal can be. In the second example, the crucial role of weak reflections is shown for the structure analysis of the largest known intermetallic structure so far, with more than 23,000 atoms per unit cell. In both cases diffraction data within an intensity range of ten orders of magnitude was collected at the Swiss Light Source (SLS) employing the novel, noise-free, single-photon-counting X-ray pixel detector, PILATUS 6M.

11:20 AM Break

11:30 AM Invited

Mapping Phonon Dispersions and Anomalies with X-Ray Thermal Diffuse Scattering: *Tai Chiang*[1]; Ruqing Xu[1]; Hawoong Hong[2]; [1]University of Illinois; [2]Argonne National Laboratory

X-ray thermal diffusion scattering (TDS) has proven to be a powerful method for studying phonon dispersion relations and phonon anomalies related to phase transitions. The intensity of TDS at a given momentum transfer is determined by the corresponding phonon frequencies and thermal populations. Thus, measurements of the TDS intensity as a function of momentum transfer and temperature under selected polarization configurations provide a means for extracting the phonon properties. In this talk, we will review recent advances in the development of the methodology including two new approaches: one based on calorimetric determination of the phonon frequency, and the other based on direct intensity comparison between TDS and Compton scattering. Results will be presented of recent studies of various prototypical systems including Cu (a simple metal), SrTiO3 (a prototypical complex oxide), Pu (a 5f metal with a complex electronic response), and Cr (a spin density wave system).

11:50 AM Invited

X-Ray Studies of Structural Effects Induced by Pulsed (30 Tesla), High Magnetic Fields at the Advanced Photon Source: *Zahirul Islam*[1]; [1]Argonne National Laboratory

A portable 30 Tesla pulsed-magnet system for materials studies in high magnetic fields is presented. A split-pair magnet (Tohoku design) cooled on a closed-cycle cryostat is used for scattering studies. This system is unique because it uses two closed-cycle cryostats, implemented on a standard diffractometer. Such a scheme allows the sample to be cooled near the liquid helium temperature and the magnet to its operating temperature, independently, in a cryogen-free environment. Pulsed magnetic fields (~ 1 ms in total duration) are generated by discharging a configurable bipolar capacitor bank into the magnet coils. Time-resolved scattering data are typically collected using a multi-channel scaler and an oscilloscope, respectively. Seminal work on structural effects in pulsed fields of a geometrically frustrated system has been performed revealing strong magneto-strictive effects in the spin-liquid state. Use of the APS is supported by the DOE, Office of Science, Contract No. DE-AC02-06CH11357.

12:10 PM

Investigation of the Nanoscale Nial Precipitates in the Ferritic Superalloy by USAXS: *Shenyan Huang*[1]; Xin Li[2]; Gautam Ghosh[3]; Jan Ilavsky[4]; Zhenke Teng[1]; Morris E. Fine[3]; Emily Liu[2]; Peter Liaw[1]; [1]University of Tennessee; [2]Rensselaer Polytechnic Institute; [3]Northwestern University; [4]Argonne National Laboratory

Ultra-small-angle X-ray scattering (USAXS) technique was employed to measure the nanoscale NiAl B2-type precipitates in a ferritic superalloy for elevated temperature applications. Alloys annealed with various aging times and post-creep alloys as a function of applied stress and composition were investigated. A theoretical model on the absolute intensity scale was developed to quantitatively predict the precipitate size distribution, inter-particle spacing, and volume fraction. Complementary TEM characterization was performed to compare and evaluate the microstructural information obtained from the small-angle scattering analysis.

12:20 PM Invited

Studies Phase Evolution of Triblock Copolymer Solutions by Small Angle Neutron and X-Ray Scattering: Effects of Molecular Weight, Temperature, Pressure and Salt: *Lixin Fan*[1]; Liang Guo[2]; Papanan Thiyagarajan[3]; [1]Rigaku Innovative Technologies; [2]Argonne National Laboratory; [3]Office of Basic Energy Sciences, U.S. Department of Energy,

We have investigated the self-assembly and phase behavior of 5 wt% aqueous solutions of triblock copolymers of PEO and PPO, Pluronic F38, F68, F88, F98 and F108, at different temperatures and sodium carbonate concentrations by using small angle neutron and X-ray scattering. The pressure-induced phase transition of 5 wt% F108 was studied and its phase diagram as function of pressure and temperature is obtained. We determined the thermodynamic parameters of micellization by measuring the critical micellization temperature as a function of F108 concentration in the presence of sodium carbonate. We obtained information on the size of the core and corona, inter-micelle distance, aggregation number and the volume fraction of the micelles. The mechanisms of micellization and spherical-to-cylindrical micelle transformation are interpreted to be gradual dehydration of the copolymer chains and progressive insertion of PEO segments from the corona into the core upon the increase in either temperature and/or salt concentration.

12:40 PM

Role of External Stimuli on Phase Transformation in Ferromagnetic Shape-Memory Alloys and Related Properties: *Yandong Wang*[1]; Yang Ren[2]; Zhihua Nie[1]; Gang Wang[3]; Ru Lin Peng[4]; Sten Johanson[4]; Daoyong Cong[5]; Stefen Roth[5]; Tomoyuki Terai[6]; Tomoyuki Kakeshita[6]; Dennis Brown[7]; [1]Beijing Institute of Technology; [2]Argonne National Laboratory; [3]Northeastern University; [4]Linköping University; [5]IFW Dresden; [6]Osaka University; [7]Northern Illinois University

Materials that can reversibly change their dimension upon the application of external fields, such as magnetic or electric fields, have been used as actuators or sensors in many applications. Among them are magnetic-driven shape-memory alloys (SMAs), which can be stimulated by a magnetic field. The possibility of a magnetic-field control of the shape-memory effect has been demonstrated

in the ferromagnetic alloys, such as Ni-Mn-Ga, and antiferromagnetic alloy such as and Ni-Co-Mn-In and CoO. Here we used the synchrotron high-energy X-ray diffraction technique and EBSP method to study the selection of martensite variants with ferromagnetic or antiferromagnetic state under the external (magnetic and stress) fields. Some important progresses on the in-situ investigations of the phase transformation behavior will be presented in many aspects, including the principle of variant selections in both ferromagnetic and antiferromagnetic alloys and the mechanism of 'magnetic field' and 'stress' training in the advanced metallic materials.

Pb-Free Solders and Emerging Interconnect and Packaging Technologies: Reliability (I)

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Kwang-Lung Lin, National Cheng Kung University; Sung Kang, IBM; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Iver Anderson, Iowa State University; Fu Guo, Beijing University of Technology; Thomas Bieler, Michigan State University; Andre Lee, Michigan State University; Rajen Sidhu, Intel Corporation

Tuesday AM Room: 204
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Thomas Bieler, Michigan State University; Fu Guo, Beijing University of Technology

8:30 AM Invited
The Interaction between Imposed Current and Creep of Idealized Snagcu Solder Interconnects: *John Morris*[1]; Christopher Kinney[1]; Xio Linares[1]; Tae-Kyu Lee[2]; [1]University of California - Berkeley; [2]Cisco Systems

The work reported here concerns the effect of an imposed current on the creep of simple SnAgCu interconnects. Both double-shear and multiple-ball samples were tested in shear with and without current, and with various thermal histories. These tests consistently yield two unexpected results. First, the relative increase in creep rate with current is nearly the same over a range of temperatures and variety of starting microstructures. Second, when tests are done at the same temperature (including the effect of Joule heating) the rate of creep is lower under current than under isothermal conditions. These results have a fairly straightforward, quantitative interpretation. Given constant temperature and a microstructure that includes interfacial voids, the current depletes the joint of vacancies, lowering the average creep rate, and introducing observable heterogeneities in the creep pattern.

8:55 AM
Analysis of Simple Shear of Lead-Free Solder Joints to Examine Heterogeneous Strain and Slip System Activity: Bite Zhou[1]; *Thomas Bieler*[1]; Tae-Kyu Lee[2]; Kuo-Chuan Liu[2]; [1]Michigan State University; [2]Cisco Systems, Inc.

Simple shear deformation at a strain rate of about 3x10-6/s was imposed on several 4x4 sections of a plastic ball grid array package having different aging conditions. One side was polished before deformation and characterized using OIM (orientation imaging microscopy) before deformation so that changes in crystal orientation could be measured after deforming to a shear of about 0.5. Strain was concentrated on the side closer to the package having a smaller cross sectional area. Some joints showed a strongly defined shear band where crystal orientations rotated to a softer orientation very close to the package side (often showing topographic features), and others showed a much more diffuse region of deformation. A ranking of slip system facility was identified from analysis of surface topography and changes in crystal orientation, based upon the hypothesis that more easily activated slip systems would lead to a greater amount of homogeneous deformation.

9:10 AM
Bending and Strain/Stress Distribution on Flip Chips Measured by Using Synchrotron X-Ray Laue Microdiffraction: *Kai Chen*[1]; Nobumichi Tamura[1]; Wei Tang[2]; Martin Kunz[1]; King-Ning Tu[2]; [1]LBNL; [2]UCLA

The thermal expansion coefficient mismatch of the Si and the substrate material, such as FR-4, induces residual strain / stress into the flip chips, and thus causes serious reliability issues for semiconductor industry. In this study, the bending of Si chips was measured at both room temperature and enhanced temperature by applying the technique of scanning synchrotron polychromatic X-ray Laue microdiffraction with micro-scale spatial resolution and 0.01 degree angular resolution. The Si chip surface was scanned by dedicatedly focused polychromatic X-ray beam and a two-dimensional Laue pattern was taken by a CCD detector at each sample position. The orientation was analyzed at each spot on the surface of the Si chip, so that the bending angle was measured. The topography of the Si chip was mapped by this technique, and furthermore the strain / stress distribution was calculated based on a simple model.

9:25 AM
Wafer Bonding Using an Amorphous Si-Au Eutectic Structure: *Maryam Abouie*[1]; Qi Liu[1]; Douglas G. Ivey[1]; [1]University of Alberta

MEMS typically involve the integration of mechanical elements, sensors, actuators and electronics on a common silicon substrate, through the utilization of micro-fabrication technologies. Packaging is crucial to the function of MEMS devices, as it is key in determining the cost, size and reliability of the device. To reduce the costs, either the package must provide functionality or its cost must be significantly reduced. Wafer-level packaging using eutectic bonding, which was originally developed for the microelectronics industry, can provide a cost effective alternative for MEMS device packaging. In this work, the Au-Si eutectic system (363°C and 3.16 wt% Si) is investigated, whereby amorphous Si is bonded with electrodeposited Au, in order to produce a more structurally sound bond. To optimize the process, bonding temperature, pressure and duration are altered. Microstructural and mechanical property characterization are done using electron microscopy and X-ray diffraction techniques, as well as shear testing and ultrasonic testing.

9:40 AM
Effect of Pb Addition on Creep and Tensile Behavior of SAC 305 Solder: *Jonathon Tucker*[1]; Carol Handwerker[1]; Ganesh Subbarayan[1]; [1]Purdue University

Comprehensive characterization of the mechanical behavior of Pb-mixed SAC solders is necessary to understand the reliability of reworked legacy components and mixed assemblies. Three alloys of 1, 5 and 20 weight percent Pb were selected so as to represent reasonable ranges of Pb contamination expected from different Sn-Pb components reworked with Sn3.0Ag0.5Cu. Monotonic and creep tests were performed on specially designed assemblies at temperatures of 25°C, 75°C, and 125°C using a double lap shear test setup that ensures a nearly homogeneous state of plastic strain at the joint interface. The observed changes in creep and tensile behavior with Pb addition were related to phase equilibria and microstructure differences observed through DSC and SEM cross section analysis. With increasing Pb content, the steady state creep strain rates increased while primary creep decreased. The experimental data were used to fit for the parameters of the rate-dependent constitutive models.

9:55 AM
Impact Testing of Sn3.0Ag0.5Cu Solder with Ti/Ni(V)/Cu under Bump Metallization after Aging at 150 C: *Kai-Jheng Wang*[1]; Jenq-Gong Duh[1]; Bob Sykes[2]; Dirk Schade[2]; [1]National Tsing Hua University; [2]XYZTEC bv

Non-magnetic Ni(V) metal and low consumption rate with solders are the advantages of sputtered Ti/Ni(V)/Cu under bump metallization (UBM). Sn-patch composed of Sn and V would form in the Ni(V) layer after reflow and aging. In the lead-free solder, Sn-patch was formed and grown more quickly than that in SnPb solder. Thus, the effect of Sn-patch formation on the reliability of solder joint becomes critical. In this study, the Sn3.0Ag0.5Cu solder was reflowed with Ti/Ni(V)/Cu UBM at 250 C for 60 s, and then aged at 150 C for various periods of duration. The high-speed impact test was used to determine the reliability of solder joints. After impact test, the more Sn-patch, the more brittle fracture of solder joint. The correlation between Sn-patch and the reliability of solder joint was discussed and proposed.

10:10 AM Break

10:25 AM

Modeling of Pb-Free BGA Solder Joint Fatigue Life during Random Vibration: *Fengjiang Wang*[1]; Matthew O'Keefe[1]; [1]Missouri University of Science and Technology

Development of a vibration fatigue life model to predict solder joint reliability in Pb-free ball grid array (BGA) electronic packages as a function of components location, the number and size of the solder joints, mechanical clamping positions, and the vibration Power Spectral Density (PSD) during random vibration has been investigated. Assessing solder joint reliability under these conditions is especially important for harsh environments such as space, military and automobile applications. A 3-D global/local finite element modeling technique is used to simulate the random vibration responses of different size BGAs soldered onto a polyimide printed circuit board (PCB) and to determine the stresses/strain of BGA solder joints. A vibration fatigue life model based on the Miner's cumulative damage index (CDI) and the derived solder effective strain is then used to predict the BGA solder joint reliability.

10:40 AM

Improved Reliability of Sn-Ag-Cu-In Solder Joint by the Addition of Trace Elements: A-Mi Yu[1]; Mok-Soon Kim[1]; Jong-Hyun Lee[2]; Jeong-Han Kim[3]; *Jun Ki Kim*[3]; [1]Inha University; [2]Seoul National University of Technology; [3]Korea Institute of Industrial Technology

Among the various Pb-free solder alloys, Sn-3.0Ag-0.5Cu has been an industrial standard in consumer electronics due to its moderate wetting behavior and reliability in thermal fatigue. Recently, however, its high material cost and low reliability in drop impact condition resulted in the use of Sn-0.3Ag-0.7Cu and the development of Sn-1.2Ag-0.7Cu-0.4In solder alloys. Authors have reported that the Sn-1.2Ag-0.7Cu-0.4In showed as good wettability as Sn-3.0Ag-0.5Cu and load drop reliability as Sn-1.0Ag-0.5Cu. It was believed that the small addition of In could make up for the large reduction of Ag with the material cost benefit of about 20%. It was also noteworthy that the load drop reliability of Sn-1.2Ag-0.7Cu-0.4In could be improved beyond Sn-1.0Ag-0.5Cu by the small addition of some elements. In this study, effects of the trace elements, such as Mn and Pd, on the load drop reliability of board-level CSP and failure mechanism in solder joint were investigated.

10:55 AM

Improvement of Heat Dissipation in High-Power Light-Emitting Diodes Using Highly Heat Conductive Die-Attach Material: *Chia-ju Chen*[1]; Chih-ming Chen[1]; [1]National Chung Hsing University

In recent years, applications of high-power light-emitting diodes (HP LED) have constantly increased. HP LED requires high current drives than before and then generates much heat. Only 15~20% of the input power converts to light and the rest converts to heat. The heat generated by LED chip must be dissipated to the environment effectively in order to maintain the thermal stability of the LED devices. In this poster, we proposed a novel composite die-attach material for LED packaging. This composite die-attach material is prepared by adding proper amounts of nanosized diamond particles into commercial Sn-3wt.%Ag-0.5wt.%Cu (SAC305) solder paste. Since diamond is a highly heat conductive material with a excellent heat conductivity of 2300 W/mk, its incorporation into the SAC305 solder paste (~ 20 W/mk) can promote greatly the heat conductive capacity of the die attach materials and thereby dissipates heat more effectively.

11:10 AM

Mechanism of Microstructure Evolution and Fatigue Failure in Lead Free Solder Joint: *Jeong Min Kim*[1]; Woong Ho Bang[1]; Choong-Un Kim[1]; Tae-Kyu Lee[2]; Hongtao Ma[2]; Kuo-Chuan Liu[2]; [1]University of Texas at Arlington; [2]Cisco System Inc.

There have been extensive studies on the microstructural characteristics on the solder joint and their evolution with thermal history. Such studies are motivated by anticipation that fracture reliability is keenly affected by solder joint microstructure, and in fact identifies several potential contributing factors including Sn phase texture, grain size, IMC thickness, its morphology and interface composition. However, the exact mechanism by which such factors become influential to fracture reliability is not well understood. Spurred by this, we have conducted series of experiments that 1) track the microstructural evolution of lead-free solder joint with aging and 2) correlate the result to fracture reliability by conducting cyclic fatigue testing. This paper discusses the major findings of our research and shows 1) fracture resistance and even fracture location changes greatly with aging and 2) such change can only be explained by consideration on changes in mechanical constraints collectively induced by all contributing factors.

11:25 AM

Effect of Joule Heating on Thermo-Electromigration Induced Failure in Lead-Free Solder: *Di Xu*[1]; Luhua Xu[1]; Shih-Wei Liang[2]; Stephen Gee[3]; Luu Nguyen[3]; Marshall Andrews[4]; K.N. Tu[1]; [1]UCLA; [2]National Chiao Tung University, Taiwan; [3]National Semiconductor Corporation; [4]High Density Packaging User Group international, Inc

Electromigration tests have been performed on lead-free solders for wafer level chip scale package(WL-CSP). It has been found that under current stressing, joule heating introduces temperature increase in solders and creates a thermal gradient from chip side to substrate side. Electromigration in the solder joints is thus accompanied by thermo-migration. Statistical failure analysis of the combined effect has been studied by multichannel time to failure (TTF) recording and Weibull distribution. By analyzing TTF data for several different current densities and temperatures, we are able to calculate the activation energy (Ea) and the current density exponent (n) in Black's Mean Time to Failure (MTTF) equation. The effect of joule heating on the increase of temperature has been considered in Black's equation. It is believed that the joule heating plays an important role in void nucleation and growth process thus has a dominant influence on the value of n.

11:40 AM

Effect of Minor Alloying on the Performance of Snagcu Solder Joints under Ball Impact Test: *Yao-Ren Liu*[1]; Jenn-Ming Song[1]; Yi-Shao Lai[2]; Ying-Ta Chiu[2]; [1]National Dong Hwa University, Taiwan; [2]Advanced Semiconductor Engineering, Inc.

In this study, the effect of minor alloying on interfacial reaction products and the ball impact reliability of SAC solder joints was investigated. The solders selected were SAC305, SAC105, SAC105RE and SAC105Mn. The substrates included OSP/Cu and electroplated Ni/Au. There were 3 kinds of fracture modes for the BITed joints. They were the S-mode with ductile fracturing, the I-mode with completely brittle fracturing and the M-mode with the mixed ductile-brittle fracturing. The impact performance for the Ni/Au samples was better than those with Cu for as-mounted joints. For the aged Ni/Au joints, the SAC105RE joints possessed a high S-mode fracturing proportion and thus superior impact toughness under high impact rate. As for aged Cu joints, the performance of the SAC105Mn samples was greater, which may due to the higher thickness ratio of Cu3Sn/Cu6Sn5 at interface. It was demonstrated that Cu3Sn had a better fracture toughness than Cu6Sn5.

11:55 AM

Uncovering the Driving Force for Massive Spalling: *Wei-Ming Chen*[1]; Su-Chun Yang[1]; C. Robert Kao[1]; [1]National Taiwan University

The phenomenon that intermetallic compounds spall massively from the interface during soldering reaction is both technologically important and scientifically interesting. To verify that the driving force for the spalling phenomenon is purely thermodynamic in nature, well designed experiments were carried out. Copper-doped solder was reacted with Ni to form the Sn(Cu) / (Cu,Ni)6Sn5 / Ni structure first; the original Sn(Cu) solder was then removed and replaced with pure Sn. The swapping of Sn(Cu) with Sn caused the massive spalling of (Cu,Ni)6Sn5. A layer of (Ni,Cu)3Sn4 formed at the solder / Ni interface. The results of this study unequivocally prove that the massive spalling here was driven by thermodynamics.

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials IX: Session III

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee
Program Organizers: Chih-ming Chen, National Chung Hsing University; Srinivas Chada, Medtronic; Sinn-wen Chen, National Tsing-Hua University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-ho Lee, Hongik University; Kejun Zeng, Texas Instruments; Yee-wen Yen, National Taiwan University of Science and Technology; Wojciech Gierlotka, AGH University of Science and Technology; Chao-hong Wang, National Chung Cheng University

Tuesday AM Room: 203
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Sinn-wen Chen, National Tsing Hua University; Alexandre Kodentsov, Eindhoven University of Technology

8:30 AM Invited
New Challenges and Solutions for Metal-Semiconductor Contacts: *Suzanne Mohney*[1]; [1]Pennsylvania State University

The requirements for ohmic contacts for many state-of-the-art transistors have become more stringent in recent years due to aggressive scaling of the devices, which places limitations on both contact size (necessitating even lower specific contacts resistances) and process integration. In this presentation, three examples of studies that address these issues are described. First, the development of low-resistance selective ohmic contacts to p-InGaAs using electroless deposition is described. These contacts are suitable for the base of self-aligned heterojunction bipolar transistors. Next, the phase transformations that occur in ohmic contacts for low-power antimonide-based compound semiconductor field effect transistors are described, and the role of interfacial reactions in determining the contact resistance is discussed. Finally, nickel silicide formation in wrap-around-gate silicon nanowire field effect transistors is examined. An intriguing dependence of the identity of the nickel silicide that forms on the original silicon nanowire growth direction is highlighted.

8:55 AM Invited
Reaction Diffusion in GaSb/Co Metallization Contacts during Thermal Processing: *Alexandre Kodentsov*[1]; [1]Eindhoven University of Technology

The utility of thermodynamic potential diagrams in predicting the diffusion zone morphology developed in the GaSb/Co metallization contacts during thermal processing is demonstrated. A number of experiments were designed to test the model. These are aimed at determining phase equilibria in the Ga-Sb-Co system and studying the microstructural evolution of the reaction zone in bulk as well as thin-film diffusion couples. Interfacial reactions between cobalt and single-crystal (001) GaSb have been investigated at 500 C. No ternary phases exist in the system at this temperature. The cubic CoGa and CoSb3 phases were observed to be dominant growing compounds in the semi-infinite bulk as well as in thin-film reaction couples, the latter intermetallic being formed next to the GaSb-substrate. When Co-film is consumed by the reaction, the final configuration of the metallization layer GaSb/CoSb3/CoGa was found. This information is important in designing uniform, stable contacts for the metallization of gallium antimonide.

9:20 AM
Development of Advanced Barrierless Interconnect Using Novel Cu Alloy Seed: *Chon-Hsin Lin*[1]; Jinn P. Chu[2]; C.H. Wu[2]; W.K. Leau[3]; [1]Chin-Min Institute of Technology/Environmental Engineering; [2]National Taiwan University of Science and Technology/Graduate Institute of Materials Science and Technology; [3]National Taiwan Ocean University/Institute of Materials Engineering

Copper metallization can be used to manufacture nanoscale interconnects. Although Cu is widely used as an interconnect material in Si-based devices, it diffuses rapidly into the Si layer, deteriorating the device properties. To prevent device failure, a diffusion barrier must be inserted between Cu and Si. However, these barriers make device miniaturization difficult. Herein, we propose a novel alloy seeding technique for barrier-free Cu metallization. The Cu seed layer—which acts as a barrier that minimizes Cu/Si interdiffusion—was alloyed with small amounts of insoluble substances, e.g., VN and ReN. The seed layer was characterized by X-ray diffraction, focused ion beam microscopy, secondary-ion mass spectroscopy, and transmission electron microscopy, and by film resistivity and current-voltage measurements. The reliability of the seed layer in copper interconnects was assessed by studying the time-dependent dielectric breakdown in MOS structures. The results revealed enhanced thermal stability of the Cu film.

9:40 AM
The Effect of Arsenic Dopant in Nickel Silicide Formation: *S.Y. Tan*[1]; Yi-Lun Hsia[1]; Hsing-Hung Chen[1]; Ming-Yuan Wu[1]; [1]Chinese Culture University

The thermal stability of fully silicided NiSi with arsenic dopant on a different dielectric (SiO2, HfSiO, and HfO2) was investigated. It was found that arsenic-incorporation demonstrated some improvement in both morphology and phase stability of NiSi films at high processing temperatures regardless underlying gate dielectrics. Furthermore, the modulation of the workfunction (WF) of Ni fully silicided gates by arsenic doping is presented, comparing the effects of dopant (As) on the WF for different dielectrics (SiO2, HfSiO, and HfO2) and silicide phases (NiSi and NiSi2). We confirmed also that the work function of NiSi can be tuned by implanting As dopant, which segregate to the silicide/oxide interface.

10:00 AM Break

10:20 AM Invited
Thermal Stability of Advanced Gate Stacks for Microelectronic Devices-the Case of Pt/Gd_2O_3/Si: *Moshe Eizenberg*[1]; Eran Lipp[1]; [1]Technion

In order to realize future metal/oxide/semiconductor devices, the currently used SiO_2 gate oxide and poly-Si gate electrode should be replaced by a high dielectric-constant (high-k) material and a metal electrode, respectively. Gd_2O_3 is a promising high-k material for future devices, offering a sufficiently high k-value and low leakage currents. Pt is considered as a high-workfunction metal for Gd_2O_3-based devices. In this work, the stability of Pt/epitaxial Gd_2O_3/Si stacks is studied after annealing in forming-gas or in vacuum. It is found that stack instability, which is observed after annealing at temperatures above 550°C, is caused by out-diffusion of Gd through Pt grain-boundaries. The out-diffusion kinetics has been quantified and will be reported. Gd diffusion is enhanced and is accompanied by Si out-diffusion and formation of PtSi clusters when the annealing is in vacuum. The better stability in forming-gas is correlated with the content of oxygen in the Pt during the treatment.

10:45 AM
Observations on the Melting of Metallic Nanoparticle Deposites via In-situ Synchrotron Radiation X-Ray Diffraction: Tzu-Hsuan Kao[1]; *Jenn-Ming Song*[2]; In-Gann Chen[1]; Teng-Yuan Dong[3]; Weng-Sing Hwang[1]; [1]National Cheng Kung University, Tainan; [2]National Dong Hwa University, Hualien, Taiwan; [3]National Sun Yat-Sen University, Kaohsiung, Taiwan

Through monitoring the evolution of the Au (111) diffraction peak, the low temperature melting of Au nanoparticles, as well as the transient nano-size induced liquid-solid low temperature alloying behavior, were demonstrated via in-situ synchrotron radiation X-ray diffraction. Upon heating, the broad diffraction peak of nanosized Au particles with the average diameter of 2.5nm was suppressed at around 200°C and soon became sharp due to melting and the following solidification. If the test was performed on a Ni film, an unstable intemetallic compound, Au3Ni, appeared with well crystallized Au, which resulted from the reaction between the supercooled liquid of Au and Ni substrate. However, it decomposed when the heating temperature reached 275°C.

11:05 AM
Control of the Interface Traps in Hf-Based Gate Dielectric Films on Silicon: *S.Y. Tan*[1]; Yi-Lun Hsia[1]; Ming-Yuan Wu[1]; Hsing-Hung Chen[1]; [1]Chinese Culture University

The continuous scaling of the dimensions of CMOS transistors has caused the thickness of the silicon dioxide to decrease below 1.6nm.The replacement oxides must satisfy various requirements as satisfactory gate oxides.(i)thermo dynamically stable in contact with the Si (ii)Oxygen diffusion(iii)form a high quality interface with Si. Hafnium based oxide films have potential to form a silicon oxide comparable interface with the Si.In order to fully understand the origins of the interface trap generation and deep oxide traps in Hf-based films,we introduced two unique process to control the deep trap centers in Hf-based/Si.A

combined approach of (i)Thermal annealing treatment(ii)Different Hf contents in HfO2 (iii)Incorporating N atom into HfO2(iv)Electrical characterization–C-V and J-V,to study the effect of the thermal annealing and Hf contents on the interface charge and oxide charge densities.It is the first time,two unique process techniques are employed to control of the deep interface traps in Hf-based gate dielectric films on Silicon.

11:25 AM

Phase Stability and Phase Transformations, in the Ternary Cd-Sb-Zn: Application to the Growth of a Thermoelectric Material: *Jean Claude Tedenac*[1]; Ya Liu[1]; [1]ICG

High performance thermoelectric materials are obtained by a good knowledge of the systems involved in their fabrication. The phase transformations and phase stabilities of such new materials are still unknown; consequently, a thermodynamic study of these systems is needed. The development of thermodynamic and kinetic databases of such practical materials is important for the microstructural evolution of the materials during processing and service for improving the knowledge. In this paper we will present the results obtained in the study of the ternary Cd-Sb-Zn in a general Calphad procedure. This system contains a well known intermetallic material Zn4Sb3 showing a high conversion factor. Crystal growth of such ternary material is study at the light of a phase diagram analysis. The phase stability of such intermetallic compound is particularly important in this system. It has been studied in the whole range composition.

11:45 AM

Mechanical Properties of (Ni, Cu)3Sn4 Ternary Crystal Structure Using First-Principle Calculation: *Feng Gao*[1]; Jianmin Qu[1]; [1]Northwestern University

In electronic packaging, a ternary (Ni, Cu)3Sn4 intermetallic compound (IMC) is often found at the interface of solder joints due to the Cu present in Sn-Ag-Cu solders. The IMC compromises the solder joint reliability due to the brittle properties if present in excessive amounts. However, data on mechanical properties of (Ni, Cu)3Sn4 IMC are sparse and fall in a broad range. We report the Cu solubility effect on the mechanical properties of Ni3Sn4-based crystal structure using first-principle calculation. Based on the calculated single crystal stiffness, the Young's modulus and Poisson's ratio of polycrystalline are extracted. Moreover, the anisotropic elasticity of (Ni, Cu)3Sn4 is explored by computing the electronic structures, such as band structure and density of states (DOS), when the single crystal structure is subjected to a principle strain along different directions.

Processing Materials for Properties: Advanced Steel Processing

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division
Program Organizers: Brajendra Mishra, Colorado School of Mines; Akio Fuwa, Waseda University; Paritud Bhandhubanyong, National Metal and Materials Technology Center

Tuesday AM Room: 617
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Florian Kongoli, FLOGEN Technologies Inc; Sven Vogel, Los Alamos National Laboratory

8:30 AM Keynote

Control Microstructures in Iron-Based Alloys by Directional Recrystallization: *Z. W. Zhang*[1]; G. Chen[1]; H. Bei[2]; G. L. Chen[3]; C. T. Liu[4]; [1]Nanjing University of Science and Technology; [2]Oak Ridge National Laboratory; [3]USTB; [4]Auburn University

Microstructure with controlled grain structures can improve and/or enhance material properties. Directional recrystallization technique matches the requirement of simultaneously producing directional microstructure and enhancing material properties. For example, Directional recrystallization of an Fe–6.5wt%Si alloy was investigated by changing hot zone temperatures and growth rates. Elongated (columnar) grains with an aspect ratio more than 10 can be produced when growth parameters are carefully adjusted. In this study, we show a kinetic approach to model the growth of the columnar grains for Fe-based alloys. The aspect ratio of the columnar grains predicted by our model is in good agreement with experimental observation. The mechanism of the columnar gain growth essentially can be described as selective growth of grains or competitive migration of the grain boundaries.

9:00 AM

Casting Practice for High-Carbon Nitrogen-Alloyed Chromium-Manganese Austenitic Stainless Steels: Meredith Heilig[1]; *Brajendra Mishra*[1]; Manuel Marya[2]; David Olson[1]; [1]Colorado School of Mines; [2]Schlumberger Reservoir Completion Center

A development regarding carbon additions in nitrogen-alloyed stainless steels presents intriguing possibilities for the future of chromium-manganese based stainless steels. With proper levels of carbon and nitrogen, a cast material can be made that will not form carbides or nitrides, alleviating concern over localized corrosion. The metallurgical requirements to achieve enhanced properties will be considered. Work has been done to economically produce austenitic stainless steel alloys with improved mechanical properties and corrosion resistance. The combination of a series of casting practices is crucial in manufacturing high-carbon nitrogen-alloyed austenitic stainless steel castings with minimal inclusions. The practices to economically produce these castings at atmospheric pressure will be discussed. Alloys of moderate to high nitrogen and carbon concentrations have been produced and examined in comparison to commercially available stainless steels. A review of melting trials, microstructural features, and the results of mechanical testing and corrosion resistance assessment will be presented.

9:20 AM

The State of the Indian Steel Industry: *Sanak Mishra*[1]; [1]Arcelor Mittal India

Year 2007 was one of the most exciting periods in the history of Indian steel industry; the capacity utilization was more than 85%, with a production of 53 MT of finished steel, corresponding to 7% growth over 2006 and the profitability had reached a peak. However, in 2008, due to the unprecedented global economic meltdown, it mustered only a marginal growth of 3.7%. Consumption had declined, in fact, from July 2008 onwards. To de-stock the inventory, integrated steel producers had announced up to 30% production cut from the beginning of November 2008. Most interestingly, though, the year 2009 was a year of great resilience and recovery for the Indian steel industry. The expansion projects bounced back on track and production ramp-up started from as early as March. India moved to third position in the world steel production arena, after China and Japan, during the first six months of the year 2009. Indian steel industry took gainful advantage of the price correction in iron ore, coke and freight and this helped in reducing the per ton cost of production by about 30% from September 2009. The events of the last few months in fact suggest that Indian steel producers were, on the whole, better equipped to deal with market turbulence due to relatively stronger domestic demand, their competitive position in respect of cost of production, primarily on account of higher levels of capacity utilization, and growing competence of human resource working in the industrial sector in India. In this paper a forecast of the Indian steel industry in the coming five years will also be presented.

9:40 AM

Processing and Electrochemical Corrosion Resistance of a Nanocrystalline Fe-20Cr Alloy: Rajeev Gupta[1]; *Raman Singh*[1]; Carl Koch[2]; [1]Monash University; [2]North Carolina State University

It was hypothesised that it may be much easier to develop a protective film on nanocrystalline Fe-Cr alloys. However, successful processing of nanocrystalline alloy samples suitable for oxidation/corrosion testing proved to be a non-trivial task. This paper will present description of the processing of nanocrystalline Fe-Cr alloy powders by mechanical alloying. Powders this produced were successfully compacted and sintered to nearly 100% density while retaining nanocrystalline structure. When oxidation and aqueous corrosion resistance of nanocrystalline and microcrystalline alloys of same composition were compared, the nanocrystalline structure was found to provide in excess of an order of magnitude superior oxidation/corrosion resistance.

10:00 AM Break

10:10 AM

Towards Modelling of Phase Transformation and Mechanical Properties in Hot Rolled Dual Phase Steel: *Piyada Suwanpinij*[1]; Krishnendu Mukherjee[1]; Marcel Graf[1]; Ulrich Prahl[1]; Wolfgang Bleck[1]; Rudolf Kawalla[2]; [1]RWTH Aachen University; [2]Institute for Metal Forming (IMF), Freiberg University of Mining and Technology

The excellent combination of mechanical properties of dual phase (DP) steels arises from the distribution of hard martensite islands in the soft ferrite matrix. Hot rolling is a potential route for commercial production of flat DP steel products in which the present work systematically studies the rolling schedule and run out table cooling strategy. All the processing parameters: rolling temperature and strain, interpass time, temperature control on the run-out-table and the quenching time before coiling were simulated by deformation dilatometer and transferred to a 4-stand pilot rolling mill. The recrystallization behaviour during rolling has been investigated by a hot compression simulator. The morphology, phase fraction, and distribution of ferrite are found to be dependent on the run-out-table cooling condition. The mechanical properties of the sheet were determined by tensile test. The influence of different alloying concepts and the interrelationship between the resulting microstructure and mechanical properties will be investigated.

10:30 AM

A Novel Asymmetric Rolling Method for Controlling Texture of Plates and Sheets: *Dincer Bozkaya*[1]; Peter Jepson[1]; [1]H.C. Starck Inc.

Crystallographic texture of plates and sheets produced by conventional rolling is not uniform through the thickness due to non-uniformity of shear strain distribution induced during rolling. No shear strain is introduced at mid-thickness while shear strain is non-zero elsewhere. Texture non-uniformity through thickness can be eliminated by improving the shear strain distribution. In this paper, a novel asymmetric rolling method, named tilt-rolling, is introduced. Coupling of finite element models (FEM) and crystal plasticity models employed to develop the tilt-rolling process will be explained. Texture results of tantalum plates produced by tilt rolling will be demonstrated. The advantages of tilt-rolling in comparison to other asymmetrical rolling methods such as rolls with different diameters and speeds will be discussed.

10:50 AM

Laser Surface Modification of AISI 410 Stainless Steel with Brass for Enhanced Thermal Properties: *Felix Espana*[1]; Susmita Bose[1]; Amit Bandyopadhyay[1]; [1]Washington State University

Brass coating was applied to AISI 410 stainless steel substrate using high power laser in a Laser Engineered Net Shaping (LENS™) system. Laser deposition resulted in a diffused and sound interface between metallurgically incompatible brass coating and AISI 410 stainless steel substrate. The thermal conductivity of AISI 410 steel increased from 27 W/mK to a maximum of 37 W/mK depending on the coating thickness, almost a 50% gain. The absence of sharp interface between the coating and the substrate, as a result of laser processing, resulted in low interfacial thermal contact resistance. Based on thermal performance tests, the brass coating found to enhance not only the heat transfer by conduction but also the convective heat transfer rate. These results show that novel and efficient feature based coatings can be created by exploiting the capabilities of laser based coating approach and advanced manufacturing technologies for various industrial applications.

11:10 AM

Development of Ferritic Steels with Increased Strength and Ductility: *Semyon Vaynman*[1]; Monica Kapoor[1]; Dieter Isheim[1]; Gautam Ghosh[1]; Morris Fine[1]; Yip-Wah Chung[1]; [1]Northwestern University

A high yield strength of 1600 MPa and elongation-to-fracture up to 25% was achieved in ferritic steels by addition of Cu, Ni, Mn and Al for precipitation strengthening. It was demonstrated that the strength of the steel is proportional to the total amount of these alloying elements; thus, the strength of the steel can be tailored to a specific application by changing the amount of these elements. Atom probe studies demonstrate that two types of coherent slightly misfitting nanosized precipitates are formed in the steel: Cu-rich and NiAl-type. The interaction of these precipitates with the matrix locally lowers the Peierls stress for dislocation motion thus improving dislocation mobility and hence ductility in these steels.

11:30 AM

Effect of Deformation Ratio and Cooling Rate on Mechanical Properties and Microstructure of 0.08wt% C HSLA Steel Microalloyed with Nb and Mo: Taher El-Bitar[1]; *Ahmed Zaky Farahat*[1]; Almosilhy Almosilhy[1]; Ahmed Hegazy[1]; [1]Central Metallurgical Research and Development Institute

A low carbon steel microalloyed with Nb and Mo is thermomechanically processed. The effect of two important parameters; deformation percent and post deformation cooling rate; is studied. It was found that increasing amount of hot deformation decreases strength and increases elongation in C-Mn-Nb-Mo steel till a definite value then increases strength appreciably and decreases elongation with a less amount. This is due to the refinement of grain size. Moreover, water quenched specimen had higher strength and lower ductility than air cooled ones. This is due to the smaller grain size and higher amount of hard phases like acicular ferrite and bainite. However, the effect of cooling rate weakens with increasing amount of deformation. This can be attributed to mechanisms.

11:50 AM

Influence on Non-Metallic Inclusions and Magnetic Properties by Deoxidation Method in Non-Oriented Electrical Steel: *Zhang Feng*[1]; Li Guang-qiang[2]; Chen Xiao[1]; [1]Silicon Steel Department, Baoshan Iron and Steel Co. Ltd; [2]Wuhan University of Science and Technology

The magnetic properties mainly lie on the grain size, crystal texture and non-metallic inclusions in non-oriented electrical steels. Especially the inclusions, their exist will restrain grain growth, accelerate crystal lattice aberrance and block magnetic domain movement. Thus the inclusions in non-oriented electrical steels must be constrained vigorously. In this paper the distribution, variety, shape and size of inclusions and the magnetic properties were investigated under different deoxidation method in two typical non-oriented electrical steel grades. The results showed, there were distinct differences on variety, size, removal speed and magnetic properties under Si-Al and Al-Si deoxidation method, and the magnetic properties will be suffered more salient after the second annealing. The main variety of inclusions were FeO•SiO2, FeO•Al2O3, the complex of FeO•Al2O3 and MnS under Si-Al deoxidation method, and AlN, Al2O3, FeO•SiO2, FeO•Al2O3, the complex of FeO•Al2O3 and MnS, the complex of FeO•Al2O3 and AlN under Al-Si deoxidation method, separately.

Refractory Metals 2010: Oxidation of Alloys and Coatings

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Refractory Metals Committee
Program Organizers: Brian Cockeram, Bechtel-Bettis; Gary Rozak, H.C. Stark

Tuesday AM Room: 2A
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Brian Cockeram, Bechel Marine Propulsion Corporation; Gary Rozak, H. C. Starck, Inc.

8:30 AM

Development of Oxidation Protective Coatings for Molybdenum Alloys: Ridwan Sakidja[1]; Travis Sossaman[1]; *John Perepezko*[1]; [1]University of Wisconsin-Madison

Molybdenum alloys including TZM and Mo-rich Mo-Si-B alloys have great potential for high temperature structural applications due to their high melting point and high temperature strength. The main limitation has been their high temperature oxidation resistance. In previous work Mo disilicide coatings were used for oxidation protection. However, the CTE mismatch between the coatings and Mo alloys and the loss of coating by Si diffusion into the substrate are two main challenges. In the current study, we evaluated the synthesis and performance of two types of Mo-based coatings: borosilicide and aluminosilicide. The borosilicide coating is comprised of a three layer structure: borosilicate, Mo-rich silicide and Mo-rich borosilicide, whereas the aluminosilicide coating is composed of Al-saturated Mo silicide and Mo-aluminide. The new coatings exhibit a significant improvement in the oxidation protection for Mo alloys over the conventional disilicide coatings under various oxidation conditions. The financial support from ONR(N00014-07-1-1083) is greatly appreciated.

8:55 AM
Refractory Metal Alloys for Ultra High Temperature Applications: *Panayiotis Tsakiropoulos*[1]; [1]The University of Sheffield

The search for new materials to replace Ni based superalloys in gas turbine engines has included alloys of refractory metals, in particular Mo and Nb based alloys. Alloys selected from Nb-Si-X-Y (X=sd element(s), Y=sp element(s) or metalloid) systems can provide a good combination of high strength, adequate toughness and creep as well as high melting temperature. This presentation will concentrate on the development of such alloys with emphasis on processing, phase selection and phase stability. Alloys based on Mo-Si-Y (Y as above) systems offer opportunities to exploit multiphase microstructures with good oxidation resistance. This presentation will also concentrate on alloys of the Mo-Si-Al system as a basis for providing environmental protection to refractory metal alloys.

9:20 AM
Microstructures and High Temperature Oxidation Behavior of Mo-Ni-Al Alloys: *Pratik Ray*[1]; Travis Brammer[1]; Mufit Akinc[1]; Matthew Kramer[2]; [1]Iowa State University; [2]Ames Laboratory

Discovery of high temperature materials that go beyond current state-of-the-art Ni based alloys has proven to be an extreme challenge. Based on the body of research available on silicides and Ni based alloys, we attempt to design an alloy that will combine the ultra-high temperature phase stability of the silicides with adequate ductility and oxidation resistance.). The first criterion in the choice of the alloy system is high melting temperature. Additionally, there is the need for an oxidation resistant as well as relatively ductile high temperature phase (which is usually a refractory metal based solid solution). The experimental design is to closely couple the semi-empirical methodologies to ab initio methods to assess the best candidate for high temperature alloy design. The architectural framework for our material's design is a refractory base metal with a high temperature intermetallic which provides both high temperature rigidity and a source of oxidatively stable elements.

9:45 AM
Comparison of the Oxidation Behavior of Nb-20Mo-15Si-25Cr and Nb-20Mo-15Si-25Cr-5B Alloys from 700 to 1300°C: *Benedict Portillo*[1]; Shailendra Varma[1]; [1]The University of Texas at El Paso

Nb-20Mo-15Si-25Cr and Nb-20Mo-15Si-25Cr-5B alloys have been subjected to oxidation in air from 700 to 1300°C. Weight gain per unit area as a function of time has been used for the estimation of the oxidation resistance of these two alloys. There is a noticeable improvement in the resistance for the alloys containing B. Both alloys can withstand the oxidation for 24 hours up to 1300°C. However, alloys without B are susceptible to extensive pesting at 900°C. As cast structure in both alloys contains Laves, Nb based solid solution and silicide phases. Their relative proportions influence the oxidation resistance in the range of temperature used in this study. Results from EDS, XRD and TGA characterization will be discussed.

10:10 AM Break

10:25 AM
Effect of Al on the Oxidation Behavior of Nb-Si-Cr Alloys in Air from 700 to 1300°C: Clemente Parga[1]; *David Alvarez*[1]; Shailendra Varma[1]; [1]The University of Texas at El Paso

Nb-10Si-20Cr-5Al, Nb-10Si-30Cr-5Al, Nb-30Si-10Cr-5Al, and Nb-30Si-20Cr-5Al alloys (compositions are in atomic percent) have been subjected to oxidation in air from 700 to 1300°C. Oxidation resistance of these alloys has been monitored using weight gain per unit area as a function of time isothermally. Static heating for 24 hours and cyclic heating of 4 and 24 hours of cycles up to 24 and 168 hours, respectively, at a given temperature has been used to compare the oxidation characteristics. The observed microconstituents have been compared with those predicted by the isotherms calculated by PandatTM software program. The oxide scale identification has been performed by EDS in SEM and BSE imaging, and XRD. The effect of Al on the oxidation behavior can then be determined by comparing the results with the reported work from earlier studies.

10:50 AM
General Chemical Solution Deposition to Epitaxial Growth of Transition Metal (Ti, Nb, V, Ta, etc.) Carbide Films: *Guifu Zou*[1]; Haiyan Wang[2]; Nathan A. Mara[1]; Quanxi Jia[1]; [1]Los Alamos National Laboratory; [2]Texas A&M University

Transition metal carbides possess a number of useful properties including exceptional hardness, high-temperature stability, low electrical resistivity, and corrosion and oxidation resistance. Due to high performance, their films/coatings are widely interesting for materials scientists. This work shows the epitaxial growth of some metal carbide films on the sapphire substrates by general chemical solution deposition. Both X-ray diffraction analyses and HRTEM images shows the epitaxial growth of carbides films with high quality. The electron transports of TiC and NbC films have the properties of semiconductor and superconductivity, respectively. The transition temperature of NbC is close to 10.5 K. Furthermore, these carbides films show strong hardness in the nanomechanical tests. The hardness and Young's modulus of TiC are achieved to ~ 23 and 420 GPa, respectively.

Solid-State Interfaces: Toward an Atomistic-Scale Understanding of Structure, Properties, and Behavior through Theory and Experiment: Mechanical Properties and Interaction with Dislocations

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Michael Demkowicz, Massachusetts Institute of Technology; Douglas Medlin, Sandia National Laboratories; Emmanuelle Marquis, University of Oxford

Tuesday AM Room: 602
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Sylvie Aubry, Stanford University; Kedarnath Kolluri, Massachusetts Institute of Technology

8:30 AM Invited
TEM Analysis of the Structure and Deformation Behavior of an Incommensurate Grain Boundary in Gold: *Ulrich Dahmen*[1]; Jia Ye[1]; Andy Minor[1]; Tamara Radetic[1]; Damien Caliste[2]; Frederic Lancon[2]; [1]NCEM; [2]CEA

The atomic structure of an incommensurate grain boundary in gold has been characterized by high resolution electron microscopy and compared with atomistic simulations. Local relaxation of the boundary near the surface leads to a chevron-like defect of whose size and stability is related to the stacking fault energy. Simulations predict that such local relaxations play a dominant role during deformation of finite-sized bicrystals because the surfaces act as defects in the incommensurate structure of the infinite boundary, which is expected to deform by superglide. We have tested these predictions by compressing bicrystalline nanopillars of gold using a nanoindenter inside an electron microscope. A detailed analysis of the observed structure and deformation behavior is in good agreement with atomistic simulations.

9:00 AM
Coupling between Grain Boundary Sliding and Migration: Analysis of Possible Mechanisms: *Askar Sheikh-Ali*[1]; [1]Kazakh-British Technical University

The coupling between grain boundary sliding and migration has been established for boundaries with coincidence misorientations in metals and ceramics. However, the exact mechanism of this process remains unclear. In the present investigation the behavior coincidence and near-coincidence symmetric tilt boundaries in zinc bicrystals subjected to high-temperature deformation have been studied. Sliding-migration ratio and direction of boundary migration have been determined. The obtained results are analyzed in terms of two possible mechanisms: the glide of extrinsic DSC dislocations and motion of secondary grain boundary dislocations.

9:20 AM

Structure and Hardness of V/Ag Multi-Layers: *Qiangmin Wei*[1]; Amit Misra[1]; [1]Los Alamos National Lab

Microstructure and hardness of polycrytalline V/Ag multilayers with different individual thickness ranging from 1nm to 50 nm were investigated. It was found that, with decreasing individual layer thickness, interface structure changes from incoherent to semicoherent, companied by the decrease of defect density. For individual layer thickness less than 5 nm, although the interface exhibits considerable variation, close packed planes of face centered cubic (FCC) and body centered cubic (BCC) connect each other at the interface and as a consequence Bain orientation was generated. When layer thickness is 1 nm, semicoherent interface was observed in which most of Ag becomes BCC structure. The role of interfaces and layer thickness on the structure and hardness of V/Ag multilayers is discussed. This research is funded by the US DOE, Office of Basic Energy Sciences.

9:40 AM

Atomic-Scale Study of Nanoindentation in FCC Crystal with Internal Interface: *Yury Osetskiy*[1]; Anna Serra[2]; Roger Stoller[1]; [1]ORNL; [2]UPC

We present results of an extensive molecular dynamics study of material deformation during the nanoindentation process of an fcc crystal containing an internal interface. As a first example, we have used Al and Cu and elementary twin boundary in the ½{111} plane. The indentation process was simulated by spherical or cylindrical intenders with diameter up to 40 nm moving at a constant speed of 10 or 2 m/s.Features of plastic deformation made by spherical and cylindrical indenters, formation of different glissile microstructures and their interactions with twin boundary were studied and the effects of indenter type, size and rate as well as material, i.e. low stacking fault energy Cu versus high stacking fault energy Al, were observed and will be discussed.

10:00 AM

Simulations of Dislocation Pile-up at Asymmetric Tilt Boundary in Aluminum: *Steven Valone*[1]; Timothy Germann[1]; Richard Hoagland[1]; Authur Voter[1]; Danny Perez[1]; Zhiqiang Wang[1]; [1]Los Alamos National Laboratory

Problems in materials deformation processes are becoming approachable for the first time through the largest available computers that implement both conventional and accelerated molecular dynamics. In one deformation process, dislocation pile-up at a grain boundary, a greater understanding is required as to how dislocations are either transmitted through grain boundaries, cause plastic deformation in an adjoining grain, or cause the grain boundary to fail. Here dislocation pile-ups in an aluminum bicrystal with an asymmetric tilt grain boundary are simulated at several levels of resolution of the pile-up, gradually introducing effects of dislocation interactions beyond linear elastic ones. The observed responses as functions of the number of explicitly modeled dislocations and the magnitude of the applied stress are discussed. Longer-time responses are examined more fully through accelerated molecular dynamics simulations, thereby allowing more realistic applied stresses, strain rates, and mechanisms to be modeled.

10:20 AM Break

10:40 AM Invited

Ductility, Interfacial Shear, and Fracture of Cu/Nb Nanolayered Composites: *Nathan Mara*[1]; Dhriti Bhattacharyya[1]; Pat Dickerson[1]; Richard Hoagland[1]; Amit Misra[1]; [1]Los Alamos National Laboratory

Cu/Nb nanoscale multilayered composites have shown ultra-high strength as well as high ductility in a variety of mechanical test methods (nanoindentation, tensile testing, and micropillar compression). Individual layer thicknesses tested range from 100 nm to 5 nm, with flow stresses of nearly 3 GPa (5 nm Cu/Nb case), and deformation in excess of 20% during micropillar compression. It is found that the relatively low Cu/Nb interfacial shear strength leads to localization of deformation at the interface when loaded directly in shear. When the material is loaded perpendicular to the interface, homogeneous deformation of over 10% true strain is evident at individual layer thicknesses as low as 5 nm, followed by shear band formation. TEM evaluation of the microstructure within the shear band exhibits large plastic deformation and grain rotation relative to the compression axis, and the layered structure remains continuous even after local strains in excess of 70%.

11:10 AM

Computer Simulation of Boundary – Dislocation and Boundary – Loop Interactions in the {10-12} Twin in Alpha-Zirconium: *Anna Serra*[1]; David Bacon[2]; [1]Technical University of Catalonia; [2]The University of Liverpool

The development of a method that allows an interface containing a disconnection line to be simulated with periodic boundary conditions in the interface plane has allowed to investigate the interaction of a moving {10-12} twin boundary with two types of crystal defect, namely, a straight 1/3<11-20> (0001) dislocation lying perpendicular to the direction of the twinning shear and a periodic row of perfect dislocation loops. Boundary reactions with crystal dislocations are likely to be important for assisting the twinning process by providing a simple mechanism for twin growth/shrinkage. The boundary attracts both vacancy and interstitial dislocation loops with inclined Burgers vector but is not transparent to them as the complete loop is swept along its glide prism by the moving interface. The results indicate that twinning is efficient at sweeping loops from the microstructure when their density is low and is suppressed by loops when their density is high.

11:30 AM

Flexible Boundary Condition Methods for Interfaces: Dislocation/Twin-Boundary Interactions: *Maryam Ghazisaeidi*[1]; Dallas Trinkle[1]; [1]University of Illinois at Urbana-Champaign

Flexible boundary condition methods couple an isolated defect to bulk through the bulk lattice Green's function. Application of such methods to materials involving an interface requires the computation of interfacial lattice Green's functions. We present a method to compute the lattice Green's function for a planar interface with arbitrary interactions. The interface is coupled to two different semi-infinite bulk regions, and the Green's function for interface-interface, bulk-interface and bulk-bulk interactions are computed individually. The elastic bicrystal Green function and the bulk lattice Green function give the interaction between bulk regions. Direct inversion of the force constant matrix using a partial Fourier transform to account for translational invariance provides the interface terms. The general method makes no assumptions about the atomic interactions or crystal orientations. We simulate a screw dislocation interacting with a (10-12) twin boundary in Ti using flexible boundary conditions, and compare with previous results.

11:50 AM

The Behavior of Σ 11, <110> {252}{414} Grain Boundary in Aluminum under Shock Loading by Molecular Dynamics Simulations: Chiara Pozzi[1]; Timothy Germann[2]; *Donato Firrao*[1]; Richard Hoagland[2]; [1]Politecnico di Torino; [2]Los Alamos National Laboratory

Metastable configurations can be obtained for the Σ 11<110>{252}{414} asymmetric grain boundary (GB) in aluminum by varying the reciprocal positions of the two grains before relaxing the initial structure. Molecular Dynamics (MD) simulations were performed in order to study the behavior of some of these GB configurations under shock loading. For different shock strengths and EAM potentials, observed plastic deformation modes include the emission of perfect and partial dislocations from the GB and, in some cases, the formation of nanotwins. These features and their nucleation mechanisms, as related to the shock strength and direction, the GB arrangement, the size of the model, and the material properties (via comparison of different EAM potentials) will be discussed on the basis of our MD simulation results.

12:10 PM

Microstructural Stability and Plastic Deformation in Nanocrystalline Copper Doped with Antimony: Experiments and Molecular Dynamics Simulations: *Douglas Spearot*[1]; Rahul Rajgarhia[1]; Ashok Saxena[1]; [1]University of Arkansas

Experimental and simulation results have provided a wealth of information on the behavior of pure metallic materials with nanocrystalline microstructures. However, the influence of alloying elements at the grain boundaries in nanocrystalline materials is still unknown. In this work, the microstructural stability and plastic deformation of a nanocrystalline Cu-Sb alloy is evaluated via experiments and molecular dynamics (MD) simulations. MD simulations are used to study grain boundary stability and dislocation activity in nanocrystalline Cu with antimony (0.0-2.0 at.%Sb) at the grain boundaries. Microhardness and microtensile test experiments are performed on nanocrystalline Cu with 0.2 and 0.5 at.%Sb located predominantly at the grain boundaries. Results show that small concentrations of Sb can retard grain growth mechanisms, raising the

potential service temperature of nanocrystalline Cu. MD simulations also show that small concentrations of Sb at the grain boundaries increase the flow stress of nanocrystalline Cu, in agreement with the experimental results.

Surface Engineering for Amorphous-, Nanocrystalline-, and Bio-Materials: Session III

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Surface Engineering Committee

Program Organizers: Sandip Harimkar, Oklahoma State University; Arvind Agarwal, Florida International Univesity; Sudipta Seal, University of Central Florida; Narendra Dahotre, University of Tennessee

Tuesday AM Room: 604
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Indranil Manna, Indian Institute of Technology; Ramki Kalyanaraman, University of Tennessee

8:30 AM Introductory Comments

8:35 AM Invited

Materials Research in the Materials and Surface Engineering Program at the National Science Foundation: *Clark Cooper*[1]; [1]National Science Foundation

Materials research is supported by several programs at the National Science Foundation (NSF), including the Materials and Surface Engineering (MSE) program. While submissions to NSF programs typically focus on the advancement of fundamental science, most proposals identify application areas that are expected to be underpinned through successful execution of the proposed research. Examples of increasingly important application areas for proposals submitted to myriad programs at NSF, including MSE, are (1) energy and sustainability and (2) life sciences and biomedicine. This presentation will provide an overview of the MSE program, including funding opportunities and the identification of program foci, and will highlight exciting recent advances that have been achieved through NSF research grants from this program. Also to be underscored in this presentation is the importance of modeling and simulation, experimentation, theory development, and their interplay to advance frontiers in the fundamental understanding of materials.

9:00 AM Invited

Glass-Forming Metallic Films for Enhancing Mechanical Property of Structural Materials: Cheng-min Lee[1]; *Jinn P. Chu*[1]; Peter K. Liaw[2]; T. G. Nieh[2]; [1]Nation Taiwan University of Science and Technology; [2]The University of Tennessee

Metallic glass-forming materials show desirable properties, including good surface finishes and high strength in the thin film form, thus making them ideal candidates for coating materials to improve mechanical properties, especially fatigue properties of structural materials. Zr- and Cu-based glass-forming metallic films were deposited on stainless steels, using magnetron sputtering. Four-point-bending fatigue tests were conducted on these coated materials. The fatigue life and the fatigue-endurance limit were considerably improved, depending on the different films and the maximum applied stresses. Fractographic studies indicated the good adhesion between the glass-forming film and the substrate. The surface-roughness measurements showed the improved surface conditions due to the film deposition. Analyses showed that the high strength and the moderate bending ductility of the glass-forming metallic films might play beneficial roles, revealing the deposition of glass-forming metallic films on structural materials as a potentially novel and effective method to enhance fatigue properties of structural materials.

9:25 AM Invited

Surface and Bulk Nanostructures for Optical Absorption Enhancement in Thin Si Films: Ritesh Sachan[1]; J. Strader[1]; A.W. Paradies[1]; W. Yueying[1]; H. Uk[1]; H. Garcia[2]; P.D. Rack[1]; G. Duscher[1]; *R. Kalyanaraman*[1]; [1]University of Tennessee-Knoxville; [2]Southern Illinois University

Crystalline thin film Si is considered as an important photovoltaic material for sustainable solar energy harvesting applications. However, Si has an inherent limitation due to its poor light absorption in the visible wavelengths. In the present study, this drawback is addressed by utilizing optical materials design models to guide the preparation of surface or embedded metallic nanostructrues with Si thin films. Various metals and silicides have been prepared by sputtering, pulsed laser deposition and laser-induced self-organization routes. The resulting structure, morphology and interfaces have been investigated using electron and scanning probe microscopies and EELS analysis in TEM. Optical and electrical studies of various structures integrated with Si show promise for enhancing these functional characteristics. From such studies, we expect to identify the composition and nanoscale morphologies that could be relevant towards increasing the absorption of visible light in thin film solar devices.

9:50 AM

Formation of Amorphous Metallic Coatings by the LENS™ Process: *Hongqing Sun*[1]; Katharine Flores[1]; [1]The Ohio State University

Direct laser deposition is a useful technique to create coatings and large-scale components with amorphous or uniquely tailored, non-equilibrium microstructures. We use the Laser Engineered Net Shaping (LENS™) process to deposit Zr-based metallic glass forming powders on both amorphous and crystalline substrates. In both cases, amorphous melt zones are observed surrounded by crystalline heat-affected zones (HAZs). The microstructures of the deposited layers and underlying substrates were characterized as functions of the processing parameters. Optimization of the heat input results in the formation of a continuous amorphous layer without crystallization in the HAZ. However, multilayer deposition results in devitrification of previously deposited material. To better describe the thermal history of the glassy melt zone and underlying substrate material, finite element modeling analysis was performed and compared to in-situ thermal imaging measurements. The crystalline HAZ is found to occur only in regions reaching temperatures more than 100 K above the crystallization temperature.

10:10 AM Invited

Fe-Cr-Mo-Y-B-C Bulk Metallic Glass Coating on AISI 4340 Steel by Laser Surface Cladding: *Indranil Manna*[1]; S. Harimkar[2]; Jyotsna Dutta Majumdar[1]; Manoj Debnath[1]; N. Dahotre[3]; [1]Indian Institute of Technology Kharagpur; [2]Oklahoma State University; [3]University of Tennessee

In the present study, Fe48Cr15Mo14Y2C15B6 bulk metallic glass has been laser surface cladded on AISI 4340 steel substrate to develop an amorphous coating. Due to solute redistribution within the clad zone and across the clad-substrate interface, complete amorphous surface microstructure has not been observed. The incident laser power and interaction time controls the thickness of coating. The thickness of the clad layer was found to increase with increase in applied power and decreases with increasing scan speed.XRD profiles shows α-Fe, iron carbide (Fe7C3), chromium carbide (Cr7C3), iron boride (Fe2B), Molybdenum boride (Mo2B) and yttrium boride (YB12) phases. The microhardness of the clad layer is significantly improved (to as high as1100 VHN) as compared to the substrate hardness (240 VHN). Laser surface cladding has also improved wear resistance. The microhardness and wear resistance properties of the clad zone were found to vary with laser parameters and depth.

10:35 AM Break

10:50 AM

Spark Plasma Sintering of Amorphous Coatings on Metallic Substrate: *Ashish Singh*[1]; Sandip Harimkar[1]; [1]Oklahoma State University

There is increasing commercial and scientific interest in developing metallic glass coatings due to their enhanced mechanical properties. However, deposition of dense and amorphous coating is still a challenge by using conventional thermal processing methods like thermal spray coatings Spark plasma sintering is an emerging technology and is used to compact nano ceramics, bulk metallic glass, and functionally graded materials at comparatively low temperature and pressure. In the present work, we will discuss the results of deposition of amorphous coatings on metallic substrates using spark plasma sintering method. The influence of spark plasma sintering processing parameters on the degree of coating amorphosity, coating density, and strength of coating/substrate interface will be discussed.

11:10 AM
Surface Amorphization in "Chromium-on-Silicon" System Resulted by Compression Plasma Action: *Vladimir Uglov*[1]; Nikolai Kvasov[2]; Yury Petukhou[2]; Valiantsin Astashynski[3]; Anton Kuzmitski[3]; [1]Belarusian State University; [2]Belarusian State University of Informatics and Radioelectronics; [3]B.I. Stepanov Institute of Physics, National Academy of Sciences of Belarus

The results of studies of pre-surface layers structure, phase and elemental composition of plasma-intermixed "chromium-on-silicon" system are reported. Plasma intermixing was carried out by quasi-stationary compression plasma flows. Power density absorbed by target varied from 0,8 to 1,3 GW/cm^2, pulse duration about 100 µs. XRD-studies revealed the formation of amorphous phase and crystalline hexagonal chromium disilicide $CrSi_2$. Coordination sphere radius of amorphous phase estimates about 0,95 nm and increases with power density. SEM-studies showed amorphous phase to locate in the pre-surface layer (about 1 µm thickness). In accordance with XMA results it contains about 65 at.% of chromium and 35 at. % of silicon. Thermal stability of amorphous phase after 400°C and 600°C anneal is discussed.

11:30 AM
Laser and E-Beam Generated Micro-Nanostructures on the Surface of Amorphous Chalcogenide Layers: *Sandor Kokenyesi*[1]; Viktor Takats[2]; Istvan Chernovich[1]; Mihail Trunov[3]; Attila Csik[2]; Csaba Cserhati[1]; [1]University of Debrecen; [2]ATOMKI; [3]Uzhgorod National University

Holographic gratings and optical waveguide structures are important in optoelectronics. They can be rather easily created by optical or e-beam recording in special materials due to the stimulated changes of their optical and other parameters. Last time special interest is increasing to the direct, one step recording processes, especially connected with surface modulation at micro- and nanoscales. In a wide class of functional materials amorphous chalcogenides are interesting because of the complex of light- and e-beam induced effects like giant surface deformations. These data show on the prospects of direct, one-step recording process which may be especially useful for prototyping optical, photonic elements, but the correlations with composition of the material and experimental conditions are not clear yet. It was a reason of our detailed investigations on the surface deformation processes in amorphous layers made from different As-S(Se) chalcogenide glasses and some nanolayered films.

11:50 AM
A High Throw Bright Acid Copper for Rack Plating of Printed Circuit Boards: *Xiao Faxin*[1]; Shen Xiaoni[1]; [1]Henan University of Science and Technology of China

A novel acid copper plating process produces a bright deposit with excellent throwing power from a sulfate system. Particular attention is placed on the effect of phenyl poly disulfide propane sulfonate, PEG and 2-Mercapto benzimidazole on the copper coating and the appropriate concentration of these additives is 20 mg/L, 60 mg/L and 0.6mg/L, respectively. The bright smooth copper layer may be deposited at 1-5 A/ dm^2 and 20-45° in this solution. The throwing power can reach 91.5% and the deepening plating ability of hole with L/d of 5 is 100%, which shows that this process may be used for rack plating applications of printed circuit board. The corruption rate of the coating is 0.0548g/(m^2•h) in 5%NaCl solution. The XRD results show that the crystal face is mainly assigned to the (112) crystal face on the deposits. The SEM results show that the appearance is uniform and the grain is superfine.

12:10 PM
Electroless Cu Metallization of Carbon Fiber by Precious-Metal Free Process: *Che Dehui*[1]; Yao Guangchun[1]; Liu Kai[1]; Cao Zhuokun[1]; [1]Institute of Materials and Metallurgy, Northeastern University

The paper introduces the new technique of electroless copper deposition on the carbon fibers under absence of precious-metal as catalyzer. Cu layer were electrolessly deposited on the surface of carbon fiber without having to use the conventional palladium or silver catalyst to initiate the redox reaction leading to metallization. This new technique showed that Ni seeds can serve as excellent catalyst, which expedite the re-deoxidation reactions. Through experiment, the activation temperature, concentration, the PH value, were optimized, and a orbicular copper plating layer of carbon fiber in copper sulfate salt-based conventional electroless solution was obtained. The Surface morphology of copper coating was characterized by scanning electron microscope (SEM) and X-ray diffraction (XRD). The results indicate that uniform and smooth copper coating could be obtained by the new precious-metal free activation process. The whole copper coating thickness is about 1 micron.

Sustainable Materials Processing and Production: Motivating Sustainability II

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee, TMS: Education Committee
Program Organizers: Christina Meskers, Umicore; Randolph Kirchain, Massachusetts Institute of Technology; Diana A. Lados, Worcester Polytechnic Institute; Markus Reuter, Ausmelt Limited

Tuesday AM Room: 2B
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Christina Meskers, Umicore Precious Metals Refining; Elsa Olivetti, MIT

8:30 AM Introductory Comments

8:35 AM Keynote
Title Not Available: *Guido Sonnemann*[1]; [1]United Nations Environmental Program (UNEP)
Abstract not available.

9:00 AM Keynote
Title Not Available: *Jim Puckett*[1]; [1]Basel Action Network
Abstract not available.

9:25 AM
European Recycling Platform – Experiences from a New Venture: *Hans Korfmacher*[1]; [1]The Procter & Gamble Company

Product take-back regulations implemented in Europe have presented significant challenges to the consumer electronic product and battery industries. Initially companies established single compliance organisations within individual Member States. These programs met business needs but were inefficient and costly. They were protected from pressures of marketplace competition and could not leverage economies of scale. In order to address these problems P&G, Hewlett Packard, Sony and Electrolux conceived a pan-European compliance organisation to collect waste electronic equipment Europe-wide. This organization, called the European Recycling Platform (ERP), operates in competition with existing national compliance systems. Since 2002 ERP has collected 700,000+ tons of waste electronic products and delivered millions of dollars in cost savings. This presentation reviews the steps needed to establish successful competitive collection organizations that provide sustainable recycling at the lowest cost.

9:50 AM Break

10:05 AM Invited
Scarce Metals and Emerging Technologies: Strategies towards a Sustainable Governance: *Patrick Wäger*[1]; Daniel Lang[2]; [1]Empa; [2]ETH Zürich

Emerging technologies such as photovoltaics or information and communication technologies (ICT) play an increasingly important role in our society. For several of these technologies it is claimed that they significantly contribute to a transition towards a more sustainable society. As many of these technologies are based on scarce metals, a scholarly and societal discussion has emerged regarding, the questions if (ii) the accessibility to the scarce metals might restrict the diffusion of emerging technologies and (ii) the increasing demand for scarce metals might counterbalance the positive effects expected from these technologies in view of sustainable development. Scarce metals supply is governed by a multitude of geological, geopolitical, technological, ecological and other factors. The identification of options for a more sustainable scarce metals supply hence requires the application of an integrative, systemic perspective that considers existing uncertainties as well as the partly diverging interests of different societal actor groups (e.g. state, company or individuals). In our contribution we fwill derive major challenges with respect to a more sustainable supply of scarce metals, sketch a framework of possible approaches to cope with these challenges and illustrate the approaches with selected examples. The framework is intended to be a first step towards sustainable

governance of scarce metals, which, in addition to supply issues also will have to consider the demand side.

10:30 AM
Lithium-Ion Batteries: Examining Material Demand and Recycling Issues: *Linda Gaines*[1]; Paul Nelson[1]; [1]Argonne National Laboratory

Use of vehicles with electric drive, which could reduce our oil dependence, will depend on lithium–ion batteries. But is there enough lithium? Will we need to import it from a new cartel? Are there other materials with supply constraints? We project the maximum demand for lithium and other materials if electric-drive vehicles expanded their market share rapidly, estimating material demand per vehicle for four battery chemistries. Total demand for the United States is based on market shares from an Argonne scenario that reflects high demand for electric-drive vehicles, and total demand for the rest of the world is based on a similar International Energy Agency scenario. Total material demand is then compared to estimates of production and reserves, and the quantity that could be recovered by recycling, to evaluate the adequacy of supply. Finally, we identify producing countries to examine potential dependencies on unstable regions or future cartels.

10:55 AM
Critical and Strategic Failure of Rare Earth Resources: *James Kennedy*[1]; [1]Wings Enterprises, Inc.

Rare Earths: Understanding The United States complete failure of status and relevance in Rare Earth Oxides, Elements and Alloys. Although rare earth oxides are strategic and critical in their own right, elements and alloys are even more important for the development of "Green Technologies" and advanced weapons systems. America does not have an active domestic source of rare earth oxides. America does not have any Heavy Rare Earth Elemental or Alloy capabilities. The magnitude of this failure continues to become more apparent as the manufacture of REO based electronics and magnetic components moves eastward towards China. China has succeeded in outmaneuvering The United States and the world in its quest to control the Rare Earth Oxides, Elements and Alloys, and the many industries dependent upon REO. What can be done?

11:20 AM
Motivating Sustainable Material Use through Industry-Level Simulation Modeling of Platinum Stocks and Flows: *Elisa Alonso*[1]; Richard Roth[1]; Frank Field[1]; Randolph Kirchain[1]; [1]MIT

Manufacturing firms who rely on finite resources have a stake in ensuring sustainable materials use. Higher material prices and uncertain material supply, consequences associated with scarcity, can be damaging to firms. Firms have a number of options to improve material sustainability including dematerialization, material substitution and recycling. However, these options require technological capability, capital investments and supply-chain infrastructure and firms may decide to only make such investments if material prices increase. Once decisions are made, there are further delays before changes can be implemented, delays that may harm the slower-to-react firms. The impact of these delays on firms facing material scarcity was examined for the case of different platinum-using industries. It was found that these delays are particularly problematic for firms when limited material availability leads to volatile market conditions. Industries can benefit especially if they ensure supply diversity by using recycled materials as well as primary.

11:45 AM
A Collaborative Tool for Waste Management in the Industry: *Marisa Borges*[1]; Humberto Riella[1]; Paulo Janissek[2]; [1]Universidade Federal de Santa Catarina; [2]Positivo University

The industry needs to reach environmental patterns, technological innovations and demands of the productive sector, is covered by this study. The environmental care, responsibility in the industry activities and the necessary cultural changes for companies and entities is also emphasized. A collaborative tool was developed to help all partners involved in the environmental administration. This tool is focused on the essential interaction among residue generators belonging to the all productive chain. It was addressed to the industrial unit of a vehicle assembler company, with additional support of entities or institutions with emphasis in the research, development and innovation. To find the best environmental solution it is necessary an expertise and capacity to develop new applications and products, as well as reuse, recycling and reduction of waste generated on industrial processes. The aim of this work is to establish a tool for socio-environmental management.

12:10 PM Concluding Comments

The Vasek Vitek Honorary Symposium on Crystal Defects, Computational Materials Science and Applications: Dislocations I

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Mo Li, Georgia Institute of Tech; David Srolovitz, Institute for High Performance Computing, Agency for Science, Technology and Research, Singapore; Adrian Sutton, Imperial College London; Vaclav Paidar, Institute of Physics AS CR vvi; Jeff De Hosson, University of Groningen

Tuesday AM Room: 603
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Roman Groger, Academy of Sciences of the Czech Republic; Jeff De Hosson, University of Groningen

8:30 AM Invited
First Principles Study of Dislocation Cores and Solute Interactions: *Christopher Woodward*[1]; Dallas Trinkle[2]; Louis Hector[3]; [1]Air Force Research Laboratory; [2]University of Illinois, Champaign Urbana; [3]General Motors Research and Development Center

The local strain field around a dislocation, the dislocation core, can have a strong influence on local and macroscopic deformation behavior in both FCC and BCC metals. This presentation will review the variety of dislocation cores that have been derived using density functional theory coupled with a flexible boundary condition method. These include a/2<111> screw dislocations in Mo, Ta, and Fe, and a/2<110> screw dislocations in fcc Al, Ir, and L10 TiAl. In the BCC transition metals, contrary to classical central-force potentials, this first principles method always produces a screw dislocation with an isotropic core. First principles methods have also been used to reassess solution softening in Mo-Re and Mo-Pt alloys. Finally, we have been calculating the solute dislocation interactions of a/2<110> edge dislocations with Mg, Si, Cr, and Ge solutes in FCC Al. Progress towards understanding solute diffusion effects in the core region will be reviewed.

8:55 AM Invited
Multiscale Modeling of Cross-Slip: Knowns and Unknowns: *Ladislas Kubin*[1]; Benoit Devincre[1]; [1]CNRS

The impact of cross-slip on dislocation microstructures and the mechanical response of fcc crystals is discussed from two examples. 1 - In stage I, mesoscale observations and atomistic simulations indicate that screw dipoles are annihilated by athermal cross-slip, which leads to the formation of superjogs. Dislocation dynamics simulations show that the interactions of superjogs with primary dislocations lead to the observed microstructures containing bundles of elongated prismatic loops. From these results the strain hardening rate can be estimated. 2 – During the dynamic recovery stage III, the thermally activated annihilations of screw dislocations can be described as involving the formation of screw dipoles and their annihilation in a complex local stress field. The incorporation of cross-slip events into dislocation-based continuum models and the reasons why current atomistic simulations or elastic models cannot provide sufficient information for a parameter-free modeling of strain hardening during stage III are discussed.

9:20 AM
Dislocation Nucleation and Re-Ordering of Bicrystal Interfaces: Garritt Tucker[1]; *David McDowell*[1]; [1]Georgia Institute of Technology

Nanostructured materials produced by severe plastic deformation techniques contain a higher percentage of grain boundaries of high-angle character in a non-equilibrium or energetically metastable state. Excess free volume associated with this metastable state modifies fundamental attributes of grain boundaries such as atomic mobility, energy, and mediation of dislocations. Atomistic simulations with importance sampling are used to construct representative non-equilibrium bicrystalline grain boundaries in both copper and aluminum using an embedded atom method potential. The excess free volume results in changes to the initial atomic composition and deformation response under both uniaxial

tension and simple shear at low homologous temperature. Uniform and gradient free volume distributions are used to investigate the free volume redistribution and atomic reordering within the grain boundary plane during straining. A detailed comparison between copper and aluminum is provided with regard to boundary strength, deformation mechanisms, and stress-assisted free volume migration during both tensile and shear simulations.

9:35 AM

Thermally-Activated Glide of Dislocations at the Atomic Scale in High Peierls Stress Crystals: *David Rodney*[1]; Laurent Proville[2]; [1]Grenoble Institute of Technology; [2]Commissariat a l'Energie Atomique

The thermally-activated glide of dislocations in crystals such as BCC and HCP metals is difficult to account for at the atomic scale because the nucleation of kink pairs on dislocation lines occurs on timescales long compared to those accessible to molecular dynamics. So far, thermally-activated glide has been modeled only the case of mesoscale Dislocation Dynamics simulations, using phenomenological laws fitted on macroscopic experiments. Here, in a multi-scale approach, we employ 2 saddle-point search methods, the Nudged Elastic Band method and the Activation-Relaxation Technique, to determine in different atomic systems the activation enthalpy for kink-pair nucleation as a function of applied shear stress. The validity of these static enthalpies is proved by comparison with molecular dynamics and we show that the enthalpy-stress curve can be predicted from a simple line tension model if one accounts for a shear-stress dependence of the Peierls potentials evidenced by the atomic scale simulations.

9:50 AM Invited

Dislocations and Phase Transformations in Energetic Molecular Crystals: *Marc Cawkwell*[1]; [1]Los Alamos National Laboratory

Despite their extreme brittleness, plastic deformation mediated by dislocations and/or phase transformations is thought to control many of the properties of energetic molecular crystals. I will review recent computational and experimental work toward a rigorous characterization of the mechanisms for plastic flow in orthorhombic cyclotrimethylene trinitramine (RDX) single crystals under shock and quasi-static loading conditions. Owing to the low symmetry of these crystals and their degrees intramolecular freedom it will be demonstrated that a surprisingly large number of deformation mechanisms are possible which depend sensitively on orientation and strain rate. Finally, contradictory results from recent nanoindentation experiments will be clarified using the calculated structure of the core of the [100] screw dislocation in RDX.

10:15 AM Break

10:35 AM Invited

Determination of Dislocation Type by X-Ray Line Profiles: *Geza Tichy*[1]; [1]Eotvos University, Budapest

The width of diffraction profiles of Bragg reflection in not monotonous function of the diffraction angle. This behaviour is caused by the anisotropic contrast effect of dislocations. The broadening of the line is characterized by the contrast factor. The calculation of the anisotropic contrast factor is based on the elastic field of dislocation determined by Stroh formalism. The effect is illustrated by examples of severely deformed polycrystalline materials

11:00 AM

Thermally Activated Glide of Partial Dislocations in Nickel Based Superalloys: Libor Kovarik[1]; Raymond Unocic[1]; Ning Zhou[1]; Yunzhi Wang[1]; *Michael Mills*[1]; [1]The Ohio State University

A rich variety of deformation processes can operate in Ni-based superalloys depending on the deformation condition and the nature of the γ/γ' microstructure. In the temperature range of 600-800°C, and for low stress and strain rate conditions, the deformation mechanisms include microtwinning, a[112] dislocation ribbon and superlattice intrinsic and superlattice extrinsic stacking fault formation. Complex arrangements of Shockley partial dislocations are responsible for the operation of these mechanisms, as revealed by high-resolution STEM imaging and ab-initio calculations of the fault configurations. It is found that while these mechanisms are distinct, they are all controlled by the same thermally activated process of chemical reordering in the γ' precipitates after shearing. In addition to reordering, other factors such as segregation of heavy elements have been identified as a simultaneous process. Professor Vitek's pioneering work on dislocation cores in L12 compounds will be discussed in connection to these mechanisms in the two-phase microstructures.

11:15 AM

Predicting Dislocation Mobility from Explicit Atomistic Details: A Kinetic Monte Carlo Study: *Mukul Kabir*[1]; Timothy Lau[1]; David Rodney[2]; Sidney Yip[3]; Krystyn Van Vliet[1]; [1]Department of Materials Science and Engineering, Massachusetts Institute of Technology; [2]Science et Ingenierie des Materiaux et Procedes, Grenoble Institute of Technology; Department of Materials Science and Engineering, Massachusetts Institute of Technology; [3]Department of Nuclear Science and Engineering and Department of Materials Science and Engineering, Massachusetts Institute of Technology

We report kinetic Monte Carlo simulations of dislocation climb at elevated temperatures for heavily deformed body-centered cubic or alpha iron. This approach explicitly incorporates energy barriers associated with iron vacancy-dislocation interactions as determined from atomistic calculations, and enables observations of self-diffusivity and climb over experimentally relevant timescales. We find that, as vacancies approach the edge dislocation cores, the energy barriers to vacancy migration rapidly decrease and consequently, the diffusivity is accelerated in the vicinity of dislocation cores. We also find that the climb velocity to be a complex function of applied stress through the corresponding dislocation density and vacancy supersaturation density as well. The calculated macroscopic creep rates at elevated temperatures are in quantitative agreement with available experiments in terms of the so-called creep stress exponent of power law creep, and in qualitative agreement with our calculated dependence of activation barriers on applied stress.

11:30 AM

Short Range and Long Range Spatial Correlation Effects on Dislocation Distributions: *Juliette Chevy*[1]; Claude Fressengeas[2]; Mikhail Lebyodkin[2]; Vincent Taupin[2]; Pierre Bastie[3]; Paul Duval[4]; [1]University of Illinois; [2]Université Paul Verlaine-Metz-France; [3]Institut Laue Langevin; [4]Laboratoire de Glaciologie et Géophysique de l'Environnement

Ice single crystals deformed in torsion at different strain levels were analyzed by hard X-ray diffraction. Because of the very strong viscoplatic anisotropy of this material and the geometry of the tests, this technique enabled obtaining from the diffracted intensity the local geometrically necessary dislocation densities along the samples. The local density distribution appeared to be heterogeneous and characterized by a complex scale invariance, which was analyzed by means of a multifractal technique. The analysis revealed that the dislocation density distribution is controlled by different kinds of interactions depending on the strain level. Whereas long range elastic interactions between dislocations are responsible for scale invariance at low strain level, short range interactions inherent to dislocation multiplication mechanisms progressively prevail as deformation proceeds. A field dislocation dynamics model taking into account interactions linked to both long range elastic stresses and dislocation transport was used to support this interpretation.

11:45 AM

Fractals versus Scaling: Self-Similarity in Dislocation Cell Wall Simulations: Yong Chen[1]; Woosong Choi[1]; Stefanos Papanikolaou[1]; *James Sethna*[1]; [1]Cornell University

Some experiments of dislocation cell wall structures evolving in deformed metals have observed fractal structures; others have been analyzed in terms of distributions of cell sizes and misorientations that appear non-fractal, but scale with increasing deformation. We analyze a continuum simulation of geometrically necessary dislocations, relaxing in time. In the absence of climb, we observe self-similar (fractal) cell-wall structures, which we exhibit with a visual real-space renormalization group and analyze in terms of critical exponents for correlation functions of dislocation density, orientation, and plastic distortion. For the same simulation, we analyze the distribution of cell sizes and cell wall misorientations, and compare to the corresponding experiments. In the presence of climb (roughly simulating grain boundary polygonization)we observe non-fractal scaling.

Three-Dimensional Materials Science VI: Applications of 3D Microstructural Data

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, ASM-MSCTS: Texture and Anisotropy Committee, TMS/ASM: Phase Transformations Committee

Program Organizers: Alexis Lewis, Naval Research Laboratory; Anthony Rollett, Carnegie Mellon University; David Rowenhorst, Naval Research Lab; Jeff Simmons, AFRL; Stuart Wright, EDAX Inc-TSL

Tuesday AM Room: 401
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Anthony Rollett, Carnegie Mellon University; Andrew Geltmacher, U S Naval Research Laboratory

8:30 AM Invited

3D Characterisation of Fatigue Cracks Using X-Ray Tomography: From Synchrotron to Laboratory Sources: *Jean-Yves Buffiere*[1]; Nathalie Limodin[1]; Julien Rethoré[1]; Wolfgang Ludwig[1]; Anthony Gravouil[1]; François Hild[2]; Stéphane Roux[2]; [1]Universite de Lyon INSA LYON; [2]LMT, ENS-Cachan / CNRS / UPMC / PRES UniverSud Paris

3D images of fatigue cracks in metals are obtained using X-ray micro-tomography. The presentation is twofold. First, synchrotron phase contrast imaging and a small voxel size are used to achieve a high spatial resolution. Results obtained in a fine grain alloy are shown to illustrate the quantitative use of 3D data for modelling propagation through finite element calculations. In a second part, it is shown that laboratory X-ray tomography can also be used quantitatively. Although voxel sizes are somewhat higher, and the beam is incoherent and non-monochromatic, 3D image correlation compensates those weaknesses. This technique provides the 3D morphology and front location of the crack as well as the displacement fields within the specimen. Crack opening displacement maps are obtained in the whole sample cross-section and stress intensity factors are measured all along the crack front. They allow for a quantitative estimation of crack closure/opening.

9:00 AM Invited

Three-Dimensional Validation of Deformation Simulations: *Corbett Battaile*[1]; Luke Brewer[1]; Remi Dingreville[2]; [1]Sandia National Laboratories; [2]Polytechnic Institute of New York University

Many approaches exist to simulate the deformation of engineering materials, but most are based on, and therefore tuned to, macroscopic observables produced by collective phenomena. The fidelity of the material's micro-scale behavior, however, is not guaranteed, especially when the material's internal structure, and therefore it's response, is inhomogeneous (e.g. in a polycrystal). We have used electron backscatter diffraction to characterize the microstructure-scale deformation of pure, polycrystalline nickel and brass in tension. We employ both conventional, two-dimensional characterization of the surface; and in-situ, three-dimensional focused ion beam milling to enable volumetric analysis. These data serve as both input (via microstructure morphology and texture) to, and validation of, finite element simulations of the experiments. We will discuss the approaches used to ensure registry between the experimental and simulation datasets, and the implications of the validation for the micro-scale fidelity of deformation simulations in cases where meso- and macro-scale agreement is established.

9:30 AM

Finite Element Analysis of Large Three-Dimensional Microstructural Datasets: *Alexis Lewis*[1]; M. A. Qidwai[2]; Surya Kalidindi[3]; Stephen Niezgoda[3]; Andrew Geltmacher[1]; [1]Naval Research Laboratory; [2]SAIC; [3]Drexel University

Advances in three-dimensional Materials Science have resulted in the availability of large microstructural datasets representing real materials whose mechanical response can be simulated using advanced Finite Element (FE) codes. Due to the computationally intensive nature of most FE codes, however, the volumes that can be incorporated into FE models often do not contain a statistically significant number of grains. Using n-point correlation algorithms to select statistical volume elements from a large 3D reconstruction of a single-phase beta-Ti, we have simulated the mechanical response of a representative sample of sub-volumes. Using appropriate weighting factors based on grain orientation and boundary crystallography, the data from these simulations is combined to investigate the relationship between microstructure, applied load, and mechanical response on the level of individual grains as well as nearest-neighbor interactions. A correlation between the alignment of individual grains with the loading direction and their plastic response is observed.

9:50 AM

Stagnation of Thin Film Grain Growth under the Effect of Stress: *Fatma Uyar*[1]; Myrjam Winning[2]; Anthony Rollett[1]; [1]Carnegie Mellon University; [2]Max Planck Institute für Eisenforschung

A three-dimensional curvature driven moving finite element model has been adapted to account for the effect of stress field on grain growth in thin films. In this analysis, grain boundaries are treated as dislocation structures and net Burgers vectors have been calculated using the misorientation information and boundary inclinations. Then using Peach-Kohler relation, additional forces due to stress field are calculated. For this study, an experimental thin film texture has been fit to a microstructure represented by 3D digital mesh. The microstructures were allowed evolve under curvature driving forces only and then with an additional stress field. Grain growth dynamics are slowed down by the stress field under certain circumstances. Alternate methods will be also employed to further investigate the stagnation effect.

10:10 AM Break

10:30 AM Invited

Permeability Determination via 3D Reconstruction of the Mushy Zone of Nickel-Base Single Crystals: *Jonathan Madison*[1]; Jonathan Spowart[2]; Dave Rowenhorst[3]; Katsuyo Thornton[1]; Tresa Pollock[1]; [1]The University of Michigan; [2]Air Force Research Laboratory; [3]Naval Research Laboratory

Convective flow at the solid-liquid interface within directionally solidified superalloys can result in the formation of freckles and misoriented grains. These defects can result in reductions in performance and component life. Predictions for the onset of convective flow have primarily employed the Rayleigh number, which is sensitive to the permeability of the mushy zone. Permeability varies with fraction liquid and the geometry of the dendritic array and is difficult to measure experimentally. This research utilizes 3D dendritic reconstructions from solid-liquid interfaces of a model ternary nickel alloy and commercial nickel-base superalloy as direct inputs to fluid flow simulations. These models yield permeability and permit analysis of the role of dendrite morphology and interfacial surface area on flow processes. Permeabilities determined from modeling will be compared to Kozeny-Carmen and modified Blake-Kozeny empirical models and implications for improved directional solidification processing will be discussed.

11:00 AM

A Phase Field Model of Dual-Phase Multi-Grain Material Applied to Duplex Steel with Experimental Verification: *Stefan Poulsen*[1]; Peter Voorhees[2]; Erik Lauridsen[1]; Wolfgang Ludwig[3]; Richard Fonda[4]; Dorte Jensen[1]; [1]Risø DTU; [2]Northwestern University; [3]European Synchrotron Radiation Facility; [4]U.S. Naval Research Laboratory

A phase field model for the microstructural evolution of a binary dual-phase system, where both phases are polycrystalline, has been developed. The model handles both the long-range diffusion processes driving the coarsening and growth of the two phases, and the short-range ordering processes driving the growth of grains. The model has specifically been used to predict the evolution of the microstructure of a duplex stainless steel, which is a material attracting increasing attention due to its strength and corrosion resistance. The phase field simulation employs the microstructure measured using X-ray tomography, holotomography for the phase information and diffraction contrast tomography for the crystallographic information, as an initial condition. Since X-ray tomography is non-destructive we have the unique ability to compare the computed microstructure to that taken experimentally following an ex-situ anneal.

11:20 AM

Anisotropic 3D Phase Field Simulations of Grain Growth: A Comparison between Simulation and Experiment: *Ian McKenna*[1]; Mogadalai Gururajan[2]; Stefan Poulsen[3]; Dave Rowenhorst[4]; Erik Lauridsen[3]; Peter Voorhees[1]; [1]Northwestern University; [2]Indian Institute of Technology - Delhi; [3]Risø; [4]Naval Research Laboratory

We employ phase field simulations to study the evolution of grains in polycrystalline materials. Many materials undergoing grain growth exhibit anisotropic grain structures. Thus, one of the challenges of performing accurate simulations is to develop a robust model capable of handling systems with varying levels of anisotropy in the grain boundary energy and mobility. We have developed such a model which incorporates all five macroscopic degrees of freedom of the grain boundary energy. To validate our model we utilize two different experimental datasets as initial conditions: a serial sectioned sample and an in-situ X-ray tomography sample. Since the X-ray tomography data is taken in-situ it is possible to compare the morphology of individual grains computed using the phase field method with that observed experimentally. Despite the relatively isotropic appearance of the grain structures that are measured experimentally, the one-to-one comparison between simulation and experiment reveals clear evidence of anisotropy.

11:40 AM

Modeling the Effect of Eutectic Nucleation Behavior on Permeability during Solidification of Al-19.5wt%Cu: *Ehsan Khajeh*[1]; Daan Maijer[1]; [1]The University of British Columbia

The nucleation and growth behavior of eutectic phase and the resulting distribution of eutectic colonies within the mushy zone can have a significant influence on interdendritic permeability. Therefore, it will also significantly affect the amount and distribution of feeding related defects. In the present study, 3D geometry of eutectic/primary phases obtained by X-ray microtomography (XMT) has been used to generate a computational domain in which the eutectic transformation takes place. By applying conventional theory of eutectic nucleation and the Monte Carlo technique for simulating the growth of eutectic colonies, the evolution of interdendritic channels has been modeled. The mushy zone permeability during eutectic evolution has then been calculated by solving the full Navier-Stokes equation on the evolving microstructures. The calculated permeability shows the impact of the eutectic solidification behavior.

12:00 PM

Dealloying and Coarsening Behavior of Nanoporous Gold by X-Ray Nanotomography: *Yu-chen Chen*[1]; JaeMock Yi[2]; Wah-Keat Lee[2]; Ian McNulty[2]; Peter Voorhees[1]; David Dunand[1]; [1]Northwestern Univeristy; [2]Advanced Photon Source of Argonne National Lab.

Nanoporous gold has novel mechanical, optical, and chemical properties leading to potential applications for sensors, actuators, catalysts, and fuel cell electrodes. Its nanoporous structure is formed by dealloying and can be coarsened by subsequent annealing. The 3D morphology has been studied by TEM tomography. However, the kinetics underlying structural evolution have not been fully resolved and morphological evolution during dealloying and coarsening has not been directly measured in 3D.Here, by using 3D X-ray nanotomography, we reconstructed the structural evolution of nanoporous gold during both dealloying and coarsening. We have observed a dealloying front progressing within the Ag-Au alloy and quantified the relationship between dealloying distance and time. We have also quantified the nanostructure by creating probability maps for the surface curvature and surface normal during coarsening. These results will advance our understanding of the diffusion mechanism of 3D in a complex nanoporous metallic structure.

12:20 PM

Microstructures Simulation of Magnesium-Based Alloys during Solidification by Phase-Field Method: *Tao Jing*[1]; Mingyue Wang[1]; [1]Tsinghua University

An expression is proposed for the anisotropic function of crystal-melt interfacial free energy for hexagonal metals, based on the combination of experiments and crystal structure. The phase field model of alloys, whose density free energy is built on the basis of thermodynamic extended substitutional-regular-solution approximation, incorporated into anisotropic function reflecting hexagonal symmetry, is established. Numerical computations of primary hcp-Mg phase dendritic growth in the freezing process of magnesium-based alloys melts are implemented. The three-dimensional dendritic morphologies of magnesium alloys microstructures, whose hierarchical branches can be seen clearly, are obtained, which have been well in agreement with experimental results and are obviously different from that of would be usually expected.

Ultrafine Grained Materials – Sixth International Symposium: Deformation and Processing Mechanics

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS: Shaping and Forming Committee

Program Organizers: Suveen Mathaudhu, U.S. Army Research Laboratory; Mathias Goeken, University Erlangen--Nürnberg; Terence Langdon, University of Southern California; Terry Lowe, Manhattan Scientifics, Inc.; S. Semiatin, Air Force Research Laboratory; Nobuhiro Tsuji, Kyoto University; Yonghao Zhao, University of California - Davis; Yuntian Zhu, North Carolina State University

Tuesday AM — Room: 607
February 16, 2010 — Location: Washington State Convention Center

Session Chairs: Gerhard Wilde, University of Muenster; Rainer Hebert, University of Connecticut; Yulia Ivanisenko, Forschungszentrum Karlsruhe; Matthias Hockauf, Chemnitz University of Technology

8:30 AM

Plasticity and Grain Boundary Diffusion at Small Grain Sizes: *Gerhard Wilde*[1]; Nancy Boucharat[2]; Sergiy Divinsky[1]; Joern Leuthold[1]; Gerrit Reglitz[1]; Harald Roesner[1]; [1]University of Muenster; [2]Research Center Karlsruhe

Bulk nanostructured - or ultrafine-grained materials are often fabricated by severe plastic deformation to break down the grain size by dislocation accumulation. Underlying the often observed property enhancement is a modification of the volume fraction of the grain boundaries. Yet, along with the property enhancements, several important questions arise concerning the accommodation of external stresses if dislocation-based processes are not longer dominant at small grain sizes. One such factor are so-called “non-equilibrium” grain boundaries that have been postulated to form during severe deformation and that might be of importance not only for the property enhancement known already today, but also might serve as internal transport pathways offering new applications in the context of e.g. gas permeation or fast matter transport for self-repairing structures. This contribution addresses the underlying issues by combining quantitative microstructure analysis at high resolution with grain boundary diffusion measurements. Funding by DFG (FG714) is gratefully acknowledged.

8:50 AM

Twist Extrusion - Technique for Bulk Ultrafine- and Nanomaterials Obtaining: *Viktor Varyukhin*[1]; Yakov Beygelzimer[1]; Sergey Synkov[1]; [1]Donetsk Institute for Physics and Engineering of NASc of Ukraine

Twist extrusion (TE) is the severe plastic deformation technique for bulk ultrafine- and nanomaterials manufacture. We present an experimental study of the kinematics of TE and show that TE has the following properties: • The mode of deformation in twist extrusion is simple shear. There are two shear planes; one of them is perpendicular and the other is parallel to the specimen axis. • The following processes are present during twist extrusion: vortex-like flow with large strain gradient, stretching and mixing of metal particles.We argue that due to these properties, TE opens possibilities for investigating and forming new structures. It has already been successfully used to obtain ultrafine and nanostructure with good properties in Al, Cu and Ti alloys.

9:05 AM

Plastic Instability during Accumulative Roll Bonding (ARB) of Metallic Multilayers: *Rainer Hebert*[1]; Jyothi Suri[1]; Girija Marathe[1]; [1]University of Connecticut

A common conception is that the flow stress differences between individual layers of multilayers determine if layers codeform or respond with plastic instabilities during cold-rolling. Following this conception Cu-Ni multilayers were prepared with different initial flow stress levels of the Cu and Ni foils to test if codeformation can be extended to higher strain levels during ARB processing for a particular set of work-hardening levels. Microstructure analyses

demonstrate, however, that the plastic instability behavior of the Ni layers is not significantly affected by the choice of the initial thermomechanical treatment of the Cu and Ni foils and thus the flow stress ratio. The experimental results are compared with predictions for plastic instabilities during rolling of multilayers from continuum mechanics models. The comparison shows that additional factors such as shear stresses, texture formation, stress partitioning, or shear band formation have to be considered to rationalize the experimental results.

9:20 AM Invited

Role of Dislocations during Processing and Deformation of Nanocrystalline Materials: *Farghalli Mohamed*[1]; [1]University of California, Irvine

Nanocrystalline (nc) materials are characterized by a unique substructural feature: grain sizes are less than 150 nm. Recent research activities have demonstrated that dislocation activity can play a key role not only in processing nc-materials via severe plastic deformation (SPD) but also in accounting for their mechanical behavior. Various aspects of this role will be presented.

9:40 AM

How do Partials Multiply to Produce Deformation Twins in Nanocrystalline fcc Metals?: *Yuntian Zhu*[1]; [1]North Carolina State University

Deformation twins are often observed in nanocrystalline face-centered cubic (fcc) metals and alloys. However, since the conventional twin growth mechanisms such as the pole mechanism no longer operate in nanocrystalline materials, there has been a puzzle on how the partials multiply to glide on consecutive slip planes. It is statistically improbable for a partial to exist on every slip plane. Here I review several recently proposed mechanisms for the nucleation and growth of four different single and multifold twins. These mechanisms provide continuous generation of twinning partials for the growth of the twins after nucleation. Relatively high-stress or high strain rate is needed to activate these mechanisms, making them more prevalent in nanocrystalline materials than in their coarse-grained counterparts. Experimental observations that support the proposed mechanisms are presented.

9:55 AM Break

10:10 AM Invited

Deformation Mechanisms in Multiscale Nanostructured Materials: Yonghao Zhao[1]; Ying Li[1]; Troy Topping[1]; Xiaozhou Liao[2]; Yuntian Zhu[3]; Ruslan Valiev[4]; *Enrique Lavernia*[1]; [1]University of California-Davis; [2]The University of Sydney; [3]North Carolina State University; [4]Ufa State Aviation Technical University

Multiscale nanostructured materials containing multimodal grain size distributions can be engineered to display unusual balance of properties, such as strength and ductility. Despite well documented reports of enhanced ductility in multiscale nanostructured materials, systematic identification of the underlying deformation mechanisms has not been accomplished. In this work, we selected high-purity Cu and commercial pure Ti as a model material system in an effort to elucidate the underlying mechanisms. The multimodal Cu and Ti were prepared by equal-channel-angular pressing (ECAP) and subsequent annealing. In situ atomic force microscopy (AFM) in combination with tensile testing and ex situ transmission electron microscopy (TEM) are used to characterize the deformation mechanisms and mechanical behavior.

10:30 AM

Information on Deformation Mechanisms in nc Pd-10% Au Inferred from Texture Analysis: *Yulia Ivanisenko*[1]; Werner Skrotzki[2]; Robert Chulist[2]; Lilia Kurmanaeva[1]; Hans-Jörg Fecht[3]; [1]FZK; [2]Technische Universität Dresden; [3]Universität Ulm

There is still a lack of understanding of deformation mechanisms in nanocrystalline (nc) materials. We have studied microstructures formed in nc Pd-10% Au (grain size about 15nm) after high pressure torsion, and have revealed signatures of various deformation processes as cooperative grain boundary sliding, shear banding, dislocation slip and twinning. In order to establish contributions of these processes to total strain, we compared torsion textures formed in nc and coarse grained (cg) samples after identical shear strain. The textures were measured with synchrotron radiation. Intensities of characteristic components of the shear texture are two times stronger in the cg sample than in the nc one, indicating that dislocation slip is less significant in the nc sample. Furthermore, distribution of intensities between different shear texture components presumably points to the contribution of deformation twinning in the nc sample in agreement with microstructure investigations.

10:45 AM Invited

Extending Kocks' Equation for Work Softening in UFG Materials: *Tamás Ungár*[1]; Li Li[2]; Géza Tichy[1]; Hahn Choo[2]; Peter Liaw[2]; [1]Eötvös University Budapest; [2]University of Tennessee

When the grain size is reduced to below about 20 nm and dislocation density is larger than about 10^{16} m^{-2} plastic deformation may cause grain growth and reduction of dislocation density. It is shown that the equation of Kocks describing stage-III deformation can account for the above mentioned behavior by appropriate selection of materials constants. The kind of grain growth, often called "abnormal grain growth" along with the reduction of dislocation density produced by plastic deformation can be considered perfectly "normal" within the frame of the extended Kocks' equation.

11:05 AM

Plastic Deformation in Nanocrystalline and Ultrafine Carbon Steel: *Rodolfo Rodríguez-Baracaldo*[1]; Jose Antonio Benito Páramo[2]; José Maria Cabrera Marrero[2]; [1]Universidad Nacional de Colombia; [2]Universitat Politècnica de Catalunya

This work is focused on the evaluation of plastic deformation of near fully dense nanostructured and ultrafine grained bulk samples of carbon steel (0.55%C). The specimens were obtained by warm static pressing from mechanically milled powder. Compaction parameters and annealing treatments were selected to promote a relatively wide range of the grain sizes (15 nm to 2.8 μm).Hardness, compression and nanoindentation test were performed at room temperature. It was found that the nanocrystalline grained steel samples presented very high strength with low ductility, as the ferritic grain size was increased, the amount of strain hardening and total plastic strain increased and the maximum strength diminished. A constant decrease of strain rate sensibility parameter (m) was found as the grain size decreases in the ultrafine regime. On the contrary, in the nanocrystalline regime as the grain size decreases a slight increase of m was observed.

11:20 AM

Microstructure and Creep Properties of Nanocrystalline Oxide Dispersion Strengthened Fe-18Cr-8Ni-2W-0.25Y2O3 Austenitic Steel Synthesized by High Energy Ball Milling and Vacuum Hot Pressing: P. Susila[1]; D. Sturm[2]; M. Heilmaier[3]; B. S. Murty[1]; *Vadlamani Subramanya Sarma*[4]; [1]Indian Institute of Technology Madras; [2]Otto-von-Guericke University Magdeburg; [3]TU Darmstadt; [4]Indian Institute of Technology Madras and North Carolina State University

Materials for future power plants require enhanced creep resistance, corrosion resistance and oxidation resistance. Austenitic stainless steels (ASS) have good corrosion and creep resistance. The creep properties of the ASS can be further enhanced through oxide dispersion strengthening (ODS). There is limited knowledge base on the microstructure-properties correlations in nanostructured ODS alloys. High energy ball milling is considered to be a better technique to synthesize large quantities of nanostructured materials and well suited to produce ODS alloys. In this presentation, we report the results of studies on microstructure and mechanical properties of nanostructured (Fe-18Cr-8Ni-2W-0.25Y2O3) austenitic ODS alloy. The nanostructured base and ODS alloys were synthesized by mechanical alloying of elemental powders. The mechanically alloyed ODS powders were characterized by X-ray diffraction (XRD) analysis and transmission electron microscopy (TEM). The results show that fcc phase formation is accelerated in the ODS alloy in comparison to the base alloy and a crystallite size of 10 nm is achieved in ODS alloy after 25 h of milling. The ODS alloy powders have a hardness of 1800 HV and this is significantly higher than the hardness of 1200 HV reported for nanostructured ferritic ODS alloy powders. The powders are consolidated to near theoretical density by vacuum hot pressing (VHP). The grain size following VHP is about 75 nm in the ODS alloy. The creep properties will be evaluated and an attempt is made to quantitatively assess the impact of particle as well as grain size strengthening on the high temperature strength.

11:35 AM Invited

Plastic Flow Stability and Detwinning in Cu with Nanoscale Growth Twins: *Amit Misra*[1]; X. Zhang[2]; J. Wang[1]; N. Li[3]; O. Anderoglu[3]; J. Huang[4]; R. Hoagland[1]; J. Hirth[1]; [1]LANL; [2]Texas A&M University; [3]Texas A&M University & LANL; [4]Sandia National Laboratories

Nanotwinned Cu, with a very fine scale structure consisting of twin lamellae with an average thickness of 5 nm, exhibited stable plastic flow with no shear

localization or fracture, even at thickness reduction of over 50% in room temperature rolling. The {111} fiber texture was retained after rolling indicating insignificant out-of-plane rotation of the columnar grains and is interpreted in terms of a symmetric slip model. No significant change in the average twin lamellae thickness was seen even at thickness reduction of over 50%, suggesting that some twin boundaries were annihilated during deformation. The detwinning of nanotwins was also shown via in situ indentation experiments in a transmission electron microscope. Molecular dynamics simulations show that de-twinning is accomplished via a collective glide of multiple twinning dislocations and that de-twinning becomes the dominant deformation mechanism when the thickness of twins is on the order of a few nanometers.

11:55 AM

Processing and Oxidation Resistance of Nanocrystalline Fe-Cr Alloys: Rajeev Gupta[1]; *Raman Singh*[1]; Carl Koch[2]; [1]Monash University; [2]North Carolina State University

For their unique physico-mechanical properties and exciting industrial applications, nanostructured materials are the most widely investigated materials research topic of recent times. However, corrosion of nano materials has received very limited research attention, even though the materials will be required to demonstrate acceptable corrosion resistance in most potential applications. This paper will present a review of the fundamentals of likely corrosion mechanism of nanocrystalline metallic materials with examples. The paper will present description of the processing of nanocrystalline Fe-Cr alloys by mechanical alloying. The paper will also discuss the most recent findings to address the hypothesis that it may be much easier to develop a protective film on nanocrystalline alloys. Validation of the hypothesis will have highly attractive economic/industrial implications.

Ultrafine Grained Materials – Sixth International Symposium: Stability

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS: Shaping and Forming Committee

Program Organizers: Suveen Mathaudhu, U.S. Army Research Laboratory; Mathias Goeken, University Erlangen--Nürnberg; Terence Langdon, University of Southern California; Terry Lowe, Manhattan Scientifics, Inc.; S. Semiatin, Air Force Research Laboratory; Nobuhiro Tsuji, Kyoto University; Yonghao Zhao, University of California - Davis; Yuntian Zhu, North Carolina State University

Tuesday AM Room: 606
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Kristopher Darling, U.S. Army Research Laboratory; Brady Butler, U.S. Army Research Laboratory; Christopher Saldana, Purdue University; Jane Adams, U.S. Army Research Laboratory

8:30 AM Invited

Thermal Stability of SPD Microstructures: *Günter Gottstein*[1]; Xenia Molodova[1]; [1]RWTH Aachen University

The annealing of plastically deformed crystals generally causes softening processes, recovery and/or recrystallization, to occur. During severe plastic deformation pro¬nounced cellular microstructures of submicron dimensions develop which convey beneficial properties to such materials. Since softening processes are usually accom¬panied by microstructural coarsening they may be detrimental to the mechanical performance of the processed material, though. On the other hand an increasing strain leads to the transformation of the cellular dislocation structure to a granular arrangement with a large fraction of high angle grain boundaries that may resist recovery and recrystalli¬zation.We will present results of investigations into the thermal stability of ECAP generated microstructures of Cu and Al alloys, identify the softening mechanisms and propose strategies to improve the resistance to coarsening and softening during annealing of SPD materials.

8:50 AM

Mechanical Properties of Nanocrystalline Aluminum Stabilized with Dimondoids: *Khinlay Maung*[1]; Ali Yousefiani[2]; Farghalli Mohamed[1]; James Earthman[1]; [1]UCI; [2]The Boeing Company

Nanocrystalline aluminum stabilized with either 1 wt% diamantane, 5 wt% diamantane, or 7 wt% of diamantane nanoparticles were processed by cryomilling. The as-cryomilled powders containing diamantane had an average grain size of 22 nm. Thermal stability analysis shows that the average grain size for 1% diamantane and 5% diamantane were not significantly different and the average grain sizes for these samples annealed at 500°C for 10hr were well under 100 nm. The cryomilled alloy powders were consolidated by hot isostatic pressing (HIP) followed by extrusion. The average grain size of as HIP'd samples are slightly larger than the powders heat treated at the same temperature range. This effect will be discussed in terms of stress assisted grain growth model. Mechanical properties of the three different compositions will also be presented.

9:05 AM

Characterization of Thermally Stable Nanocrystalline Nickel Powders: *Brady Butler*[1]; Kristopher Darling[1]; Bradley Klotz[1]; Matthew Kelly[1]; Micah Gallagher[1]; Eric Klier[1]; [1]U.S. Army Research Laboratory

The development of nanocrystalline and ultra-fine grained bulk materials through powder metallurgy processes has long been constrained by the thermal stability of nanostructures. Many difficulties arise due to the fact that grain growth and densification occur within the same temperature regime. Additionally, the mechanisms supporting densification require some degree of grain coarsening in order to achieve full density. This study outlines the development of a thermally stable nanocrystalline nickel powder and discusses the influence of this grain size stability on the overall densification rate during pressureless sintering in hydrogen atmosphere. Furthermore, the activation energy of sintering is calculated to determine whether the mechanisms of grain growth and densification can be decoupled through the addition of thermal grain stabilizers.

9:20 AM Invited

Room Temperature Instability of Super Saturated Solid Solution in a Nano Crystalline Al-4Cu Alloy Produced by SPD: *Phil Prangnell*[1]; Joe Robson[1]; Yan Huang[1]; [1]The University of Manchester

The reduction in grain size achievable and long term stability of severely deformed aluminium has been investigated when copper in solution is used to inhibit recovery. It is shown that copper is more effective than magnesium in inhibiting dynamic recovery. A grain width of only 70 nm was obtained in an Al-4wt%Cu alloy, after processing by ECAE to a strain of εeff ~15, resulting in a lamellar nano-grain structure. However, post-processing the severely deformed solid solution was found to be unstable at room temperature and copious precipitation of θ occurred at new grain boundaries within the deformed state, leading to recovery of the deformation structure and a loss of strength. The solute level fell to equilibrium within ~ 9 months. The precipitation kinetics were shown to occur at many orders of magnitude higher than can be predicted by classical theory. The reasons for this discrepancy are discussed.

9:40 AM

Characterizing Ultrafine Grained Ti-6Al-4V Thermal Stability with Long-Term High-Resolution EBSD: *Andrew Deal*[1]; Radhakrishna Bhat[1]; Richard DiDomizio[1]; Judson Marte[1]; PR Subramanian[1]; [1]GE Global Research

A near-isothermal multi-axis forging (MAF) process was used to produce ultra-fine grained (UFG) Ti-6Al-4V alloys. To better understand the coarsening kinetics of the primary alpha, the thermal stability of the material was evaluated at temperatures well below the beta transus. Both static and dynamic coarsening were examined using heat treatments and hot compression tests, respectively. To precisely quantify the alpha grain size as a function of time and temperature, a FEG-SEM equipped with EBSD was enclosed to isolate it from thermal oscillations inherent to HVAC systems. The isolation chamber kept the short-term temperature fluctuations of the microscope below ±0.1°C and long-term temperature drift below 1°C. This, in combination with appropriate EBSD protocols, achieved a stable 60nm resolution for periods up to 60 hours. Such long periods of stability enabled more statistically meaningful regions of the UFG Ti-6Al-4V to be characterized with EBSD to study primary alpha coarsening.

9:55 AM Invited
Microstructural Stability and Damage Evolution in Ultra-Fine Grained Alloys under Cyclic Loading: *Thomas Niendorf*[1]; Hans Maier[1]; Ibrahim Karaman[2]; [1]University of Paderborn; [2]Texas A&M University

In the present study the mechanical behavior of ultra-fine grained alloys, e.g. interstitial-free steel and niobium-zirconium alloy, under cyclic loading is investigated and correlated to the microstructural evolution of the materials tested. All materials were processed by ECAP, employing different processing routes in order to obtain microstructures with significant different characteristics. The results obtained in the current study revealed that in addition to the grain size and impurity content the grain boundary characteristic and the grain size distribution have significant influence on the fatigue response of the materials tested. Thorough microscopical observation in combination with the digital image correlation technique allowed for the investigation of the early stages of damage evolution, i.e. crack initiation. By means of EBSD it was found that elongated grain structures dominated by low-angle grain boundaries were the reason for early crack initiation, independent of the ECAP route.

10:15 AM Break

10:30 AM Invited
Stability of Nanocrystalline Alloys: *Christopher Schuh*[1]; [1]MIT

Through alloying, it is possible to dramatically increase the thermal stability of nanocrystalline metals, even without the introduction of secondary phases. Solid solutions are potentially more stable in nanocrystalline form than are pure metals, owing to the possibility of solute segregation to intergranular regions. Segregation provides a deep metastable state for grain boundaries that is both thermodynamically and dynamically more stable. This talk summarizes our modeling efforts on nanocrystalline alloys, which permit the evaluation of structural stability with respect to grain coarsening, phase separation, and chemical ordering. Such models provide guidelines for the design of alloys that are sufficiently stable for use at ambient or even elevated temperatures.

10:50 AM
Thermal Stability of Ultra-Fine Grained Ti-6Al-4V Alloys Processed via Multi-Axis Forging: *Radhakrishna Bhat*[1]; Andrew Deal[1]; Richard Didomizio[1]; Judson Marte[1]; P.R. Subramanian[1]; [1]GE Global Research Center

A near-isothermal multi-axis forging (MAF) process was used to produce ultra-fine grained (UFG) Ti-6Al-4V alloys. The thermal stability of the resulting ultra-fine grained structure was evaluated at low temperatures below the beta transus under both static and dynamic conditions in order to investigate the coarsening kinetics of the alpha grains in the alpha+beta phase field. Specimens were heat-treated for varying durations at different temperatures within the alpha+beta phase field for the static studies, while hot compression tests were conducted at different strain rates to evaluate the thermal stability under dynamic conditions. The results of a new characterization technique, namely electron back scattered diffraction (EBSD) line-scans, optimized for quantitative image analysis of ultrafine alpha and beta grains will be presented. The coarsening mechanisms and thermal stability of the ultra-fine grain Ti-6Al-4V will be discussed, especially in the context of using UFG Ti-6Al-4V for producing near-net shape Ti components via superplastic deformation.

11:05 AM
Grain Growth Kinetics of Thermally Stabilized Nanocrystalline Fe-Alloys: *Kris Darling*[1]; Brian Schuster[1]; Brady Butler[1]; Suveen Mathaudhu[1]; Laszlo Kecskes[1]; [1]ARL

Interfacial energy reduction has been suggested to be a superior method to prevent grain growth in nanocrystalline materials. Recently, examples of thermally stabilized nanocrystalline alloys have emerged showing dramatic high temperature stability owing to grain boundary segregation of solutes and the associated interfacial energy reduction. Such samples are typically synthesized through high energy non equilibrium processes and therefore limited to small product geometries such as particles. Furthermore, the very same mechanism responsible for the high thermal stability can often lead to frustrated atomic diffusion and hinder the densification required for production of bulk samples. The primary objective of this study was to delineate the grain growth kinetics of thermally stabilized Fe based nanocrystalline alloys by use of differential scanning calorimetry. Knowledge of such grain growth kinetics will establish the appropriate consolidation parameters for successful densification in the future.

11:20 AM Invited
Elemental Redistribution Induced by High-Pressure Torsion in Alloys: *Xiaozhou Liao*[1]; Song Ni[1]; Yanbo Wang[1]; Gang Sha[1]; Simon Ringer[1]; Terence Langdon[2]; Yuntian Zhu[3]; [1]The University of Sydney; [2]University of Southern California; [3]North Carolina State University

Severe plastic deformation techniques including high-pressure torsion have been widely used to refine materials for superior mechanical properties. Complex structural evolutions occur during severe plastic deformation, which include not only grain refinement but also composition redistribution for alloys. The composition redistribution is expected to affect significantly the mechanical properties and structural thermal stability of the alloys. This presentation discusses the effect of high-pressure torsion on the compositional distribution in two materials systems. The starting materials are both solid solutions – one in an equilibrium state and the other in a supersaturated non-equilibrium state. The composition distribution was investigated using transmission electron microscopy and atom probe tomography.

11:40 AM
Engineering Stored Energy in Ultra Fine Grained Metals Created by Severe Plastic Deformation: M. Ravi Shankar[1]; *Shashank Shekhar*[1]; Jiazhao Cai[1]; [1]University of Pittsburgh

Severe plastic deformation (SPD) induces very high density of dislocations and other defects in material systems. The storage of these defects results in nanostructured interfaces which are characterized by a finite stored energy. This energy offers a component of the driving force for thermally-induced coarsening which leads to softening, and therefore directly influences microstructure stability. Here, we demonstrate means for manipulating the stored interfacial energy for achieving tunable stability as well as mechanical property combinations. It is demonstrated that by manipulating strain, strain-rate and temperature of severe plastic deformation, it is possible to engineer fine microstructures with subdued stored interfacial energy, low enough so that the as prepared material remains stable over a relatively larger range of temperature, but still retain high strength and sometimes, even enhanced ductility.

2010 Functional and Structural Nanomaterials: Fabrication, Properties, Applications and Implications: Synthesis of Nanomaterials II

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee
Program Organizers: David Stollberg, Georgia Tech Research Institute; Nitin Chopra, University of Alabama; Jiyoung Kim, University of Texas - Dallas; Seong Jin Koh, University of Texas at Arlington; Navin Manjooran, Siemens Corporation; Ben Poquette, Keystone Materials; Jud Ready, Georgia Tech

Tuesday PM Room: 214
February 16, 2010 Location: Washington State Convention Center

Session Chair: Jiyoung Kim, University of Texas - Dallas; David Stollberg, Georgia Tech Research Institute

2:00 PM Introductory Comments

2:05 PM Invited
Nanowires of Phase Change Materials for Memory Applications: Jeong-Soo Lee[1]; Chan Hoon Park[1]; Jung Hyun Cho[1]; Ki Hyun Kim[1]; Yoon-Ha Jeong[1]; *Meyya Meyyappan*[2]; [1]National Center for Nanomaterials Technology; [2]NASA Ames Research Center

Nanoscale memory technologies providing an alternative to flash memory have been receiving much attention in recent years. Among them, phase change random access memory(PCRAM) has shown considerable promise in terms of endurance, programming current and other metrics and therefore is ready for market. But continued scalability, density and reduction in current for many more generations to come will have unique challenges. The nanowire(NW) form of the same phase change materials such as germanium telluride(GT), GeSbTe(GST) and indium selenide provides an alternative to tackle future challenges. First, the NW memory cell volume is inherently smaller in size thus driving the thermal budget down. Second, it is well known that nanomaterials

exhibit a lower melting point relative to their bulk counterparts, which will also help to reduce the thermal budget. Other thermal properties are also favorable in the nanowire form. These aspects together will reduce the programming current needs. We have grown nanowires of the above phase change materials using a vapor-liquid-solid approach and characterized them extensively. This presentation will provide details of growth results, characterization, evidence of melting point reduction and preliminary device results for programming current and switching ratio between SET/RESET.

2:25 PM

Effects of Process Parameters of ALD on High-k Dielectric Deposition on HOPG for Graphene Based Nanoelectronics: *Greg Mordi*[1]; Bongki Lee[1]; Jiyoung Kim[1]; [1]University of Texas at Dallas

Graphene, a single layer of graphite with 2-D honeycomb structure, has been the center of attention as an emerging material for novel nanoelectronics, thanks to its unique electronic properties and potential applications. In order to realize high performance nanoelectronic devices, it is required to build localized gate structures on a graphene layer. Atomic layer deposition (ALD) is expected to be a suitable technique to deposit conformal thin high-k dielectric films while avoiding damages on the graphene layer. Unfortunately, chemical inertness of a pristine graphene layer causes an additional challenge for high-k dielectric deposition. Process conditions of ALD such as dielectric deposition temperature, type of oxidant, oxidant exposure dose, the dielectric material used and its growth rate must be carefully understood and controlled to achieve a high quality and conformal gate dielectric deposition while limiting any process induced defects. In this work, we extensively and systematically examine the effects of temperature and oxidant exposure dose on highly ordered pyrolytic graphite (HOPG), a chemically inert surface of 3-D bulk form of graphite that accurately mimics graphene and characterize the surface before and after ALD process using atomic force microscope, Raman spectroscopy and high resolution transmission electron microscopy (HRTEM) in order to optimize a standard for dielectric deposition. We also investigate electrical characteristics of the dielectric. .

2:45 PM

Dual-Gated Graphene Devices with High-k Dielectric Using Ozone-Based Atomic Layer Deposition (ALD): *Bongki Lee*[1]; Greg Mordi[1]; Jiyoung Kim[1]; [1]University of Texas at Dallas

Graphene, a recently discovered single sheet of carbon atoms, stands out as an outstanding material that possesses a unique band structure as well as a high electronic mobility, which allows for fundamental studies in condense-mater physics and potential applications in nanoelectronic devices. In order to realize graphene-based devices, many challenging issues must be overcome. One of the challenges is the absence of a high quality dielectric layer on graphene required for local gate application due to its chemically inert surface. We have reported a facile process that allows for a conformal deposition of ALD dielectric on HOPG (highly oriented pyroltic graphite) by using O3 treatment. In this study, we will present an O3-based atomic layer deposition process to deposit high-quality of gate dielectrics on single layer graphene for localized gate applications. The performance of a top-gated graphene device utilizing an O3-based Al2O3 dielectric in a dual-gated structure will be presented.

3:05 PM

The Interface Characteristics and the Electrical Properties of ZnO/ITO/ZnO Nano Thin Films: Fei-Yi Hung[1]; *K. J . Chen*[2]; S. J. Chang[2]; Z. S. Hu[2]; Y. T. Chen[1]; [1]Institute of Nanotechnology and Microsystems Engineering, Center for Micro/Nano Science and Technology, National Cheng Kung University; [2]Institute of Microelectronics and Department of Electrical Engineering; Center for Micro/Nano Science and Technology, National Cheng Kung University

ZnO film has the advantage of thermal stabilization while ITO film plays an important role in electrical conductivity. The present ZnO/ITO/ZnO tri-layer thin films were designed and fabricated by RF sputtering. The results showed that increasing the thickness of the ITO interlayer had no obvious effect on the optical transmittance. Notably, a 13 nm ITO interlayer, the ZnO/ITO(13nm)/ZnO thin film possessed better crystallized structures and electrical properties. Also, the interfacial characteristics of the ZnO/In/ZnO thin film was studied to confirm the contribution of the ITO interlayer on the electrical properties of the ZnO/ITO/ZnO thin film. Under the thermal effect, the indium atoms in the ITO film had migrated into the ZnO matrix and improved the conductivity of the thin film.

3:25 PM

Nanotube Grafting on Porous Solids for Multifunctional Applications: *Sharmila Mukhopadhyay*[1]; [1]Wright State University

Recent investigation in this group allows grafting of strongly bonded carbon nanotubes (CNT) in porous cellular materials for creating robust structures with very high surface to volume ratio. These form multi-scale hierarchical surfaces similar to those in hair and capillary type structures abundantly seen in biological systems. Once the core substrate is created, the increased surface area can be modified for functional applications such as porous electrodes, catalysts, filters, thermal radiators, tissue scaffolds etc. This paper will discuss the nucleation-growth mechanisms involved in the two-step fabrication technique that makes such structures possible. The effectiveness of CNT-grafted porous structures fabricated so far have been found to be very promising in the following areas, and will be discussed: (i) Porous supports for nano-catalyst particles such as Pd, (ii) Scaffolds for cell and protein growth, (iii) Thermal management devices, and (iv) Core support for structural composites.

3:45 PM Break

4:00 PM

Synthesis and Surface Roughening of $CoSb_3$ Nanowires by Electrochemical Methods: *Dat Quach*[1]; Ruxandra Vidu[1]; Pieter Stroeve[1]; Joanna Groza[1]; [1]University of California, Davis

Cobalt antimonide ($CoSb_3$) belongs to a group of materials called skutterudites that possess promising thermoelectric properties. While most studies on CoSb3 have been focused on the so-called phonon-glass / electron-crystal approach in order to increase the material's figure of merit, the low dimensional materials approach is rarely discussed due to difficulties involved in the processing of nanostructured $CoSb_3$. In this work the synthesis of $CoSb_3$ nanowires by electrodeposition is investigated under different deposition conditions. Surface roughening of such nanowires is also applied via electrochemical treatment in order to further enhance materials properties. Characterizations of the nanowires by electron microscopy and x-ray diffraction are also discussed.

4:20 PM

Intermediate Composition of at wt. % Mn/Ni in Amorphous, Nano or Microcrystalline Alloy Films of Fe-Mn or Fe-Ni Deposited from Aqueous Solution of Simple Salt Bath: *Bassey Udofot*[1]; [1]NASA/Goddard Space Flight Center

Fe-Mn as well as that of Fe-Ni alloy films are can be deposable by electrochemical means from reducing aqueous solution or thermally reducing vacuum or furnace atmospheres on insulator, semiconductor or conductive substrate surfaces. These alloy films is soft ferromagnetic materials possessing low residual induction, low hysteresis loop and low coercive force, maximum permeability and low thermal coefficient of thermal expansion useful for various applications. Thermally produced films are thinner with limited crystalline structures ranging from nano to polycrystalline or amorphous film structures. Electrochemically produced film are cheap requiring low temperature applications and the films produce ranges from nano to polycrystalline structures, amorphous and other forms of microstructures that could not be produced by thermal means. These are soft ferromagnetic films that could become antiferromagnetic materials when combined as multilayer alloys. Intermediate composition of these alloy depositions are possible requiring good knowledge of electrochemistry of metallurgy, thermodynamic and kinetic reactions.

4:40 PM

Controlling Composition at the Individual FePt Nanoparticle Level: Chandan Srivastava[1]; David Nikles[1]; *Gregory Thompson*[1]; [1]University of Alabama

The self assembly of FePt nanoparticles is proposed as a candidate architecture for achieving ultra high magnetic storage. An outstanding issue is the compositional and size variation between individual nanoparticles. This research will demonstrate that a two-step nucleation process of Pt seeds with the subsequent heterogeneous nucleation of Fe from organo-metallic or salt precursors can significantly reduce the composition and size distribution between individual nanoparticles. This two-step process, and the narrowing of these distributions, has been demonstrated in two separate FePt synthesis techniques: the thermal decomposition of Fe(CO)5 or reduction of FeCl2 with Pt(acac) precursors. The results are explained in terms of a Monte Carol free energy simulation. Individual nanoparticle chemistry has been quantified using STEM-EDX analysis. The results demonstrate how mechanistic nucleation and growth control can be used to tune multi-component nanoparticles.

5:00 PM

Chemical Vapour Synthesis of Boron Modified Nanocrystalline Anatase Titania for Photocatalytic Applications: *Imteyaz Mohammad*[1]; Subramshu Bhattacharya[1]; Horst Hahn[2]; [1]IIT Madras; [2]Technische Universität Darmstadt

Titania is a popular wide bandgap semiconductor with high potential for diverse catalytic applications. Of the three well-known polymorphs of titania, anatase is the most effective catalyst for the degradation of many organic and inorganic pollutants. However, anatase is metastable and irreversibly transforms to rutile on heating. Further, the bandgap of anatase lies in the ultra-violet region, which reduces its efficiency under ambient light. Modification of nanocrystalline anatase with boron could result in improved high temperature stability and lower bandgap energy. In the present study, boron modified nanocrystalline anatase was synthesized in a single step by a chemical vapour synthesis (CVS) route. The powders were characterized by x-ray diffraction, nitrogen adsorption and Fourier transform infrared spectroscopy. Analysis revealed the presence of boron in BO_4 co-ordination in the titania structure. Studies on the photocatalytic degradation of rhodamine dye under UV radiation confirmed faster degradation in case of the boron modified powders.

5:20 PM Concluding Comments

Advances in Composite, Cellular and Natural Materials: Metal Matrix Composites

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee

Program Organizers: Yuyuan Zhao, The University of Liverpool; David Dunand, Northwestern University

Tuesday PM Room: 305
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Xiaodong Li, University of South Carolina; T. Venkatesh, Stony Brook University

2:00 PM

Fabrication of Al6061-SiC Composite by a Novel Semisolid Powder Processing: Yufeng Wu[1]; *Gap-Yong Kim*[1]; Iver Anderson[2]; Thomas Lograsso[2]; [1]Iowa State University; [2]Ames Laboratory of US DOE

Metal matrix composites (MMCs) offer advantages such as improved strength, stiffness and electrical properties to provide weight or performance benefits. In this study, we investigated fabrication of Al6061-SiC composite by particle-based semisolid processing (pSP), which combines the advantages of semisolid forming and powder metallurgy. High wt% SiC (>50%) particle reinforced Al6061 were fabricated by pSP. Effects of the processing parameters (SiC volume fraction and pressure) on the microstructures and mechanical properties of the composite were investigated. Composites with high wt% SiC were successfully formed. Observed microstructures indicate the SiC was uniformly distributed within the matrix. Microhardness test showed that the composite strength was significantly improved by the addition of SiC particles. The hardness increased as the SiC fraction and pressure increased. Test results show that the process has the potential to synthesize and fabricate final part simultaneously in MMCs at low cost and with high efficiency.

2:20 PM

Compressive Properties of Closed-Cell Aluminum Foams Reinforced with Fly Ash Particles: *Yong Liang Mu*[1]; Guang Chun Yao[1]; Hong Jie Luo[1]; [1]Northeasten University

The closed-cell aluminum foam reinforced by 1.5wt.% and 3.0wt.% fly ash particles were manufactured by molten body transitional foaming process. The quasi-static compression tests were conducted. Results show that the plateau stress of Al/Fly ash foams increases nearly linearly with relative density. Moreover, the addition of fly ash particles improves the plateau stress. Also, the energy absorption property of Al/Fly ash foams increase with relative density and fly ash content, which can be attributed to the reinforced cell walls by fly ash particles and the existence of sliding between fly ash and matrix.

2:40 PM

Fabrication of Carbon Nano-Fiber (CNF) Reinforced Aluminum Matrix Composites by Pressureless Infiltration: Effect of Aluminum Coatings on Infiltration Behavior: *Fumio Ogawa*[1]; Tatsuya Hirakawa[2]; Minoru Oda[1]; Chitoshi Masuda[3]; Toshiyuki Nishimura[4]; [1]Graduate School of Waseda University; [2]Waseda University; [3]Kagami Memorial Institute for Materials Science and Technology, Waseda University; [4]National Institute for Materials Science

Recently, CNF reinforced aluminum matrix composites are considered to be used as brake disc rotor of automobile or thermal management device due to their high specific strength, modulus and thermal conductivity. In general, CNF is not easily wetted by molten aluminum. So, since it is hard to infiltrate molten aluminum into CNF preform, fabrication of composites by casting is difficult. In this study, aluminum or its compounds are coated on CNF by chemical vapor deposition to enhance wetting. Pressureless infiltration of aluminum is studied and effects of aluminum coating and magnesium content in preform on infiltration behavior are investigated. Optimum condition for fabrication of composites is studied. Mechanical and physical constants such as elastic modulus, hardness, strength, and thermal conductivity are measured and compared with those of composites fabricated by powder metallurgy method.

3:00 PM

Un-Bundled Carbon Nanotubes Reinforced Titanium Composites via Powder Metallurgy Process: *Katsuyoshi Kondoh*[1]; Thotsaphon Threrujirapapong[1]; Hisashi Imai[1]; Junko Umeda[1]; Bunshi Fugetsu[2]; [1]Osaka University; [2]Hokkaido University

Zwitterionic surfactant solution process was applied to prepare titanium powders coated with un-bundled multi-wall carbon nanotubes (MWCNTs). They were consolidated by spark plasma sintering (SPS) and subsequently hot extrusion process. The microstructures and mechanical properties of titanium composites reinforced with CNTs were evaluated. The distribution of CNTs and in-situ formed titanium carbide (TiC) compounds during sintering was investigated by XRD, optical and scanning electron microscopy (SEM) equipped with EDS analyzer. The mechanical properties of TMC were significantly improved by the additive of CNTs. For example, when employing the pure titanium composite powder coated with CNTs of 0.35 mass%, the increase of tensile strength and yield stress of the extruded TMC was 157 MPa and 169 MPa, respectively, compared to those of extruded titanium materials with no CNT additive. Fractured surfaces showed uniform distribution of CNTs and TiC particles, being effective for the dispersion strengthening of the composites.

3:20 PM

Fabrication of High Strength Pure Ti Matrix Composite Reinforced with Carbon Black Particle via Wet Process: *Thotsaphon Threrujirapapong*[1]; Katsuyoshi Kondoh[2]; Hisashi Imai[2]; Junko Umeda[2]; Bunshi Fugetsu[3]; [1]Graduate School of Enginering, Osaka University; [2]Osaka University; [3]Hokkaido University

A titanium matrix composite reinforced with carbon black was prepared by spark plasma sintering (SPS) and hot extrusion. Carbon black particles were added for the in situ formation of TiC dispersoids during the SPS process. Sponge and fine Ti powders were coated with carbon black particles via a wet process using a zwitterionic solution containing carbon black spheres. The morphology and distribution of the in situ TiC phases were investigated using optical microscopy and SEM with an EDS analyzer. The mechanical properties of these composites were remarkably improved by adding a small amount of carbon black at 0.07 ~ 0.16 wt.%. The increases in the yield stress of the extruded sponge and fine Ti were 70.0 and 291 MPa, while the tensile strength increases were 67 and 231 MPa, respectively, compared to those of extruded pure Ti with no reinforcement. The advantages of the wet process are discussed in detail.

3:40 PM Break

4:00 PM
Load Partitioning in Al_2O_3-Al Composites with Three-dimensional Periodic Architecture: *Marcus Young*[1]; Ranjeet Rao[2]; Jon Almer[3]; Dean Haeffner[3]; Jennifer Lewis[2]; David Dunand[1]; [1]Department of Materials Science and Engineering, Northwestern University; [2]Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign; [3]Advanced Photon Source, Argonne National Laboratory

Interpenetrating Al_2O_3/Al composites were created by liquid-metal infiltration of 3-D periodic ceramic preforms with simple tetragonal and face-centered tetragonal symmetry produced by direct-write assembly. Volume-averaged lattice strains in the ceramic phase of the composite were measured by synchrotron X-ray diffraction for various levels of uniaxial compression. Load transfer is found to occur from the metal phase to the ceramic phase and is in general agreement with simple rule-of-mixture models and in better agreement with more complex, 3-D finite-element models that account for metal plasticity and details of the geometry of both phases. Spatially resolved diffraction measurements show variations in load transfer at two different positions within the composites. Lastly, the effects of Al_2O_3 symmetry on load partitioning in Al_2O_3/Al composites are discussed.

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Bulk Metallic Glass Composites: A New High-Performance Structural Material: *Douglas Hofmann*[1]; Maximilien Launey[2]; Robert Ritchie[3]; William Johnson[4]; [1]Liquidmetal Technologies; [2]Lawrence Berkeley National Laboratory; [3]University of California Berkeley; [4]California Institute of Technology

Bulk metallic glasses (BMGs) are well-known for having high strengths and nearly negligible ductility in unconfined loading conditions. However, when properly designed and processed, BMG matrix composites can have mechanically properties equally or surpassing the best crystalline materials. These composites exhibit >10% ductility in tension at yield strengths between 1-2 GPa, fracture toughness in excess of 150 MPa √m and endurance fatigue limits of ~30% of the tensile yield strength. In this talk, we will discuss recent collaborative efforts towards improving design, manufacturing, and mechanical properties of these new composites. This work will encompass alloy development, semi-solid processing, fracture toughness and fatigue measurements, novel manufacturing strategies, and the successful demonstration of complex net-shapes.

4:40 PM
Creep and In-situ TEM Investigations of Short Fiber Reinforced Metal Matrix Composites: *Deniz Kurumlu*[1]; Marcus Young[1]; Antonin Dlouhy[2]; Gunther Eggeler[1]; [1]Ruhr Universitaet; [2]Institute of Physical Metallurgy, Academy of Sciences of the Czech Republic

Short fiber reinforced metal matrix composites (MMCs), consisting of an AlZn11Mg0.2 alloy matrix reinforced with 15 vol. % Saffil fibers, were produced by squeeze casting. Miniature creep specimens were examined during creep under uniaxial tensile loading at temperatures ranging from 573 K to 648 K. The fiber reinforcement is found to decrease the creep rate and creep ductility and increase the creep strength. Moreover, the fiber reinforcement changes the characteristics of the stress and temperature dependence of the overall creep process. Microstructural processes, which control the creep behavior, were examined using TEM during uniaxial loading and during heating and cooling. A considerable increase in the dislocation density towards the fiber was observed in the crept material.

5:00 PM
Creep Behavior of Aluminum Based Nanocomposites Reinforced with Multi-Walled Carbon Nanotubes: *Hyunjoo Choi*[1]; Jaehyuck Shin[1]; Donghyun Bae[1]; [1]Yonsei University

Reinforcing effects of multi-walled carbon nanotubes (MWNTs) on the creep behavior of aluminum-based nanocomposites were investigated. The composites were produced by hot rolling of the ball-milled mixture of aluminum powders and MWNTs. During the specially controlled milling process, each of the MWNTs was uniformly dispersed and fully packed with aluminum atoms, providing the perfectly sticking interface between the MWNTs and the matrix. The creep behavior of the composites at high and low stress levels was examined in the temperature range from 150° to 250°. MWNTs can effectively block both thermal diffusion of aluminum atoms and dislocation movement; the composites exhibit significantly reduced thermal diffusivity and remarkably enhanced strength at elevated temperatures. The operative creep mechanism was also predicted based on the stress exponent and activation energy for creep and evaluated using transmission electron microscope (TEM) images after creep.

5:20 PM
Microstructural Evaluation of ZrC Reinforced Al–Cu Matrix Alloy Composites Fabricated by Mechanical Alloying and Vacuum Hot Pressing: *Hulya Kaftelen*[1]; Necip Unlu[1]; Mustafa Ovecoglu[1]; Hani Henein[2]; [1]Istanbul Technical University; [2]University of Alberta

In this work, Al–4wt.%Cu composites comprising two differently sized ZrC reinforcing particles (157 μm and 8 μm) were fabricated by high energy ball milling and vacuum hot pressing. Vacuum hot pressing experiments were carried out at 550oC under a pressure of 70 MPa on Al–4wt. %Cu composite powders ball milled for 3h. Effects of reinforcement size on the microstructural and physical properties of the composites were investigated by X–ray diffraction (XRD), scanning electron microscopy (SEM) with energy–dispersive spectrometry (EDS) techniques. Following vacuum hot pressing, approximately relative density values of 98 % were achieved for all hot pressed Al–4wt.%Cu composites. Highest microhardness values were measured for the hot pressed Al–4wt.%Cu composites containing the smallest sized ZrC reinforcing particles (8 m). On the basis of analysis, in addition to the Al, ZrC and Al2Cu phase reflections, a tetragonal Al7Cu2Fe phase was formed due to iron contamination from balls and/or vial in the metal matrix composite during MA process.

Alumina and Bauxite: Process Improvements and Experiences - Red Side I

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee
Program Organizers: Carlos Suarez, Hatch Associates Inc; Everett Phillips, Nalco Company

Tuesday PM Room: 611
February 16, 2010 Location: Washington State Convention Center

Session Chair: Austin Mooney, Sherwin Alumina

2:00 PM Introductory Comments

2:10 PM
Study on Ore Dressing and Characterization of Different Granulometric Fractions that Compound Bauxite from Pará/Brazil: *Fernanda Silva*[1]; Rachel Santos[1]; João Sampaio[2]; Francisco Garrido[3]; Marta Medeiros[3]; [1]IQ/UFRJ - CETEM; [2]CETEM; [3]IQ/UFRJ

Bauxite from Northeast of Pará/Brazil was ore dressed and characterized by XRD, XRF, chemical and thermal analysis. XRD was performed to determine the mineral content. Such bauxite is essentially gibbsitic and has been associated with kaolinite, aluminum-goethite, goethite and iron and titanium compounds. XRF analysis was carried out in order to determine the sample's chemical composition and how its content varied after ore dressing. However, the chemical content of Al2O3available and SiO2reactive was determined by back titration and flame atomic absorption. The results found for the Bayer Process sample were 47.2% and 5.3%, respectively. Thermal analysis was performed as a supplemental technique to observe the bauxite decomposition in the mineralogical phases. Thus, based on stoichiometric relations of the bauxite components decomposition and the mass loss observed in the thermal analysis, it was possible to confirm the following phases: gibbsite, aluminum-goethite and kaolinite.

2:40 PM
Autoclave Desilication of Digested Bauxite Slurry in the Flashing Circuit: *Andrey Panov*[1]; Alexander Suss[1]; Irina Paromova[1]; Alexander Damaskin[1]; [1]RUSAL VAMI

Bauxite digestion conditions should provide not only the maximum alumina recovery from raw material into liquor, but also the required desilication rate to produce further alumina of high quality. Several desilication equipment circuits are common for alumina industry, namely low temperature predesilication of crude and digested slurry, Sumitimo process. These processes as a rule involve heavy process equipment. The proposed option of autoclave desilication in the

flashing circuit of the bauxite slurry allows implementation of the process at higher temperature enhancing chemical reactions of green liquor desilication. This stimulation gives an opportunity to reduce the retention time from 8-10 to 1-2 hours, facilitates reduction in the balanced silica content and increases alumina to silica ratio in the liquor up to 300.The mud produced after digestion and desilication has better sedimentation properties as compared to that obtained as a result of low temperature desilication.

3:10 PM

Bauxite Grinding Practices and Options: Anthony Filidore[1]; *John Hadaway*[1]; [1]FLSmidth Minerals

Grinding of bauxite as feed to alumina refineries is undertaken in a variety of grinding circuit types utilizing a range of mill types such as Rod, Rod/Ball and Ball Mills both in open circuit and closed circuit systems. This paper considers the selection criteria in terms of the objectives of the grinding process and reviews the range of options available particularly those used in recent installations. The advantages and disadvantages of these options are discussed. Important evaluation criteria such as product size distribution, specific power consumption and costs are considered, summarised and compared with the design objectives.

3:40 PM Break

4:00 PM

The Effect of Anatase and Lime on the Transformation of Sodalite to Cancrinite in Bayer Digestion at 250°C: Bingan Xu[1]; *Peter Smith*[1]; Christine Wingate[1]; Lynette De Silva[1]; [1]CSIRO Minerals

Sodalite (SOD) and cancrinite (CAN) are important desilication products in the refining of high silica bauxites by the Bayer process. SOD, usually formed at predesilication or low temperature digestion, is transformed to CAN under high temperature digestion. CAN provides opportunities to minimize soda loss and reduce both silica contamination of product and silicate scaling. This paper reports the influence of anatase and types of lime on the transformation. It was found that the rate of transformation is greatly increased by lime, but when anatase is also present, the rate is suppressed by a coating of sodium titanate on SOD. Reducing sodium titanate by forming calcium-containing titanates was critical for the transformation. The efficiency of calcium titanate formation depended on lime charge and type. Calcite as calcium source was more efficient than CaO, enhancing the formation of CAN and promoting the formation of more stable perovskite. The underlying mechanisms are discussed.

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Study on the Rheological Behavior of Crystallized and Crystallized - Amorphous Bauxites: *Carla Barbato*[1]; Silvia França[2]; Marcio Nele[3]; [1]UFRJ/CETEM; [2]Cetem; [3]EQ/UFRJ

The Bauxite of Northern Brazil is characterized by a geological profile with five different layers: nodular, crystallized nodular, crystallized, crystallized - amorphous and amorphous. The crystallized bauxite is used for alumina production by the Bayer process. Although the mining process is selective, this layer is removed with a certain amount of crystallized-amorphous bauxite. The ore-dressed bauxite pulp is transported through pipelines to the plant where alumina is produced. It is important to predict the pulp´s behavior during its transportation through the pipeline. It is necessary to study the rheology and understand their rheological properties in the pumping. Due the difference in the ore typology it is important to verify the influence of crystallized-amorphous bauxite in the rheological behavior of crystallized bauxite pulps, different concentrations of crystallized-amorphous and crystallized bauxite pulps were studied. It was verified that the increase on the crystallized-amorphous bauxite concentration resulted on viscosity increase and tixotropy decrease.

Aluminum Alloys: Fabrication, Characterization and Applications: Materials Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee
Program Organizers: Subodh Das, Phinix LLC; Steven Long, Kaiser Aluminum Corporation; Tongguang Zhai, University of Kentucky

Tuesday PM Room: 615
February 16, 2010 Location: Washington State Convention Center

Session Chair: Zhengdong Long, Kaiser Aluminum

2:00 PM

Structure Evolution and Recrystallization in 7xxx Series Al Alloys: *Jameson Root*[1]; David Field[1]; [1]Washington State University

Recrystallization of hot-rolled 7xxx series aluminum alloy plates can vary greatly due to grain size, texture, varying solute concentration, and second phase particle distribution. The purpose of this study is to observe recrystallization during hot-rolling of a 7xxx series alloy. Hot deformation experiments were performed at various temperatures and strain rates to simulate the hot rolling process. The specimens were subsequently annealed in salt baths for various times at given temperatures. The evolution of microstructure and recrystallization behavior of these specimens were observed with electron backscatter diffraction and optical microscopy. These data are used as a basis for developing a model that tracks recrystallization behavior during hot rolling.

2:20 PM

Quantification of Marine Aluminum Alloy Sensitization Based upon Thermal Loading: *William Golumbfskie*[1]; Catherine Wong[2]; [1]Naval Surface Warfare Center, Carderock Division; [2]NAVSEA

In recent years, structural aluminum alloys have become more popular in marine applications, with the shipbuilding industry looking to reduce weight. The 5xxx series is an ideal candidate, combining high specific strength, corrosion resistance, and weldability. Of particular concern is that it becomes sensitized in service. Aluminum becomes sensitized when beta phase (Al_3Mg_2) is precipitated at the grain boundaries, which may lead to stress corrosion cracking. The ASTM G67 mass loss test is being used to quantify the extent of sensitization as a function of different thermal loading conditions. Temperature recording devices have been installed on two ships to determine the amount of in-service thermal loading. The objective of this work is to interpret the sensitization and thermal loading data into a quantitative tool that can aid in determining the degree of sensitization for in-service 5xxx alloys and estimate the lifetime until the material has fully sensitized.

2:40 PM

Influence of Grain Boundary Sliding on the Ductility of Ultrafine-Grained Al: *Yonghao Zhao*[1]; John F. Bingert[2]; Ying Li[1]; Peiling Sun[3]; Xiaozhou Liao[4]; Yuntian Zhu[5]; Enrique Lavernia[1]; [1]University of California-Davis; [2]Los Alamos National Lab; [3]Feng Chia University; [4]The University of Sydney; [5]North Carolina State University

Early reports on nanostructured materials predicted that grain boundary (GB) sliding should enhance the ductility of nanostructured materials. However, review of the literature shows that direct evidence for this mechanism is still lacking. In this work, we report on post mortem analyses of tensile fractured specimens using atomic force microscopy (AFM) and scanning electron microscopy (SEM), as a means to provide direct evidence for the influence of GB sliding on ductility. Specifically, ultrafine grained (UFG) Al was prepared by equal-channel-angular pressing (ECAP) and annealed at 250°C for 20 min. Transmission electron microscopy (TEM) and electron back-scattering diffraction (EBSD) analyses indicate that annealing changes statistically stored dislocations to low-energy dislocation walls, without significantly affecting grain size or other microstructural parameters. Tensile testing shows an evidently enhanced ductility after annealing. AFM and SEM analyses indicate that the enhanced ductility stems from the much finer micro-deformation bands formed during extensive GB sliding.

3:00 PM
Transmission Electron Microscopic Investigation of Sensitized Al-5083: *Ramasis Goswami*[1]; George Spanos[2]; Peter Pao[2]; Ronald Holtz[2]; [1]SAIC/Naval Research Laboratory; [2]Naval Research Laboratory

Microstructure of Al-5083 (H-131) sensitized at 175°C for 1, 10, 25, 50, 100, 240, 500 and 1000 hours has been investigated using transmission electron microscopy (TEM) to study the evolution of ß phase (Al3Mg2) at grain boundaries and on existing particles. The ß precipitates formed at grain boundaries thicken faster than the rate estimated assuming volume diffusion controlled growth of planar interfaces. It is suggested that pipe diffusion through dislocations contributes to the enhanced growth rate of ß, consistent with TEM observations of a high dislocation density both in the aluminum matrix in the as-received material, and in the matrix surrounding the particles in the annealed material. The temporal distribution and the grain boundary coverage of ß will be discussed.

3:20 PM
Microstructure Evolution of Pre-Strained 3xxx Aluminum Alloys during Annealing: *Payman Babaghorbani*[1]; Nick Parson[2]; Mary Wells[3]; Warren Poole[1]; [1]The University of British Columbia; [2]Rio Tinto Alcan, Arvida Research and Development Centre; [3]University of Waterloo

Room temperature deformation is often the final stage in the processing route for 3xxx aluminum alloys which are used in heat exchanger applications and cooling systems. The deformation can be low (1-5%) for sizing applications or relatively large for example in the case of tube drawing. Often the parts are annealed either as a separate processing step or in conjunction with the brazing operation. The current study examines the annealing behavior in AA3xxx alloys for different levels of pre-strain, ranging from a few percent to 80% and with different homogenization treatments which modify the dispersoid distribution. The microstructures were characterized by optical microscopy and EBSD. Preliminary observations indicate that there is a strong interaction between the homogenization treatment, the level of pre-strain and the final microstructure and corresponding mechanical properties.

3:40 PM
Characterization of Hypereutectic Al-19%Si Alloy Solidification Process Using In-Situ Neutron Diffraction and Thermal Analysis Techniques: *Wojciech Kasprzak*[1]; Dimitry Sediako[2]; Mahi Sahoo[1]; Michael Walker[1]; [1]CANMET Materials Technology Laboratory; [2]National Research Council Canada

The objective of this publication is to evaluate the effect of alloying elements on structural and mechanical properties of hypereutectic Al-Si alloy used for light weight automotive applications. Simultaneous application of the in-situ thermal analysis and neutron diffraction (source: NRU nuclear reactor in Chalk River, ON) was used to evaluate the melt characteristics using the non-equilibrium solidification analysis. Some of the factors controlling microstructural modification are melt and pouring temperatures as well as solidification rate. The alloy "dissecting" approach was used to evaluate the contributions of individual alloying elements (Si, Cu, Mg and P) and to determine their effect on alloy and casting parameters including liquid metal processing as well as heat treatment. This outcome will help to better understand the solidification and heat treatment processing of the Al-Si hypereutectic alloys used for the HPDC and LPPM cast components.

4:00 PM Break

4:15 PM
Characterization of the Spacing Selection in AlCu Alloys: *Sebastian Gurevich*[1]; Morteza Amoorezaei[1]; Nikolas Provatas[1]; [1]McMaster University

We study the relationship between growth conditions and the resulting microstructure of cast alloys in industrially relevant conditions. We directionally solidify AlCu alloys under upward unsteady-state solidification conditions utilizing a range of cooling rates, and perform phase field simulations of directional solidification, including varying the growth conditions dynamically. To address the influence of the transient states and growth history on the resulting primary spacing we apply a power spectral analysis. We find that the resulting spacing is not uniquely determined by the cooling conditions but does fall within a specific, reproducible band. This consolidates the hypothesis of a band of accessible spacings for a given growth conditions rather than one unique spacing, consistent with previous works by Warren and Langer, Trivedi and Hun and Huang et al, as well as the notion of a predictable statistical scaling theory of primary spacing selection of Greenwood et al.

4:35 PM
Development of Al-Si Alloy by Optimizing Modifiers and Grain Refiners: *Saeed Farahany*[1]; Ali Ourdjini[1]; Mohd Hasbullah Idris[1]; [1]UTM University

We studied interaction of modifiers and grain refiners in hypoeutectic aluminum-silicon alloys, Al-6.5% Si- 0.3 Mg-Cu 2% to obtain optimum microstructure. Control of grain size and morphology of eutectic are major steps to achieve desired properties in these alloys. We used ingot pure aluminum; Al-Ti-B and Al-Sr master alloys and melt them in induction furnace. We used scanning electron microscope (SEM) and image analyzer software to study microstructure and measure grain size according to ASTM standard E112-88.Microstructural observations showed that by adding modifier, such as sodium, antimony and strontium to hypoeutectic aluminum-silicon alloys coarse flake of eutectic silicon converted to fine particles. Also titanium and boron created fine grain size that improves ductility. Optimum composition for sodium, strontium and antimony in hypoeutectic Al-Si alloys is 0.001% - 0.003%, 0.01%-0.015% and 0.10% -0.15% respectively. These elements can be compatible together or have reverse effects. Antimony is not compatible with other modifying elements

4:55 PM
Patterning Surface Precipitation in Al-Cu Alloys via Localized Loading: *Jack Franklin*[1]; Jennifer Lukes[1]; [1]University of Pennsylvania

A new method for patterning precipitates at the surface of aluminum rich copper alloys is investigated. In this method, micron-scale patterned loading devices are used to apply constant localized loading to a mechanically polished surface as the samples are cooled below the solvus line from solid solution. Precipitates grow preferentially underneath the loaded regions for slowly cooled samples but for quickly cooled samples precipitate growth is suppressed. Examples and causes for the observed precipitate patterns will be discussed.

5:15 PM
Refinement of Hypereutectic Al-Si Alloy by Ca3P2: Ying Zhang[1]; Wangxing Li[1]; Danqing Yi[2]; Zhiseng Ren[1]; Xianghui Cang[1]; *Jianhong Yang*[1]; [1]Zhengzhou Research Institute of CHALCO; [2]Central South University

Refinements of hypereutectic Al-Si alloy were studied by adding calcium phosphide, which were always carried out with phosphorus or phosphides with low melting points and so on, through optical microscopy, scanning electronic microscope and chemical analysis. The results showed that adding phosphide of alkaline earth metals with higher melting points, such as calcium phosphide, could replace phosphorus modifier used to modify silicon phases and decrease the releasing of poisonous gas at the same time. Orthogonal experiments gave the optimized operational parameter.

Aluminum Reduction Technology: Hall-Héroult Cell: Energy Conservation Through Cell Design and Process Improvements

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee
Program Organizers: Charles Mark Read, Bechtel Corporation; Gilles Dufour, Aluminerie de Deschambault

Tuesday PM Room: 608
February 16, 2010 Location: Washington State Convention Center

Session Chair: William Imrie, Bechtel Corporation

2:00 PM Introductory Comments

2:05 PM
Wettable Athodes: An Update: *Rudolf Pawlek*[1]; [1]TS+C

This overview covers the development of aluminium wettable cathodes for the primary aluminium industry in the period 2000 to 2009. It continues a review of TiB2-C/composites, including their physical and mechanical properties. This overview also includes the development of binders, the manufacture of the composites, their application on the cathode surface, and their resistance to sodium penetration into the cathode lining. Mathematical modelling has

been introduced for the drained slope, the cathode current distribution, the flow of anode gas bubbles and the heat balance. Practical tests involved not only laboratory and bench scales, but also use in big electrolysis cells operated at more than 160 kA. Aluminium wettable cathode coatings appear advantageous for future multi-cell designs and for new electrolysis cell designs rather than for revamping existing Hall-Héroult aluminium electrolysis cells.

2:30 PM

Wettability of Liquid Aluminum on Carbon/Graphite/TiB2 Composite Cathode Materials: *Jilai Xue*[1]; Xing Chen[1]; [1]University of Science and Technology Beijing

Wettability of liquid aluminum on the cathode is of great importance for metal stability and cathode service life in aluminum reduction cells. Cathode material for today's reduction cells is carbon with poor wettability by Al, while emerging cathode materials are the carbon/graphite/TiB2 composites with potential better Al-wettability and cell sustainability. In this paper, a modified sessile drop method was applied to determine the contact angles for various testing materials, in which a newly formed fresh metal drop came into contact with the testing materials when the temperature was reached. The wetting process was recorded against operating time from the beginning of the Al-cathode contact. The TiB2 addition is found to improve the Al-wettability. The graphite aggregates can make better Al-wettablity with a limited addition. The obtained information can be used in improve cathode stability and cell sustainability.

2:55 PM

Study on the New Reduction Technology for Energy Saving: *Fengqin Liu*[1]; Songqing Gu[1]; [1]Chalco

A new aluminum reduction technology for energy saving recently developed in China is outlined in this paper. It is feasible to reduce ACD and to save energy by changing the properties and structures of the cathodes and lining materials in the reduction cells, which leads to minimizing impacts of magnetic field in the cells on molten metal fluctuation. The industrial tests for the cells with new structure have been conducted recently in China. The test results show that by using the new type cells the reduction operation can be kept steady at lower ACD, while the energy efficiency will be increased. The characteristics, existing problems and future development of various types of the new energy saving cells are reviewed in this paper.

3:20 PM

Effect of Atmosphere-Changing Sintering on the Corrosion Resistance of 17Ni/(10nio-Nife$_2$O$_4$) Cermet Inert Anode: Liu Kai[1]; Tian Zhongliang[1]; Li Jie[1]; Lai Yanqing[1]; Zhang Hongliang[1]; *Lü Xiao-jun*[1]; [1]School of Metallurgical Science and Engineering, Central South University

A new method termed atmosphere-changing sintering was adopted to prepare 17Ni/(10NiO-NiFe$_2$O$_4$) cermet inert anode for aluminum reduction. Its corrosion resistance against Na_3AlF_6-Al_2O_3 melts was compared with that of anodes prepared by traditional method through electrolysis tests. The results revealed that the anodes prepared by atmosphere-changing sintering method had better corrosion resistance against the melts. The wear rate of the anodes decreased from 3.12 cm/y to 1.10 cm/y due to the application of atmosphere-changing sintering. According to the SEM pictures of anodes after electrolysis tests, a 200\μm corroded layer can be noted in the outer zone of anodes made by atmosphere-changing sintering method, which was 220\956m thinner than that of anodes fabricated by traditional method.

3:45 PM Break

3:55 PM

Liquidus Temperatures of Cryolite Melts with Low Cryolite Ratio: Alexei Apisarov[1]; Alexander Dedyukhin[1]; *Elena Nikolaeva*[1]; Pavel Tin'ghaev[1]; Olga Tkacheva[1]; Alexander Redkin[1]; Yurii Zaikov[1]; [1]Institute of High Temperature Electrochemistry

Multi-component fluoride mixtures are the only possible media for the development of new ecologically clean and energy saving technologies for aluminum production. The radical decrease of operating temperature is impossible in the frame of existing technology in which sodium cryolite is a basic component of electrolyte. In order to obtain essential decrease of the electrolyte liquidus temperature potassium cryolite can be used as a basic component of electrolyte. Sodium and lithium cryolite additions are necessary in this case. Liquidus temperatures for the ternary KF-LiF-AlF3 and quaternary KF-NaF-CaF2-AlF3 systems have been determined by means of cooling curves. There are two minimums in the quasi-binary system (KF-AlF3)-(LiF-AlF3) at CR 1,3 and 1,5. The calcium fluoride solubility in the KF-NaF-AlF3 system rises with cryolite ratio and NaF content.

4:20 PM

Industrial Test of Low-Voltage Energy-Saving Aluminum Reduction Technology: Li Jie[1]; *Lü Xiao-jun*[1]; Lai Yan-qing[1]; Xie Chang-chun[2]; Zhang Hong-liang[1]; Xiao Jin[1]; Ding Feng-qi[1]; Liu Shi-wen[3]; Guo Qi-feng[3]; Li Yun-long[3]; [1]School of Metallurgical Science and Engineering, Central South University; [2]Hunan Zhongda Yexiang Technology Co., Ltd; [3]Qiya Aluminum (Group) Co, Ltd

It is main way to save energy through reducing electrolysis temperature and/or cell-voltage for the present aluminum electrolysis industry. On 300kA pre-baked cells at Qiya aluminum smelter, low-temperature and low-voltage technology was achieved with the optimization of control algorithm, bath components and process parameter. The industrial tests results for about one year show that the electrolysis temperatures and cell-voltages are about 925°and 3.80V respectively for test cells, and decrease of 30° and 0.3V compared to the reference cells. The bath superheats for test cells range from 10° to 12°. The current efficiencies reach about 92%, which remain the same as those of the reference cells. The energy consumptions for test cells are 12300kWh/t•Al, and decrease of 900 kWh/t•Al compared to the potline cells.

4:45 PM

New Cathodes in Aluminum Reduction Cells: *Feng Naixiang*[1]; Tian Yingfu[2]; Peng Jianping[1]; Wang Yaowu[1]; Qi Xiquan[3]; Tu Ganfeng[3]; [1]Northeastern University; [2]Chongqing Tiantai Aluminum Industry Co., Ltd.; [3]Northeastern University Engineering and Research Institute Co. Ltd.

Three aluminum electrolysis cells with a novel uneven cathode bottom are being operated successfully in Chongqing Tiantai Aluminum Industry Co.., Ltd., China. More and more aluminum smelters are using the model cathode structure. In the past year the three 168 kA cells have produced steadily with low cell voltages of 3.70 V to 3.75 V, and the current efficiency (CE) has been improved by at least 1% in comparison with 127 traditional model cathode cells in the same potline. The technical characteristics of the cells are described in this paper and the fluctuation of metal pad in the cells is measured.

5:10 PM

Calculation of the Aluminum Flow Field at the Interface of Molten Aluminum and Electrolyte in the New Cathode Aluminum Cells: Jiang Yanli[1]; Peng Jianping[1]; *Feng Naixiang*[1]; Wang Yaowu[1]; Qi Xiquan[2]; [1]Northeastern University; [2]Northeastern University Engineering and Research Institute Co. Ltd.

The mathematical model of electromagnetic field and the aluminum flow field in the 168 kA conventional cells and the new model cathode cells were numerically calculated with the commercial software ANSYS. The calculated results show that the voltage drop in the electrolyte of the new model cathode cells was reduced by 0.38 V, which indicates that the new model cathode cells have the potential for significant energy savings. Furthermore, the velocity of liquid aluminum and molten electrolyte at the aluminum/electrolyte interface in the new model cathode cells is lower than that of the conventional cells.

5:30 PM Concluding Comments

Biological Materials Science: Mechanical Behavior of Biological Materials II: Hard Tissues and their Replacement Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: John Nychka, University of Alberta; Jamie Kruzic, Oregon State University; Mehmet Sarikaya, University of Washington; Amit Bandyopadhyay, Washington State University

Tuesday PM Room: 205
February 16, 2010 Location: Washington State Convention Center

Session Chair: To Be Announced

2:00 PM Invited
Structural Mechanisms for the Inelastic Deformation of Haversian Bone: *Rizhi Wang*[1]; Vincent Ebacher[1]; [1]University of British Columbia

Biological tissues such as bone and teeth are known for their well-controlled ultra-structure and extraordinary mechanical performance. Bone has high fracture resistance because of its capability to undergo significant inelastic deformation. Understanding the detailed process and structural mechanisms of inelastic deformation thus hold the key to bone mechanics. In this presentation, we will report our latest experimental progress on bone structure, deformation and fracture. The emphasis will be on the most well-known hierarchical structure in human cortical bone, the Haversian system or secondary osteon. It will be shown that the inelastic deformation happens through the multiple microcrack nucleation and propagation processes, which are obviously governed by the unique structure of the osteonal lamellae and the distribution of the Haversian systems within the cortical bone. Such a unique and stable microcracking process eliminates the detrimental stress concentration effect of the Haversian canals and makes Haversian bones highly resistant to catastrophic failure.

2:30 PM
The Mixed-Mode Fracture of Human Cortical Bone: *Elizabeth Zimmermann*[1]; Maximilien Launey[1]; Holly Barth[1]; Robert Ritchie[1]; [1]Lawrence Berkeley National Laboratory and The University of California at Berkeley

Previous studies on the toughness of human cortical bone have primarily been focused on mode-I (tensile-opening) loading; however, bones *in vivo* are invariably loaded multiaxially. Consequently, an understanding of mixed-mode fracture is necessary to determine whether a mode-I fracture-toughness test provides the appropriate information to accurately quantify fracture risk. We have found that, in a transversely-oriented crack, the competition in crack paths between the maximum mechanical driving force (applied by means of an asymmetric load) and "weakest" microstructural path surprisingly cause the mode-II (shear-loaded) toughness to be ~25% less than the mode-I toughness in human cortical bone. This implies that in the transverse (breaking) orientation, bones are easier to fracture in shear than in tension. This intriguing result is further investigated by characterization of the mixed-mode (crack-initiation) toughness in the longitudinal orientation, and specifically by analyzing how a growing mixed-mode crack interacts with the hierarchical microstructure of bone.

2:50 PM
Looking at the Effects of Radiation Doses on the Fracture Toughness of Human Cortical Bone: *Holly Barth*[1]; Alastair MacDowell[2]; Maximilien Launey[2]; Robert Ritchie[1]; [1]Lawrence Berkeley National Laboratory and University of California, Berkeley; [2]Lawrence Berkeley National Laboratory

This study is designed to show that both strength and toughness of human cortical bone deteriorates with an increase in exposure to radiation. Bone is a complex hierarchical composite structure of mineral and collagen. Indeed, the toughness of bone is a result of a multi-scale suite of potent extrinsic (shielding) mechanisms, coupled with an additional role of intrinsic toughening due to the significant "plasticity" in the material. Our three control groups contain one group with no radiation exposure, one group with clinically relevant exposure and a third group with high dose exposure. Specimens were fractured and the toughening mechanisms were identified via *in situ* Environmental Scanning Electron Microscope (ESEM) and synchrotron-based x-ray computed tomography (SRμCT). *In situ* ESEM mechanical testing allows visualizing the crack growth in real time and gaining information on the salient toughening mechanisms. The SRμCT allows for non-destructive three-dimensional imaging of the microstructure and crack path.

3:10 PM
Microscale Uniaxial Compression Testing of Bone Tissue Specimens: Katrina Altman[1]; Stacey Vansickle[1]; Elise Morgan[2]; *Katharine Flores*[1]; [1]The Ohio State University; [2]Boston University

Bone is an anisotropic, hierarchically structured material, and as a result, its mechanical behavior is highly statistical in nature. It has been shown for other engineering materials that uniaxial testing at the microscale enables characterization of individual microstructural components in an effort to understand their role in the macroscopic mechanical behavior. The application of such microscale compression testing to bone will permit modeling of the aggregate material to predict effects of age, disease, or injury on the mechanical properties. The present work analyzes the mechanical behavior of 20- and 30μm nominal diameter compression pillars prepared by a femtosecond laser micromachining technique. Pillars are selectively machined within regions of interest on the bone specimen surface (i.e. within osteons). Modulus, strength, and modes of deformation are compared as functions of specimen size and anatomic position. In addition, the effects of water loss on mechanical properties are studied using the microcompression technique.

3:30 PM
Nanomechanics of Tropocollagen and Hydroxyapatite Biomaterials with an Account of Collagen Mutations and Varied Hydroxyapatite Textures: *Devendra Dubey*[1]; Vikas Tomar[1]; [1]Purdue University

In hierarchical nanocomposite materials (eg. bone, nacre), interfacial interactions between the organic phase (eg. tropocollagen (TC)) and the mineral phase (eg. calcium hydroxyapatite (HAP)) as well as the structural effects arising due to the staggered arrangement, TC mutations, and varied HAP textures significantly affect the strength of such biomaterials. In the present investigation, different idealizations of TC-HAP composite biomaterial system under tensile and compressive loadings are analyzed using explicit three dimensional (3-D) molecular dynamics (MD) simulations to develop an understanding of these factors. Analyses show that maximizing the contact area between the TC and HAP phases result in higher interfacial strength as well as higher fracture strength. Analyses based on strength scaling as a function of structural hierarchy reveal that while peak strength follows a multiscaling relation, the fracture strength does not. The peak strain for failure was found to be independent of the level of structural hierarchy.

3:50 PM Break

4:00 PM Invited
Fracture Processes and Mechanisms of Crack Growth Resistance in Human Enamel: D Bajaj[1]; Dwayne Arola[1]; [1]University of Maryland Baltimore County

Enamel of the human tooth is the hardest and highly mineralized tissue of the body. Despite the high mineral content, recent experimental evaluations of the fracture behavior using incremental crack growth have shown that this tissue undergoes a substantial rise in toughness with crack extension. In fact, the crack growth toughness of enamel (1.13 MPam^(0.5)/mm to 3.93 MPam^(0.5)/mm) exceeds that of human bone and dentin, tissues with much lower mineral content. Optical observations indicate crack growth toughening in enamel is achieved by a host of mechanisms operating across a range of length scales, and that these mechanisms are most active within the inner enamel. The predominant mechanisms appear to be crack deflection and twist, crack-bridging by unbroken ligaments of prism bundles and organic tethers, and microcracking about the protein-rich prism boundaries. This presentation will review the fracture properties of human enamel and discuss the mechanisms responsible for its exceptional behavior.

4:30 PM
Interfacial Failure of Dentin Adhesively Bonded to Quartz-Fiber Reinforced Epoxy: *Renata Melo*[1]; Nima Rahbar[2]; Wole Soboyejo[1]; [1]Princeton University; [2]University of Massachusetts Dartmouth

The aim of this study is to measure the fracture toughness and investigate the mechanisms of failure at the interfaces dentin/resin cement/ quartz-fiber reinforced epoxy. Rectangular slices of dentin from the pulp chamber were acid etched and covered with bonding agent. The slices of dentin were sandwiched by

two half-circle quartz-fiber discs covered with bonding agent, and bonded with dental cement to make Brazil-nut sandwich specimens for interfacial toughness testing. The interfacial fracture surfaces were examined using SEM and EDX to determine the failure modes when loading angles changed. Based on SEM/EDX analyses of the two halves, the crack was initiated and continued at the resin cement/quartz fiber substrate for loading angles 1 through 10º, whereas for loading angle 15° the crack was initiated at the top of the hybrid layer and continued there for the most part. The dentin/cement appears to be tougher than the resin cement/quartz-fiber reinforced epoxy.

4:50 PM

Structure and Mechanical Properties of Cementum Biocomposites: *Hanson Fong*[1]; Mustafa Gungormus[1]; Biran Foster[1]; Martha Somerman[1]; Candan Tamerler[1]; Mehmet Sarikaya[1]; [1]University of Washington

Enamel, dentin and cementum are biocomposites with unique mechanical properties and hierarchical structures that, working together, make up a mechanically functional tooth. Focus of this study is to understand the structure and mechanical properties of cementum to provide a foundation for cementum regeneration for clinical applications. Teeth from normal mice and mice with mutation or deletion of 'ank' gene which produced excessive but functional cementum, revealed similar mechanical properties in cementum and dentin, as analyzed by nanoindentation. TEM analysis revealed, while similar in mineral crystal size and collagen matrix structure, a distinct orientation difference in mineralized collagen fibers between dentin and cementum, adding overall strength isotropy to the tooth, analgous to off-angle lay-up laminate composites. Current research effort is focusing on fabricating cementum-like layers with similar structural and mechanical properties using genetically engineered hydroxyapatite binding peptides (HABP) created in our lab. Supported by NIDCR/NIH DE015109/DE09532 and GEMSEC (NSF-MRSEC at UW).

5:10 PM

Fabrication and Mechanical Properties of Calcium Phosphate Cements (CPC) for Bone Substitution: *Jingtao Zhang*[1]; Franck Tancret[1]; Jean-Michel Bouler[1]; [1]Université de Nantes

Calcium phosphate cements have been used in medical and dental applications for many years. However, their low strength and their high brittleness prohibit their use in many stress-bearing locations, which would require an improvement in mechanical properties. The influence of microstructural parameters on the latter have nevertheless barely been investigated in a systematic manner. In this aim, apatite cements have been fabricated using powders based on α-TCP (α-tricalcium phosphate), and their mechanical properties have been measured (compressive strength, Young's modulus and fracture toughness), as a function of various parameters (particle size; liquid-to-powder ratio; amount and morphology of porosity, including macropores created by porogens). This should allow, in the end, to improve mechanical properties by controlling the microstructure, and to find a compromise between strength and biological behaviour.

5:30 PM

Tricalcium Phosphates with Srontium Oxide and Zinc Oxide Dopants for Resorbable Bone Grafts: *Johanna Feuerstein*[1]; Shashwat Banerjee[1]; Susmita Bose[1]; Amit Bandhyopadhyay[1]; [1]Washington State University

Tricalcium phosphate (TCP) ceramics have recently gained a lot of attention for their chemical similarity to bone. However, they show poor mechanical strength. The purpose of this research is to develop a ceramic material with proper strength degradation kinetics and biocompatibility to be used as bone implants. In this study, four different (TCP)-based compounds were studied: (i) 0.25 wt% ZnO, (ii) 1 wt% SrO, (iii) binary dopants with 0.25wt% ZnO and 1 wt% SrO, and (iv) undoped TCP Control. Following wet mixing and pressing, the compounds were sintered at 1250° C. Sintered densities for both undoped TCP and the binary composition were above 95% of theoretical density. All compositions exhibited strengths above 200 MPa before being placed in simulated body fluid (SBF). The binary dopant seems to be the most bioactive as it has the most HCA formation The presentation will discuss processing and characterization including in-vitro strength degradation analysis.

5:50 PM

Biomimetic Chitosan-Based Nanocomposite Scaffolds for Bone Tissue Engineering: *Wah Wah thein-Han*[1]; Devesh Misra[1]; [1]University of Louisiana

In the development of bone tissue engineering, three-dimensional biomimetic nanostructured composites composed of organic and inorganic materials are of significant interest to mimic natural bone. We describe here biodegradable chitosan-based nanocomposite scaffolds with improved mechanical, physico-chemical, and biological properties compared to pure chitosan scaffolds for bone tissue engineering. The influence of the properties of chitosan such as degree of deacetylation (DD) and molecular weight (MW) were examined. The nanocomposite scaffolds were characterized by desired porous structure. Furthermore, DD and MW had influence on physico-chemical properties of the scaffolds. Favorable biological response of pre-osteoblast (MC 3T3-E1) on nanocomposite scaffolds included improved cell adhesion, higher proliferation, and well spreading morphology in relation to pure chitosan scaffold. The study underscores chitosan-based nanocomposite as a potential scaffold material for bone regeneration.

Bulk Metallic Glasses VII: Alloy Development and Application II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee

Program Organizers: Peter Liaw, The University of Tennessee; Hahn Choo, The University of Tennessee; Yanfei Gao, The University of Tennessee; Gongyao Wang, University of Tennessee

Tuesday PM — Room: 213
February 16, 2010 — Location: Washington State Convention Center

Session Chairs: Katharine Flores, The Ohio State University; Jan Schroers, Yale University

2:00 PM Invited

Processing of Bulk Metallic Glass: *Jan Schroers*[1]; [1]Yale University

The sluggish crystallization kinetic of bulk metallic glass results in two fundamentally different processing opportunities. BMG can be directly cast. But even for BMGs with low critical cooling rates geometries with high aspect ratio are particularly challenging since during casting cooling and filling of the mold must occur simultaneously. This limits the complexity of the geometries that can be cast even when processing parameters are carefully balanced. Alternatively, BMG can be thermo plastically formed in the supercooled liquid region. In this case the required fast cooling and forming are decoupled. The BMG is formed in a high viscous state where it behaves very similar to plastics when compared by processing temperature and forming pressure. A measure for the formability of BMGs will be introduced. Processing potentials and challenges will be discussed and various examples will be given including blow-molding, miniature fabrication, and nano-patterning.

2:20 PM

Development of Ti-Based Bulk Glassy Matrix Composites with Excellent Mechanical Performance: *Jin Man Park*[1]; Norbert Mattern[1]; Ka Ram Lim[2]; Do Hyang Kim[2]; Jürgen Eckert[1]; [1]Leibniz Institute for Solid State and Materials Research Dresden; [2]Yonsei University

Recently, highly toughened glassy matrix composites with different length scale heterogeneity have been developed in Zr-, Ti-, La-based glassy alloys. These heterostructured composites exhibit improved macroscopic plasticity by controlling the shear band formation and preventing the rapid propagation of major shear bands. In this study, we developed ductile dendrite reinforced composites in the Ti-Zr-Be-Cu-Ni-(Nb, Ta, V) system. Although in-situ composites were successfully formed by optimizing the alloy composition and cooling rate, ductility does not necessarily occur. When the size, the distribution and elastic constants of the dendrites are properly controlled, i.e. coarsened dendrites with lower shear modulus homogeneously distributed in the glassy matrix, large plasticity can be obtained. By optimization of the microstructure, Ti-based bulk glassy matrix composites with excellent mechanical performance (high yield strength of ~1.7 GPa and large plasticity of ~20 %) could be achieved.

2:30 PM Invited
Towards a New Class of Biodegradable Implants: Mg-Based Glasses with No Hydrogen Evolution: *Jörg Löffler*[1]; Peter Uggowitzer[1]; Bruno Zberg[1]; [1]ETH Zurich

Magnesium alloys are being increasingly studied for biodegradable implant applications because they show good biocompatibility and a suitable degradation rate. However, unfavorable hydrogen evolution during degradation has so far prevented their use in larger implants. In this context, Mg-based glasses show great promise because their degradation characteristics may be tailored by large amounts of alloying elements integrated into the glass structure. Here I will report on the dramatic reduction or complete cessation of hydrogen evolution during the degradation of MgZnCa glasses. Above a particular Zn-alloying threshold, a Zn- and oxygen-rich passivating layer forms on the alloy surface which can be explained by a model based on the Pourbaix diagram of Zn in simulated body fluid. Animal studies confirm the absence of hydrogen evolution and reveal tissue compatibility as good as that observed for crystalline Mg alloys. Thus these MgZnCa glasses show great potential for a new generation of biodegradable implants.

2:50 PM
Solute Substitution Induced Changes in Structure and Nucleation in an Al-Based Metallic Glass: *Feng Yi*[1]; Paul Voyles[1]; Seth Imhoff[1]; John Perepezko[1]; [1]UW-Madison

Fluctuation microscopy measurements have shown that strong Al crystal-like nanoscale order is a potential nucleation site for primary crystallization in high Al-like content metallic glasses. We have recently found that substitution of 1% of Cu for Fe in $Al_{88}Y_7Fe_5$ causes a dramatic change in the nanocrystal density after primary crystallization, in the delay time for the onset of crystallization, and in the order measured by fluctuation microscopy. Variable resolution STEM fluctuation electron microscopy results, interpreted in the light of computer simulations of Al-based metallic glasses, will shed new light on the size and density of the nanoscale ordered regions in this system.

3:00 PM Invited
Formation and Characterization of Individual Metallic Glassy Nanowire: *Koji Nakayama*[1]; Yoshihiko Yokoyama[1]; Takahito Ono[1]; Mingwei Chen[1]; Kotone Akiyama[1]; Toshio Sakurai[1]; Akihisa Inoue[1]; [1]Tohoku University

Metallic glasses have exciting potential for structural, chemical, and magnetic applications with the sizes ranging from micrometer to centimeter, but the fabrication and characterization down to nanoscale remains an important challenge. Progress has been hindered by the lack of bottom-up methodologies to produce amorphous nanostructures. Recently, we show the self-organized amorphous nanowires that are formed on the fracture surfaces of bulk metallic glasses [Nakayama et al., Nano Lett. 8, 516-519 (2008)]. However, it is difficult to control their morphologies because they were created by instantaneous fracture processes. Here we first report the controlled formation and mechanical characterization of individual amorphous nanowires. We find that they have a high strength with the excellent flexibility where the elastic modulus is much smaller than that of the bulk owing to the hyper-excess free volume in nanowire.

3:20 PM Invited
Role of Ductile β-Phase Dendrite in Optimizing Mechanical Properties of Ti-Based Bulk Metallic Glass Composites: Ka Ram Lim[1]; Jin Man Park[1]; Won Tae Kim[2]; *Do Hyang Kim*[1]; [1]Yonsei University; [2]Cheongju University

Recently, highly toughened bulk metallic glass (BMG) composites with mechanical properties comparable to high performance crystalline alloys have been reported in Zr-based system. Ductile-phase-reinforced BMG composites show enhanced global plasticity and more graceful failure since soft crystalline inclusions stabilize the glass against the catastrophic failure associated with unlimited extension of a shear band. In the present study, role of ductile β phase dendrite in optimizing the mechanical properties of Ti-rich Ti-Zr-Be-Cu-Ni-Nb BMG composites. Metastable equilibrium condition for β phase and glass matrix is established, therefore, the combination of strength and plasticity is optimized by tailoring relative volume fraction and morphology of β phase and glass matrix. It is considered that the presence of Nb assists the enhancement of glass forming ability as well as plasticity of the composites. In addition, possibility for ductile phase reinforced Be-free Ti-based bulk metallic glass composites will be discussed.

3:40 PM Break

3:50 PM Invited
Ductile Hypoeutectic Zr-Cu-Al and Zr-Ni-Cu-Al Bulk Glassy Alloys: *Y. Yokoyama*[1]; K. Fujita[2]; T. Yamasaki[3]; A. Yavari[4]; P. Liaw[5]; A. Inoue[1]; [1]Institute for Materials Research; [2]Department of Mechanical Engineering; [3]School of Engineering; [4]LTPcSIMAP-CNRS; [5]The University of Tennessee

Ductility of metallic glasses is probably originated from the flexibility of the metallic bond and open volume in random structures. The glass-transition temperature (Tg) has large positive correlation coefficients against Young's modulus (E) and Vickers hardness (HV). Ultimately, we found that only the Zr-enriched hypoeutectic composition exhibits low values of Tg and E and a high value of Poisson's ratio. Especially, hypoeutectic Zr-Cu-Al bulk glassy alloys exhibit high-fracture toughness equivalent to tool steels, and also do not show the remarkable embrittlement after annealing below Tg. In this presentation, we will talk about ductile mechanical properties of cast glassy ternary Zr-Cu-Al and quaternary Zr-Ni-Cu-Al alloys in hypoeutectic composition. Especially, the hypoeutectic Zr70Ni16Cu6Al8 bulk glassy alloy with extremely a low Young's modulus (70 GPa) and high Poisson's ratio (0.39) reveals the distinct tensile plastic elongation at room temperature, and the plastic strain depends on the strain rate.

4:10 PM
On Interfacial Bonding in Mg-Cu-Gd Metallic Glass during Spark Plasma Sintering Processing: *Baolong Zheng*[1]; Troy Topping[1]; Yizhang Zhou[1]; Chi Y.A. Tsao[2]; Enrique Lavernia[1]; [1]University of California, Davis; [2]National Cheng Kung University

Metallic glasses (MGs) show superplastic behavior in the super-cooled liquid region. This provides a pathway for MG powder consolidation via powder metallurgy processes. The nature of the interface between particles critically influences the mechanical properties of consolidated bulk MGs, whereas the low atomic diffusivity, characteristic of MG, hinders diffusion bonding. In this work, Mg-Cu-Gd amorphous powder was consolidated by Spark Plasma Sintering (SPS), while cast Mg-Cu-Gd rods were used to simulate conditions that are analogous to those present during powder sintering. The SPS studies provided insight into the phenomena of atomic diffusion and interfacial bonding during MG powder consolidation. The microstructural evolution of the bond interface of Mg-Cu-Gd powders and bulk materials was investigated as a function of processing parameters using SEM, XRD, DSC, and TEM. In addition, interfacial bond formation, mechanical response and the underlying mechanisms are discussed in an effort to provide insight into fundamental phenomena in MGs.

4:20 PM
Effect of Heat Treatment at Semisolid Region on Zr-Based Metallic Glass Matrix Composites: *Takuya Tamura*[1]; Advenit Makaya[1]; Kenji Miwa[1]; [1]National Institute of Advanced Industrial Science and Technology (AIST)

It was reported that micrometer-sized ductile crystalline phases can improve the ductility of Zr-based bulk metallic glasses. However, it is known to be difficult that the sample, for which the crystal particles are dispersed into the metallic glass phase, is prepared by Cu mold casting, because the cooling rate is different between the outside and the inside. The present authors reported that heat treatment at semisolid region is effective in forming uniform dispersed crystalline particles. Thus, this study aims to investigate effect of heat treatment at semisolid region on Zr-based metallic glass matrix composites.

4:30 PM Invited
Improving the Deformation Ability of Bulk Metallic Glasses by Different Approaches: *Ke-Fu Yao*[1]; Sheng-Bao Qiu[1]; Yang Li[1]; Hong-Yu Ding[1]; [1]Tsinghua University

Bulk metallic glasses (BMGs) have attracted tremendous attentions due to their excellent properties and potential applications. But lack of whole plasticity is a road block which needs to be overcome before their real application, especially in structural application. Then developing possible way for improving the plasticity of BMGs is important and meaningful. Here, we report that the deformation ability of BMGs can be significantly improved by both outside treatment and inside structural relaxation. It has been found that when the surface of a BMG sample is coated with a thin crystalline metallic layer its global plasticity can be effectively enhanced. On the other hand, the global plasticity of BMGs can also been increased greatly by treating them with high-density pulsing current, which could result in obvious structural relaxation

of the glassy alloys. The present results indicate that the global plasticity of BMGs can be enhanced by different ways.

4:50 PM
Thermodynamic Optimization of the Cu-Zr-Ag System and Its Applications to Amorphous Alloy Development: *Dae Hoon Kang*[1]; In-Ho Jung[1]; [1]McGill University

Recently a great amount of research has been carried out to understand the characteristics of the Cu-Zr-Ag amorphous alloys. However, the thermodynamics and phase diagram information of the system is still poorly investigated. In the present study, all available thermodynamic and phase diagram data of ternary Cu-Zr-Ag system as well as its sub-binary systems have been critically evaluated and optimized to obtain one set of model parameters which can reproduce all the reliable experimental data. The ternary phase diagram of the Cu-Zr-Ag was properly calculated for the first time. In particular, the existence of wide stable liquid miscibility gap was predicted in the ternary system. The roles of the metastable liquid miscibility gap and spinodal decomposition to the Cu-Zr-Ag amorphous alloys with nano Ag particles will be discussed.

5:00 PM Invited
Study of Microscopic Deformation Behaviors of Bulk Metallic-Glasses: *Yong Yang*[1]; J. Lu[1]; J.C. Ye[1]; [1]The Hong Kong Polytechnic University

In this talk, we present a summary of our recent experimental findings in the deformation behaviors of bulk metallic-glasses (BMGs) at the microscopic scale, which comprise the results from the micro-compression, micro-bending and two-dimensional nanoindentation at room temperature. The outcome of the micromechanical study of BMGs reveals the possible intrinsic mechanisms of shear-band nucleation, propagation and ductile-to-brittle transition in the metallic amorphous structures.

5:20 PM Invited
Cooling Process and Cast Structure of Zr-Al-Ni-Cu-Based Bmgs Produced in Various Atmospheres: *Junji Saida*[1]; Albertus Setyawan[1]; Hidemi Kato[1]; Mitsuhide Matsushita[2]; Akihisa Inoue[1]; [1]Tohoku University; [2]JEOL. Co., Ltd

A different dependence of apparent glass-forming ability on casting atmosphere pressure is observed in a Zr-Al-Ni-Cu-Pd alloy system. Results of a measurement of alloy temperature during casting indicate an enhancement of the cooling effect in the low-temperature region of undercooled-liquid in the high chamber pressure. However, it has not a significant difference in the high-temperature region against a chamber pressure. The cooling mechanism may change from a direct heat transfer between the cast alloy and mold in the high-temperature region into that via cavity between them in the low-temperature region. We investigate that cooling is significant in the high chamber pressure due to the existence of gas in the cavity. Such a control of cooling process by a chamber pressure brings new BMGs formation by the suppression of the metastable phase precipitation. The method is recognized as a new and simple technique for the fabrication and structure control of BMGs.

5:40 PM Invited
Recent Progress in High-Entropy Alloys: Jien-Wei Yeh[1]; *Ming-Hung Tsai*[1]; [1]National Tsing Hua University

It has been considered that alloys consisting of a greater number of principal elements will form complicated and brittle microstructures. Hence research regarding such alloys has received little attention. To overcome this misunderstanding high-entropy alloys (HEAs) have been proposed and explored since 1995. With the high mixing entropy HEAs tend to have simplified microstructures with solid-solution phases. This new alloy concept generates numerous alloys and activates a new research area beyond traditional alloys. In addition to high entropy effect, sluggish diffusion, severe lattice distortion, and cocktail effects are significant in affecting the structures and properties of HEAs. HEAs have been found to possess a wide spectrum of microstructures and properties, and hence provide a number of promising applications. This presentation reviews the recent progress in HEAs and also forecasts the future trend.

Cast Shop for Aluminum Production: Furnace Technology and Melt Handling

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee
Program Organizers: John Grandfield, Grandfield Technology Pty Ltd; Pierre Le Brun, Alcan Voreppe Research Center

Tuesday PM Room: 609
February 16, 2010 Location: Washington State Convention Center

Session Chair: Peter Whiteley, Munimula

2:00 PM
Influence of Heating Technology on Melt Quality in Ladles for Road Transportation of Liquid Aluminum Casting Alloys during Holding: *Bernd Prillhofer*[1]; Jens Knaack[1]; [1]AMAG Casting GmbH

Due to the increasing demand for liquid metal deliveries from refiners to shape casting foundries, melt quality topics, which are well known from the aluminum wrought alloy sector, are becoming more and more important. Long distance transportation of liquid metal alloys requires superheating to compensate temperature loss and to avoid any solidification. After arrival, the transport ladles sometimes replace holding furnaces or have to wait for metal transfer into the holder. Therefore the liquid metal must be heated at destination. Foundry managers can choose between gas burners and ceramic immersion heaters. So far, there hasn't been a detailed scientific investigation of the influence of both heating technologies on melt quality criteria. This paper examines the influence of heating technologies at constant holding temperature on inclusion and hydrogen content, density index, as well as chemical composition. The experimental work has been carried out on the alloy AlSi7Mg and AlSi9Cu3(Fe).

2:25 PM
Degreasing of Aluminium Turnings and Implications for Solid-State Recycling: Jirang Cui[1]; *Anne Kvithyld*[2]; Hans Roven[1]; [1]Norwegian University of Science and Technology; [2]SINTEF Materials and Chemistry

With the global warming being of concern, recycling of aluminium by new solid-state recycling techniques instead of conventional remelting and subsequent refining processing has been taken into account. Solid-state recycling processes such as extrusion and compaction at room or moderate temperature can result in significant energy savings. However, as aluminium scrap is normally contaminated by oil and/or organic coatings, decoating of scrap is an essential step for the solid-state recycling process. In the present study, degreasing of aluminium turnings with or without cold compaction was investigated by thermal methods and chemical treatment. Thermo-gravimetric furnace coupled with mass spectroscopy was utilised to understand the decomposing of machine oil on aluminium turnings in oxidising atmospheres. For further comparison, degreasing of turnings by acetone followed by solid-state processing was also studied in laboratory. The results may be used to choose a suitable pre-treatment method for solid state recycling of aluminium scrap.

2:50 PM
Retrofitting Aluminum Melting Furnaces: *Tom Schmidt*[1]; [1]Otto Junker

The average service life of aluminum melting and holding furnaces is several decennia. As a result of that, many aluminium casthouses operate with equipment of an older generation. The drawback of this is that these production tools not always meet the current standards on the fields of: Emissions, machine safety, fuel economics, Ergonomics, Process control. Unless forced by authorities or unions, it is not an easy decision if a modernization of an old furnace makes sense. Cost of a complete modernization, combined with new refractory lining takes almost the same investment as installing a new furnace. This paper will address this subject in order to make a well balanced decision on whether modernization of aluminum melting furnace is a desired option.

3:15 PM
Establishing Operational Parameters of AL-EMS Using Numerical Simulations to Promote Energy Efficiency during Final Heating in Aluminum Furnaces: *Robert Stal*[1]; Ulf Sand[1]; Olof Hjortstam[1]; [1]ABB AB

In aluminum melting and holding furnaces, electromagnetic stirring (AL-EMS) is a well established technology used to achieve enhanced thermal and chemical homogeneity, reduced cycle time, reduced energy consumption and reduced dross generation. In the present work, on site measurements and numerical simulations using a CFD-model (Computational Fluid Dynamics) have been conducted for an installed reverberatory melting furnace operating with ABB AL-EMS. Simulated and measured values of the time needed for melt temperature homogenization (homogenization time) are compared and good agreement has been found. Using the CFD-model, a parametric study is conducted for the relation between the strength of the AL-EMS and the energy efficiency in the furnace. The study clearly illustrates how the use of AL-EMS drastically reduces the homogenization time and improves the heat pick-up. A discussion of how the CFD-model can be used to optimize the strength of the AL-EMS for a given application is also presented.

3:40 PM
Energy Efficiency in Casthouse Furnaces: *Robert Voyer*[1]; Francis Caron[2]; [1]Hatch; [2]Alcoa

More than ever, the high cost of fuels and the need to reduce carbon emissions are forcing changes in casthouse equipment choices and shop practices. Some smelters have already started moving in this direction and have set in place energy tracking programs, while others are considering making these changes. Before implementing any project for reducing energy consumption, there is a need to assess the actual state of consumption, rate it with respect to technically achievable targets, and monitor the consumption in order to have a record of improvements. Continued monitoring is also essential in order to maintain the new standards.This presentation provides an overview of the status of energy consumption in typical casthouse furnaces. It quantifies the potential for reducing fuel consumption, as well as reducing carbon emissions, and proposes a cost efficient solution for reducing the fuel consumption and carbon emissions.

4:05 PM
Implementation of a Global Casthouse Furnace Energy Efficiency Program at Rio Tinto Alcan: *Mathieu Roy*[1]; Vincent Goutiere[1]; Claude Dupuis[1]; [1]Rio Tinto Alcan

Over the past several years, energy has been subjected to variable but sustained price increase. During the same time, climate change awareness has become more and more a concern for our society with the result that energy efficiency is now an environmental as well as an economic priority for all businesses. In the context of sustainable development, energy efficiency has always been a strong priority at Rio Tinto Alcan. In 2005, a global casthouse energy efficiency program was initiated to significantly reduce the consumption in all furnaces and put in place the means to maintain and improve on a continuous basis. This paper describes the key elements of this program and its successful implementation through practical examples of key operating practices and process parameters.

4:30 PM
Crucible Transfer by Siphoning: A Review of the Benefits and the Latest Technology: Jerry Locatelli[1]; Guangwei Liu[2]; *Andrew North*[2]; [1]Millennium Metals Pty Ltd; [2]Major Furnace Australia Pty Ltd

Siphoning has long been recognised as a method of transferring molten aluminium from reduction crucibles to Casthouse holding furnaces which generates the least amount of dross. This paper reviews some data on dross generation and melt loss, which compares the transfer of metal from crucibles by traditional cascade pouring with that of siphoning. The substantial difference between the two techniques is highlighted when the melt loss is viewed in terms of the amount of energy involved in producing the lost aluminium. It also becomes clear that the change from cascade pouring to siphoning represents the largest single energy reduction initiative which can be made in the Casthouse. This paper also presents examples of the latest automated siphon transfer systems which are finding application in installations ranging from the largest smelters to the smallest foundries.

4:55 PM
Heating and Melting of Single Al Ingots in an Aluminium Melting Furnace: *Jørgen Furu*[1]; Andreas Buchholz[2]; Trond Harald Bergstrøm[3]; Knut Marthinsen[1]; [1]NTNU; [2]Hydro Aluminium Deutschland GmbH; [3]SINTEF Materials and Chemistry

The efficiency of aluminium recycling and remelting processes is becoming increasingly important due to the tremendous growth of energy costs in recent years. Modeling the heating and melting behaviour of aluminium can help to better understand the interaction of different heat transfer mechanisms and the limiting factors for the heat exchange in a conventional reverberatory furnace. In the present work heating and melting experiments were carried out in a 500 kg test furnace with a conventional air-fuel burner. Small blocks of aluminium were equipped with thermocouples and insulated on all sides but one to approximate 1-dimensional heat transfer. Numerical heat transfer calculations were compared with the experimental results along with CFD modeling to investigate the contribution of various parameters to the heat transfer into the aluminium ingot.

5:20 PM
Optimised Re-Melting by the Use of Low-Temperature Oxyfuel at Hydro Aluminium's Primary Aluminium Casthouse, Övre Årdal, Norway: *Henrik Gripenberg*[1]; Ken Torvanger[2]; Johannes Lodin[1]; [1]Linde Gases Division; [2]Hydro Aluminium

Hydro Aluminium Årdal produces primary foundry alloys of mainly AA4000 series. The capacity is 130 000 tonne per year. The four furnaces at the cast house are charged with a mix of pot-room metal and commodity metal. In 2007 the Söderberg pot-room was closed and the re-melting of solid metal had to be increased by 60% to keep up the production from the cast house. This meant that the fraction of solid metal in the casting furnace batch had to be increased. The goal had to be reached using the same furnaces and casting equipment. Therefore the furnaces were converted from air-fuel to Low-temperature Oxyfuel burners from Linde. The paper will discuss how the melting process was optimized. Further how the energy efficiency depend on the fraction of solid metal in the batch and results of productivity, dross, and economy. The Low-temperature Oxyfuel technology will be described.

Characterization of Minerals, Metals and Materials: Characterization of Grain Size, Morphology, Transmittance, and Tomography

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee
Program Organizers: Ann Hagni, Geoscience Consultant; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF; Jiann-Yang Hwang, Michigan Technological University

Tuesday PM Room: 307
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Shadia Ikhmayies, University of Jordan; Mingdong Cai, Exova

2:30 PM Introductory Comments

2:35 PM
A Statistical Study of Grain Size, Grain Orientation, and Grain Boundary Misorientation Effects on Deformation Twinning: *Rodney McCabe*[1]; Irene Beyerlein[1]; Laurent Capolungo[2]; Peter Marshall[2]; Carlos Tome[1]; [1]Los Alamos National Laboratory; [2]Georgia Tech

A statistical analysis is carried out to examine correlations between deformation twinning and grain size, grain orientation, and grain boundary misorientation. The analysis uses large data sets generated using electron backscatter diffraction (EBSD) of magnesium, zirconium, and uranium. Similarities and differences between metals of the same crystal structure (hcp versus hcp) and the different crystal structures (hcp versus orthorhombic) are examined.

3:00 PM

An Automated Approach for Prior Austenite Grain Size Measurement by EBSD: *Ning Ma*[1]; Russell Mueller[1]; Timothy Anderson[2]; Raghavan Ayer[1]; [1]Corporate Strategic Research, ExxonMobil Research and Engineering Company; [2]ExxonMobil Upstream Research Company

Although prior austenite grain size has significant influence on the mechanical properties of steels, its determination has historically been a challenge. The present work describes the development of a robust and computationally efficient approach to determine prior austenite grain size from EBSD patterns in steels. The method is based on the crystallographic orientation relationships between the elevated temperature face centered cubic austenite and the body centered transformation products that form upon cooling to room temperature. The details of the procedure, such as the EBSD step width optimization and noise reduction, will be emphasized. Since the approach relies on the crystallography of the phase transformation, it is independent of the process history of the steels and it can be applied to a wide range of steels, including the grain size determination in heat affected zones in weldments.

3:25 PM

Linear Measures for Estimating Grain Growth Rates: *Martin Glicksman*[1]; Paulo Rios[2]; Daniel Lewis[3]; [1]University of Florida; [2]Universidade Federal Fluminense; [3]Rensselaer Polytechnic Institute

Average caliper, C, introduced by Minkowski in 1903, was generalized for polyhedral grains by Cahn and DeHoff in 1967. Recently, mean width, L, a linear measure applicable to non-convex objects, was introduced by MacPherson and Srolovitz, 2007, and by Le and Duc, 2009. These measures correlate geometric properties of polyhedral grains, including their areas, volumes, and curvatures, and permit prediction of curvature-mediated growth rates. Linear measures provide connections among growth rate, grain size, topological class, and shape. C and L for polyhedral grains were calculated using an exact topological representation of space-filling grains. Calculations confirmed by independent Surface Evolver computations yield relations among C and L, the geometric properties, and the growth kinetics of polycrystals during normal grain growth. In addition, easily applied formulae were found to estimate linear measures accurately, knowing the number of grain faces, plus any metrically-based measure such as grain size, volume, or grain boundary area.

3:50 PM

Serial Sectioning, X-Ray Tomography, and EBSD Analyses of Martensitic Alloys: *George Spanos*[1]; David Rowenhorst[1]; Richard Fonda[1]; Keith Knipling[1]; Richard Everett[1]; Greg Olson[2]; Stephanie Chan[2]; [1]Naval Research Laboratory; [2]Northwestern University

This talk presents investigations of microstructure and damage/defects in two alloy systems: (1) a commercial 4330 steel, and (2) model Fe - 20wt%Ni - 5-6wt% Mn alloys. A combination of serial sectioning and X-ray tomography were employed to reconstruct and study in three dimensions (3D) the inclusions, cracks, and voids (and their interrelationship) in compact tension specimens of a Ti-modified 4330 steel. Emphasis will be placed on the methodology employed, including a comparison of the 3D data between the two techniques (serial sectioning and X-ray tomography). A brief discussion will also be presented on how these experimental results are used by our collaborators for validation and input in 3D multiscale fracture models. Secondly, Electron Backscatter Diffraction (EBSD) analyses of martensite in Fe - 20wt%Ni - 5-6wt% Mn alloys will be presented, along with a discussion of possible avenues for examining these structures in three dimensions.

4:15 PM

Computed Tomography of Titanium Friction Stir Welds: *Jennifer Wolk*[1]; Richard Everett[2]; Lourdes Salamanca-Riba[3]; [1]Naval Surface Warfare Center; [2]Naval Research Laboratory; [3]University of Maryland

This work focuses on using x-ray computed tomography to assist in understanding defect formation and material flow in Ti-5111 friction stir welds. Tomographic techniques have been previously utilized to determine void geometries in ferrous and non-ferrous materials. A transverse cross-section of friction stir welded Ti-5111 containing defects was scanned using a Skyscan tomography system. Volume rendered images of the reconstructed data were revealed the internal void structures. This analysis showed a distinct void substructure on an advancing side defect. The substructure shows material bands that are unrelated to the friction stir welding tool design. The peak-to-peak distance appears to be approximately same as the forward advance per revolution. Analysis of the void formation at the bottom of the stir zone shows a complex substructure indicative of wormhole formation and material consolidation. The x-computed tomography is correlated to transverse electron backscatter diffraction scans and selected transmission electron microscopy analysis.

4:40 PM

Aberration-Corrected Vector Field Electron Tomography of Magnetic Nano-Structures: Charudatta Phatak[1]; Emma Humphrey[1]; Amanda Petford-Long[2]; *Marc De Graef*[1]; [1]Carnegie Mellon Unversity; [2]Argonne National Laboratory

Nanoscale magnets are used for a variety of applications, including spintronic devices and sensors, magnetic memory applications, and biomedical applications. To improve the functionality of these devices, it is necessary to characterize not only the particle/layer shapes and relative locations, but also the magnetostatic interactions between them. Therefore, the magnetic field distribution in and around the nano-scale magnets must be determined quantitatively. The electron-optical phase shift of electrons in a transmission electron microscope (TEM) can be expressed in terms of tomographic projections which, in combination with vector field electron tomography, can be used to reconstruct the 3D magnetic vector potential as well as the 3D magnetic induction in and around the magnetic particles. Results obtained for patterned magnetic structures will be demonstrated. The improvement due to aberration correction in the TEM, along with the development of an iterative technique for 3D reconstructions to minimize reconstruction errors will also be discussed.

5:05 PM

Using Transmittance Measurements to Investigate the Interdiffusion through the SnO2/CdS and CdS/CdTe interfaces in SnO2/CdS/CdTe Solar Cells: *Shadia Ikhmayies*[1]; Riyad Ahmad-Bitar[1]; [1]University of Jordan

The SnO2/CdS/CdTe thin film solar cells were fabricated on glass substrates by using the spray pyrolysis (SP) technique for the deposition of the SnO2 and CdS layers and vacuum evaporation for the CdTe layer. The transmittance curves were used to investigate the interdiffusion in the two interfacial regions SnO2/CdS and CdS/CdTe by taking the first derivative of the absorbance as a function of wavelength. The appearance of new bandgaps around the bandgaps of SnO2:F, CdS:In and CdTe layers is a strong evidence on the interdiffusion in the two interfacial regions and/or the formation of quantum dots.

5:30 PM

The Effect of Aluminum Content on Morphology, Size, Volume Fraction and Number of Graphite Nodules in Ductile Cast-Iron: *Ali-Reza Kiani-Rashid*[1]; A Shayesteh-Zeraati[2]; H Naser-Zoshki[3]; [1]Ferdowsi University of Mashhad; [2]Sharif University of Technology; [3]Iran University of Science and Technology, Tehran,

In this paper, we have investigated the effect of aluminum content on the formation mechanism, volume fraction, morphology and particle size distribution of graphite. Addition of aluminum to ductile iron causes some fundamental changes in iron-carbon phase-diagram and as a result, improves the graphite formation during the eutectic transformation. The results reveal that aluminum compounds have been formed in the core of graphite nodules; thus aluminum plays an important role in the formation of graphite nodules. Furthermore, it is indicated that an increase in the aluminum content also leads to an increase in the number of graphite nodules and a decrease in the nodules size. By using EPMA, the segregation of aluminum and silicon between graphite nodules has been studied.

5:55 PM

Characterization of the Deep Interface Traps in Hf-Based/Si Gate Stacks: *S.Y. Tan*[1]; Yi-Lun Hsia[1]; Ming-Yuan Wu[1]; Hsing-Hung Chen[1]; [1]Chinese Culture University

The continuous scaling of the dimensions of CMOS transistors has caused the thickness of the silicon dioxide to decrease below 1.6nm.The replacement oxides must satisfy various requirements as satisfactory gate oxides.(i)thermo dynamically stable in contact with the Si (ii)Oxygen diffusion(iii)form a high quality interface with Si. Hafnium based oxide films have potential to form a silicon oxide comparable interface with the Si.In order to fully understand the origins of the interface trap generation and deep oxide traps in Hf-based films,we introduced two unique process to control the deep trap centers in Hf-based/Si.A combined approach of (i)Thermal annealing treatment(ii)Different Hf contents in HfO2 (iii)Incorporating N atom into HfO2(iv)Electrical characterization–

C-V and J-V,to study the effect of the thermal annealing and Hf contents.(v) Physical characterization - TEM, XPS, and SIMS. It is the first time,two unique process techniques are employed to control of the deep interface traps in Hf-based gate dielectric films on Silicon.

6:15 PM
Arsenic Dopant Effect in Nickel Silicide Formation for NiSi/Hf-Based/Si Gate Stacks: *S.Y. Tan*[1]; [1]Chinese Culture University

The thermal stability of fully silicided NiSi with arsenic doping on silicon was investigated. The aims of the work were to investigate the Ni silicide phase-related issues associated with arsenic dopant and thermal annealing on Ni-FUSI/HfO2/Si and Ni-FUSI/HfSiO/Si gate stacks. It was found that arsenic-incorporation demonstrated some improvement in both morphology and phase stability of nickel silicided films at high processing temperatures regardless underlying gate dielectrics. The correlations of Ni-Si phase transformation and arsenic doapnt with their electrical and physical changes were established by sheet resistance measurements, XRD, AFM, and X-ray photoelectron spectroscopy (XPS) analysis. Furthermore, the modulation of the work function (WF) of Ni fully silicided gates by arsenic impurity is presented, comparing the effects of dopant (As) on the WF and silicide phases (NiSi and NiSi2). It confirmed that the work function of NiSi can be tuned by implanting arsenic dopant, but it ineffective for NiSi2 phase.

6:35 PM Concluding Comments

6:40 PM Question and Answer Period

Coatings for Structural, Biological, and Electronic Applications: Applications of Coatings II

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS: Thin Films and Interfaces Committee
Program Organizers: Nuggehalli Ravindra, New Jersey Institute of Technology; Gregory Krumdick, Argonne National Laboratory; Roger Narayan, Univ of North Carolina & North Carolina State Univ; Choong-un Kim, University of Texas at Arlington; Nancy Michael, University of Texas at Arlington

Tuesday PM Room: 309
February 16, 2010 Location: Washington State Convention Center

Session Chair: Nuggehalli Ravindra, NJIT

2:00 PM Introductory Comments

2:10 PM Invited
Experimental Study of Ternary Cobalt Spinal Oxides for Photoelectrochemical Hydrogen Production: *Sudhakar Shet*[1]; Kwang-Soon Ahn[2]; Heli Wang[1]; Nuggehalli Ravindra[3]; Yanfa Yan[1]; John Turner[1]; Mowafak Al-Jassim[1]; [1]National Renewable Energy Laboratory; [2]YeungNam University; [3]New Jersey Institute of Technology

In this study, we present an experimental study of ternary cobalt spinal oxides for solar driven hydrogen production. Co1+dX2-dO4 (X = Al, Ga, In) thin films were deposited using RF magnetron reactive co-sputtering system. All the thin films were deposited on silver/stainless steel and quartz substrates, because of the high temperature (800°C) oxide growth. We found that these thin films show excellent stability in solution and good visible light absorption. However, their performance as photoelectrochemical catalyst is limited by the poor transport properties induced by small polaron mobility.

2:40 PM
Effect of Superficially Applied Y2O3 Coating on High Temperature Corrosion Behaviour of Ni-Base Superalloys: Gitanjaly G[1]; *Harpreet Singh*[2]; Satya Prakash[2]; Surendra Singh[2]; [1]National Institute of Technology; [2]Indian Institute of Technology

Effect of Y2O3 on high temperature corrosion of Superni 718 and Superni 601 superalloys was investigated in Na2SO4-60%V2O5 environment at 900°C for 50 cycles. Y2O3 was applied as a coating on the surfaces of the specimens. Superni 601 was found to be having better corrosion resistance in comparison with Superni 718 in the Na2SO4-60%V2O5 environment. The Y2O3 superficial coating was successful in decreasing the reaction rate for both the superalloys. In the oxide scale of the alloy Superni 601, Y & V were observed to co-exist thereby indicating the formation of a protective YVO4 phase. There was a distinct presence of protective Cr2O3 rich layer just above the substrate/scale interface in the alloy. Whereas, Cr2O3 was present with Fe and Ni in the scale of Superni 718. Y2O3 seemed to be contributing to better adhesion of the scale as comparatively lesser spalling was noticed in the presence of Y2O3.

3:00 PM
Semiconductor Device Integration Utilizing Magnetic Films: *Rene Rivero*[1]; Michael Booty[1]; Anthony Fiory[1]; Nuggehalli Ravindra[1]; [1]New Jersey Institute of Technology

Semiconductor device integration utilizing magnetic films is described here. In particular, Indirect Template Magnetic Field Assisted Assembly (IT-MFAA), a parallel processing technique, is presented. IT-MFAA is designed to assemble devices onto substrates through a versatile and scalable methodology, which is capable of adjusting to manufacturing situations and producing 100% yields with error correction. This paper discusses existing serial and parallel techniques and compares them to IT-MFAA. It demonstrates that IT-MFAA circumvents drawbacks present in other techniques, and outlines a model of IT-MFAA.

3:20 PM
Crystallization and Thermal Stability of Amorphous and Nanocrystalline TiO_2 Magnetron-Deposited Thin Films Studied by X-Ray Diffraction: *Radomir Kuzel*[1]; Zdenek Matej[1]; Lea Nichtova[1]; Jindrich Musil[2]; [1]Charles University in Prague, Faculty of Mathematics and Physics; [2]University of West Bohemia, Faculty of Applied Sciences

Photocatalytic and other remarkable properties of titanium dioxide depend on phase composition (rutile, anatase, brookite) and on the crystallinity. Various sets of films with different thickness were studied by X-ray scattering. Reflectivity showed increase of surface roughness with annealing time and film thickness. Depth profiling of nanocrystalline films by varying angle of incidence revealed a gradient of phase composition (rutile - anatase). While amorphous films crystallized quickly above 220°C depending on the film thickness (slow crystallization for very thin films), the nanocrystalline films (about 10 nm) showed microstructural stability up to about 500°C. This was evaluated with the aid of newly-developed software for total XRD pattern fitting which also confirmed the presence of tensile residual stresses arising during crystallization of amorphous films and increasing with the decreasing film thickness. They were also measured independently. Best film hydrophilicity was usually found for nanocrystalline anatase and/or mixed nanocrystalline anatase+rutile films.

3:40 PM Break

3:55 PM Invited
Spin Coated Er-Doped SiO2 for High Efficiency Waveguide Optical Amplifiers: *Sufian Abedrabbo*[1]; Bashar Lahlouh[1]; Anthony T. Fiory[2]; Nuggehalli Ravindra[2]; [1]University of Jordan; [2]NJIT

Erbium doped fiber amplifiers (EDFAs) and erbium doped waveguide amplifiers (EDWAs) have been studied extensively due to their enormous importance in optical communications networks. Although expenses in optical communications systems have been decreasing over time, device processing remains a significant cost component. This work describes a cost-effective process for fabricating Er-doped SiO2 thin films deposited on silicon substrates by spin coating of a sol-gel containing Er and silica precursors. The process includes thermal annealing to form Er doped SiO2 films. Optical activity of Er was determined by photoluminescence as a function of the excitation wavelength, and the optical constants of the processed films were determined by near infrared (NIR) absorption techniques and Fourier transform infrared (FTIR) spectroscopy.

4:25 PM
Stellite Coatings on Hot Work Tool Steels for Tooling Applications in Semi-Solid Processing of Steels: *Agca Kayihan*[1]; Yucel Birol[1]; Kemal Demirci[2]; [1]Tubitak Mam; [2]Kobatek Surface Treatment Industry

Semi-solid processing of metals combines the advantages of forging and casting while shaping metallic components. Having already matured into an industrial practice for Al and Mg alloys, this innovative forming technology could upgrade the market and provide lightweighting for forged steel parts. However, with process temperatures above 1250°C, the surface-to-interior

temperature differentials in steel thixoforming dies are much larger than with Al and Mg. Combined with the erosive wear caused by abrasion, the impact of already solid particles in the slurries and high temperature oxidation of the die surface, these cyclic thermal stresses confer very specific requirements on tool materials. X38CrMoV5 hot work tool steel widely used in the manufacture of conventional forging dies was coated with a series of stellite coatings in the present work. The performance of the coated samples was subsequently tested under thermal fatigue and erosive wear conditions encountered in the thixoforming of steels.

4:45 PM

Phase Change Materials – An Overview: Maneesh Merwade[1]; Vishal K. Singh[1]; *Arun Ramadass*[1]; Nuggehalli Ravindra[1]; [1]New Jersey Institute of Technology

Phase Change Materials (PCM) have found wide spread applications in the field of materials science and technology due to their interesting properties. High latent heat of fusion and low melting point of Phase Change Materials make them very good candidates for thermal management systems. Phase Change Materials have been widely used as thermal storage units (in direct and indirect contact), for removal of excess heat from systems/devices, in scenarios where increase in the operating temperature of these devices would be detrimental to their performance. The ability to store thermal energy in PCM (paraffins) has been employed for effective use of solar energy in buildings for air-conditioning purposes. Phase change materials have also been utilized very successfully in memory technology wherein the transition in state of materials is from amorphous to crystalline state. Phase Change Materials offer many potential applications. This paper describes some of the recent applications.

5:05 PM Invited

Organic Coatings to Prevent Molten Metal Explosions: *Alex Lowery*[1]; Joe Roberts[2]; [1]Wise Chem LLC; [2]Pyrotek Inc.

Over 60 years ago, the first reported molten metal explosion from a bleed-out during direct chill casting in an aluminium mill was reported. Soon thereafter testing was performed to determine the root cause of the explosion. Upon determination of the root cause, an investigation to determine if any preventive measures could be instituted to prevent the explosions was conducted. Results found that a specific organic coating (e.g., Wise Chem E-212-F) prevented molten metal explosions, whereas some specific organic coatings initiated the explosions. Fifteen years ago the U.S. Department of Energy in conjuncture with the Aluminum Association reinvestigated the root cause of the molten metal explosions. Testing revealed that an initiation or trigger had to be present for a molten metal explosion to occur. Testing identified three additional coatings that could afford protection.

5:35 PM

Diaphragm Coatings to Enhance Performance of Fabry-Perot Sensors: *Ivan Padron*[1]; Anthony Fiory[1]; Nuggehalli M Ravindra[1]; [1]NJIT

The performance of sensors based on Fabry-Perot (FP) interferometry is determined by optical reflection and transmission at the FP cavity interfaces. In the extrinsic FP sensor considered here, one of the cavity surfaces is the terminus of an optical fiber, the other is a flat surface of a silicon diaphragm, and the cavity contains a fluidic or gaseous medium.Dielectric or metallic coatings and surface texturing can be used to optimize the sensitivity of the FP interference signal to the displacement of the diaphragm. Optical modeling and simulation techniques are used to determine the optimal properties of coatings and surface treatments. Experimental data and analysis for FP pressure sensors are presented.

Computational Thermodynamics and Kinetics: Wetting Phenomena II

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Jeffrey Hoyt, McMaster University; Dallas Trinkle, Univeristy of Illinois at Urbana-Champaign

Tuesday PM Room: 308
February 16, 2010 Location: Washington State Convention Center

Session Chair: To Be Announced

2:00 PM Invited

Prefreezing at Heterogeneous Solid-Liquid Interfaces: *Brian Laird*[1]; Ruslan Davidchack[2]; [1]University of Kansas; [2]University of Leicester

Through the accurate calculation of surface and interfacial free energies, we determine the thermodynamic driving forces governing the wetting of the hard-sphere-fluid/hard-wall interface by a thin layer of (metastable) fcc crystal in a [111] orientation. Because this phenomenon occurs at densities below the equilibrium freezing density of hard spheres, it is an example of prefreezing. We discuss the relationships of these findings with other recently observed prefreezing phenomena in chemically heterogeneous solid-liquid interfaces.

2:30 PM Invited

Thinning, Instability and Rupture of Thin Liquid Films in Metal Foam: *Lucien Brush*[1]; [1]University of Washington

Thinning and rupture of thin liquid films in metallic foam can occur rapidly since there are no surfactants available to slow these processes. A matched asymptotic analysis is used to derive laws governing the capillary-suction driven thinning of very thin films in metallic foams. After an initial transient, films settle into one over time-squared behavior. The thinning films are tested for instabilities that arise from van der Waals forces. The effects of the Plateau borders and the flow in the lamella are seen to stabilize the thinning films. The critical film thickness denoting the onset of instability scales as the cube root of the Sheludko number. The results show instabilities may be localized near the edges of the film lamellae when there is a drainage flow. Linear instability initiates the rupture process of a film and the effect of flow on the rupture of the film is discussed.

3:00 PM

Simulating Surface Energy Anisotropy Using Extended Cahn-Hilliard Model: *Solmaz Torabi*[1]; ZhengZheng Hu[1]; John Lowengrub[1]; [1]University of California Irvine

We study the influence of surface and strain energies on heteroepitaxial thin-film growth. We propose an alternate way of simulating anisotropy for the surface energy by using the higher order terms in the free energy. To the second order, the system only have isotropic properties. We can produce different anisotropy by adding higher order terms to the energy. By choosing the right parameters, we can study the behavior SiGe/Si thin film. This type of extended Cahn-Hilliard model has been previously studied to the 4th order, but to our knowledge no one has ever implemented all the terms in this system. One advantage of this energy is that it has the intrinsic regularization. We present numerical results using an adaptive, nonlinear multigrid finite-difference method. Finally, in order to model the misfit strains, we add the elastic energy to our diffuse model to predict different Qdot shapes, such as pyramids and domes.

3:20 PM Break

3:30 PM Invited

Nanoscale Quasi-Liquid Interfacial Films: The Interplay of Premelting, Prewetting and Multilayer Adsorption: *Jian Luo*[1]; [1]Clemson University

Nanoscale quasi-liquid intergranular films have been observed in various ceramic materials (Crit. Rev. Solid State Mater. Sci. 2007, 32:67), as well as in metals such as W-Ni (Acta Mater. 2007, 55:3131) and Mo-Ni (APL 2009, 94:251908). Several thermodynamic models are presented and discussed. First, using CALPHAD data, a premelting type model predicts onset grain boundary

disordering at as low as 60-85% of the bulk eutectic/peritectic temperatures (APL 2008, 92:101902). This model quantitatively explains mysterious subsolidus activated sintering in refractory metals. Second, a systematical spectrum of interfacial phenomena, including prewetting/premelting, critical points, multilayer adsorption, layering/roughening, and complete wetting/drying, are predicted by combining diffuse-interface and lattice-gas models and incorporating colloidal type interfacial forces. This model also produces a series of grain boundary phases (complexions) with character similar to those observed by Dillon and Harmer. Finally, analogous phenomena at free surfaces and their thermodynamic models are reviewed (Annu. Rev. Mater. Res. 2008, 38:227).

4:00 PM

Influence of Local Interface Phenomenon on Coalescence Kinetics Models in Ni-Base Alloys: *Youhai Wen*[1]; Jeff Simmons[2]; Chris Woodward[2]; [1]UES, Inc; [2]AFRL

The high coalescence rate in modeling gamma-prime microstructural evolution becomes especially serious in simulations with high volume fractions of gamma prime precipitate (e.g. higher than 60%). While the out-of-phase domains that come into contact tend to maintain a thin layer of gamma matrix phase between them due to favorable APB/interfacial energy considerations, the in-phase domains tend to coalesce into one single domain to remove the extra matrix layer between them and reduce the interfacial energy. In phase field simulations of this latter process the simulated coalescence rate is extremely high compared with experimental observations. We identified that this is a result of ignoring the local phenomenon in the gamma and gamma prime interface regions in all major simulate methods. Reasonable coalescence rate is achieved by properly addressing this local phenomenon. This implies that local phenomenon in the interface region has a strong effect on controlling the coalescence kinetics and corresponding microstructural evolution.

4:20 PM

Microstructure Engineering via Throttled Nucleation: *David Wu*[1]; Jerry Quek[1]; Kevin Chu[1]; [1]Institute of High Performance Computing

We consider the crystallization of a thin film via interface-limited growth and throttled nucleation – a transient nucleation schedule in which the nucleation rate is turned off before the sample is fully transformed. This schedule can be readily realized by two stages of isothermal anneals, where the throttling time is parameterized by the area fraction transformed when throttling occurs. The limits of throttling are simultaneous and continuous nucleation, which produce the Poisson-Voronoi and Johnson-Mehl structures, respectively. We use an efficient level set method to generate microstructure, which is subsequently used to study a crystal's geometric properties. We find that throttling produces significant differences in the distributions of grain area, grain perimeter, and number of grain edges. Our results suggest a possible way to easily engineer certain types of microstructures via isothermal annealing stages.

4:40 PM

Phase Field Modeling of Void Microstructure Evolution in Irradiated Metals: *Srujan Rokkam*[1]; Santosh Dubey[1]; Anter El-Azab[1]; Paul Millett[2]; Dieter Wolf[2]; [1]Florida State University; [2]Idaho National Laboratory

We present a phase field model for void nucleation and growth in irradiated metals. The point defects generated due to cascades are modeled using the concept of stochastic point process in space and time. The kinetics of point defects is obtained using a Cahn-Hilliard type description of vacancy and interstitial concentration fields. The void microstructure is obtained in terms of the evolution of a non-conserved order parameter, whose evolution is prescribed by a phenomenological Allen-Cahn type equation. We illustrate model capabilities using 2D and 3D examples for the case of pure metals. Void nucleation and growth is investigated in the presence of interacting point defects, defects interaction with lattice sinks, thermal fluctuations and cascade damage. Finally, the effect of spatially resolved defect sinks (such as dislocations) on void nucleation and growth is investigated.

5:00 PM

Monte Carlo Potts Simulation of Strain Induced Sub-Grain Structure Formation: *Corentin Guebels*[1]; Tien Tran[1]; Phi Thanh[1]; Joanna Groza[1]; Jean-Pierre Delplanque[1]; [1]University of California, Davis

The prediction of microstructural evolution and abnormal grain growth phenomena during high-temperature deformation requires an accurate description of recrystallization. A recovery model is presented and contributes to Monte Carlo Potts simulations for recrystallization phenomena. This approach describes the creation of low angle boundaries in the grain interior from stored strain energy, which produces a sub-grain structure. This novel recovery model investigates the often-ignored incubation period prior to a static recrystallization cycle. The model provides a flexible, physics-based methodology to investigate the initiation of recrystallization through competing sub-grain growth. The simulation results clarify the influence of the advantageous growth of a few sub-grains on recrystallization kinetics.

5:20 PM

Using Size Distributions for Determining Growth Mechanisms of Grain Boundary Precipitates: *Shirley Northover*[1]; [1]The Open University

In the past various single parameters such as the mean, mode or maximum of the precipitate size distribution have been used in experiments to determine growth mechanisms. In the present study the observed development with aging time of the size and shape distributions of bcc precipitates at grain boundaries in an fcc material (Co-20Fe at 1003°K) have been compared with various possible theoretical models to determine the rate controlling process. The growth of these precipitates is initially well described by the grain boundary dependent collector plate mechanism of Brailsford and Aaron. As the precipitates grow low energy facets are formed which can move only by the propagation of ledges and growth becomes interface controlled. The precipitates' diffusion fields soon overlap and coarsening occurs with interface control. The results demonstrate that this would not have been revealed using simpler measures of precipitate size.

5:40 PM

Genetic Alloy Design by Nanoprecipitate Control: Stainless Steels and Aluminium Alloys: *Pedro Rivera-Diaz-del-Castillo*[1]; [1]University of Cambridge

Alloy designers usually encounter a myriad of possibilities when engineering and optimising existing and new alloys. Thermochemical databases have recently become increasingly reliable, covering now a widespread number of phases, and can now be used for high-performance alloy design; however, the alloy designer encounters typically at least 5 relevant alloying elements combined with various heat treatment stages, temperatures and times. Due to computational time, thermodynamic and kinetic calculations for all possibilities are unfeasible. The present work describes a methodology to optimise properties such as strength, ductility and/or corrosion resistance in stainless steels and aluminium alloys. The key parameter to control is the formation of nanoprecipitaes. Strategies to deal with meta-stable phases for which energetics may be estimated form first principle calculations are introduced. Genetic algorithms are employed as a means to minimise the computational time to devise an optimal alloy composition and its corresponding heat treatment.

Cost-Affordable Titanium III: Powder Consolidation and Properties II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
Program Organizers: M. Ashraf Imam, Naval Research Lab; F. H. (Sam) Froes, University of Idaho; Kevin Dring, Norsk Titanium

Tuesday PM Room: 618
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Ramana Reddy, University of Alabama; Kevin Dring, Norsk Titanium AS

2:00 PM

Microwave Sintering and Melting of Titanium Powder for Low-Cost Processing: *Ralph Bruce*[1]; Arne Fliflet[2]; Hugo Huey[3]; Chad Stephenson[1]; M. Ashraf Imam[2]; [1]Bethel College; [2]Naval Research Laboratory; [3]HWave, LLC

The emerging reduction technologies for titanium from ore produce powder instead of sponge. Conventional methods for sintering and melting of titanium powder are costly, as they are energy intensive and require high vacuum since titanium acts as a getter for oxygen at high temperature, adversely affecting mechanical properties. Other melting processes such as plasma arcs have the additional problem of electrode consumption, and direct induction heating of the titanium powder is problematic. Microwave sintering or melting in

an atmospheric pressure argon gas environment is potentially cost effective and energy efficient due to the possibility of direct microwave heating of the titanium powder augmented by hybrid heating in a ceramic casket. We are investigating this approach at the Naval Research Laboratory using an S–Band microwave system. Evaluation of the product metal is underway and results will be discussed.Acknowledgment: This work is supported by the Office of Naval Research.

2:25 PM

Microwave Sintering of Titanium: *Ma Qian*[1]; Shudong Luo[1]; Ming Yan[1]; Graham Schaffer[1]; [1]The University of Queensland

Effective sintering of titanium requires use of a high sintering temperature (> 1200°C) in high vacuum (<10^{-2} Pa). Consequently, sintering of titanium has been normally pursued in high vacuum ceramic tube furnaces, mostly on a laboratory scale. This confines the heating and cooling rates to typically about 4°C/min, thereby leading to lengthy processing. This work presents an assessment of microwave sintering of titanium. Elemental titanium powders of < 20 µm; 45-63 µm; and 100-150 µm were used to make green samples at 400 MPa. Sintering was conducted at 1200°C for 2 hrs in a 3 kW high vacuum microwave furnace with a 2.45 GHz multimode cavity under a vacuum of 2-5×10^{-3} Pa. In all cases studied, the MW-sintered densities compare favorably or at least equally with the conventionally sintered densities. HRTEM examination of the MW-sintered interfaces revealed excellent bonding. Microwave sintering shows significant cost-effectiveness for processing of titanium.

2:50 PM

Reaction Assisted Ultrasonic Consolidated TiNi: *Henry Rack*[1]; Mykola Kulakov[1]; Erica Sampson[1]; [1]Clemson University

This presentation will describe the results of an investigation that was designed to examine the feasibility of fabricating near-net shape components containing TiNi shape memory constituents by combining the attributes of ultrasonic consolidation and reaction heat treatment. An optimization design of experiment was conducted to determine the ultrasonic consolidation control parameters, e.g., sonotrode rotational velocity, amplitude and normal force, which would result in the maximum weld quality as defined by the linear weld density. Subsequent thermal treatment of the consolidated compact resulted in the formation of a graded Ti-TiNi-Ni microstructure, the thickness of each constituent depending upon the exposure time and temperature.

3:15 PM

Properties of Conventionally Alloyed and Powder Alloyed Nano-Crystalline Titanium Consolidated Via Spark Plasma Sintering: Christopher Melnyk[1]; Steven Schroeder[1]; David Grant[1]; *Robert Gansert*[2]; [1]California Nanotechnologies; [2]Advanced Materials and Technology Services

Nano, near-nano, and multi-modal grained materials show great potential for application in many commercial industries. The Hall-Petch relationship cites the strengthening of materials by reducing the average crystallite size. A study is proposed to investigate the increase in mechanical properties provided by nano, near-nano as well as multi-modal grained powders used in powder metallurgical applications. Consolidations of processed materials will be produced using Spark Plasma Sintering (SPS). Nano-crystalline titanium, and titanium alloy powders and will be processed via cryogenic milling. The mechanical properties of the nano, near-nano and multi-modal crystalline materials will be compared to conventional materials of the same composition. Initial testing of titanium based materials indicates an increase in strength and hardness by 2 to 3 times from the use of nano and near nano crystalline structures. Cryo milled powders and the consolidated forms of these powders will be examined using microstructural analysis and mechanical testing.

3:40 PM Break

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Fabrication of Ultrafine/Nanostructued Ti-TiN/TiC Matrix Composites Using Low-Temperature Back Pressure Equal Channel Angular Pressing: Wei Xu[1]; Xiaolin Wu[1]; Haowen Xie[1]; Jizhong Li[1]; *Kenong Xia*[1]; [1]University of Melbourne

Fully dense nanostructured Ti-TiN/TiC composites have been synthesized by consolidating ball-milled mixtures of pure Ti and TiN/TiC particles (up to 10 vol.%) using back pressure equal channel angular pressing (BP-ECAP) for up to 6 passes at temperatures between 500-550°C which is much lower than that of conventional sintering.Microstructure analysis indicated that nanoscale reinforcing phases of several tens of nanometers were distributed mainly at grain boundaries and partially within the interior of ultrafine Ti grains (< 1 µm). The presence of nanoscale reinforcing particles was attributed to ball milling whereas the refinement of Ti matrix was ascribed to both ball milling and BP-ECAP. As a result, yield strength up to 1260 MPa and Vickers microhardness up to 5000 MPa have been attained with noticeable compressive plastic strain. It implies that the strategy of employing both ball milling and BP-ECAP is feasible to produce high-performance nanostructured Ti matrix composites.

4:20 PM

Stress-Corrosion Cracking and Fatigue Crack Growth Behavior of Ti-6Al-4V Plates Consolidated from Low Cost Powders: *Peter Pao*[1]; M. Ashraf Imam[1]; Robert Bayles[1]; C.R. Feng[1]; [1]Naval Research Laboratory

Ti-6Al-4V plates, consolidated from low cost powders, manufactured from Armstrong-process and also from hydride-process were studied. The yield strengths of these plates of Ti-6Al-4V are comparable and are about 920 MPa. The plate from hydride-process has higher oxygen content compared to the plate from the Armstrong-process (0.3 vs. 0.2 wt%). To remove the prior history of consolidation, the plates are beta annealed and the test results are compared with as received condition. Fatigue crack growth study indicates that, in the as-received condition, the fatigue crack growth threshold stress intensities of Armstrong-process and hydride-process Ti-6Al-4V approaches to that of the conventional Ti-6Al-4V. The stage II fatigue crack growth rates of hydride-process of Ti-6Al-4V are substantially higher than that of the Armstrong-process and the conventional cast-ingot-cast. The mechanism of the fatigue crack growth rates difference, fracture toughness, and stress-corrosion cracking resistance of Ti-6Al-4V will be discussed.

4:45 PM

Sintering Behavior of TiH2 for Manufacturing of Titanium Alloys and Products: *Zhigang Fang*[1]; Hongtao Wang[1]; Shuming Fang[2]; Jiamin Zhang[2]; [1]University of Utah; [2]CYMCO

Powder Metallurgy (PM) is an effective cost-saving approach for production of titanium parts due to the advantage of near net shape (NNS) capability of PM techniques. However, the traditional powder metallurgy approach for making titanium alloys faces a number of challenges with respect to issues related to contamination, porosity, and cost when highly alloys powders and high pressure consolidation processes are used. In recent years, a new process technology is emerging by which titanium and titanium alloys can be made by sintering titanium hydride (TiH2) and its mixture with alloying elements. The feasibility of this manufacturing approach has been demonstrated fully from powder to sintering and from microstructure to mechanical properties. However, to date there is little published information on fundamentals of this manufacturing technology. In this investigation, the basic chemical and physical properties of TiH2 are examined through literature as well as experiments. The behavior of TiH2 powder during compaction and sintering is reported.

5:10 PM

Structure Formation during Preparation of Variable Porosity Ti Foams by Solid State Replication: Yu. Orlova[1]; K. Maekawa[1]; *Henry Rack*[2]; [1]Kyoto University; [2]Clemson University

Fabrication of variable porosity titanium foams through incorporation of sacrificial sodium chloride powder has been investigated. While the solid foam skeleton was formed during high temperature sintering of the host commercial purity titanium powder particles, with faceting of the powder surface being noted throughout, the three-dimensional pore structure contained macro- (200-400µm), micro- (5-10µm) and sub-micropores (< 1.5µm). The largest macro-pores had a cubical topography representative of the sodium chloride powder, this having been left following vaporization of the sacrificial powder. Formation of the smaller micro-pores appeared to have occurred during the compaction process and having been kept in the specimen's body due to incomplete sintering of the host powder. Finally formation of the smallest sub-micropores was associated with high temperature gas evolution occurring during sodium chloride vaporization.

5:35 PM

Production of a Low-Cost DMD Wire Feedstock by Direct Consolidation of Ti Sponge: *Kevin Dring*[1]; Martin Lefstad[2]; Ola Jensrud[3]; [1]Norsk Titanium; [2]SINTEF - Materials and Chemistry; [3]Sintef Raufoss Manufacturing

Direct Metal Deposition (DMD) and other additive manufacturing techniques have received significant interest amongst end users of titanium

in recent years. Promises of dramatically reduced machining to obtain a final titanium component, while retaining mechanical properties approaching wrought material have engaged a broad spectrum of industries. The cost of such manufacturing processes may be prohibitively high when expensive feedstocks such as titanium powder are used. Norsk Titanium, in collaboration with SINTEF and NTNU through a Norwegian Research Council grant, has investigated the production of a low-cost wire feedstock that was produced by direct, non-melt consolidation of titanium. By eliminating the melting and primary working of titanium, significant cost-savings may be realised. The titanium wire was characterised for chemical composition and has demonstrated oxygen levels of 1500 ppm. Test specimens were built up from the wire feedstock and these were analysed for mechanical properties and compositional conformance to relevant standards.

Electrode Technology for Aluminum Production: Traditional and Inert Anode Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Ketil Rye, Alcoa Mosjøen; Morten Sorlie, Alcoa Norway; Barry Sadler, Net Carbon Consulting Pty Ltd

Tuesday PM Room: 616
February 16, 2010 Location: Washington State Convention Center

Session Chair: Jilai Xue, Unversity of Science and Technology Beijing

2:00 PM Introductory Comments

2:05 PM
Higher Softening Point Pitch as Anode Binder Pitch: *Robert Wombles*[1]; Stacey McKinney[1]; Thomas Golubic[1]; Kathryn Sickels[1]; [1]Koppers Inc.

In recent years several authors have discussed the use of higher softening point pitches as anode binder pitches. Several drivers exist to encourage movement from the use of the traditional 110°C softening point pitch as anode binder pitch to pitches with higher softening points. Some of these include the movement to higher amperage pots, the concern for employee exposure to coal tar pitch volatiles and polynuclear aromatic hydrocarbons, and the never ending search for improved anode performance. This paper will discuss several aspects of high softening point pitch including their production; physical, chemical and composition properties, and the potential for their use to improve the quality of anodes produced. In addition, the effect of their use on coal tar pitch volatile and polynuclear aromatic hydrocarbon emissions will be discussed.

2:25 PM
Study of Resistivity – Real Density Correlation in CPC Calcination Control: *Oscar Mascarenhas*[1]; Arun Mathur[1]; Jose Botelho[1]; [1]Goa Petcoke Consultancy Services

Petroleum Coke calcination process control relies on testing the Electrical Resistivity of the output product. This resistivity test is in lieu of the Real Density requirement in the Calcined Petroleum Coke quality specifications, as it takes lesser time for analysis instead of the standard methods for Real Density. The correlation between Real Density and Resistivity is suitably linear, however the correlation is affected when there is significant variation in the green coke in terms of particle size distribution or variation in proportion of multiple green cokes used. This paper presents details of an on field study and analysis of the Resistivity – Real Density correlation and suggests methods to use Resistivity as a more effective predictor of Real Density in a production run, including dynamically set Resistivity targets leading to better calcination control and optimization of production costs.

2:45 PM
The Comparison between Vertical Shaft Furnace and Rotary Kiln for Petroleum Coke Calcination: Yi Sun[1]; Haifei Xu[1]; *Yubin Wang*[1]; Yinhe Cui[1]; Chaodong Liu[1]; [1]Shenyang Aluminum and Magnesium Engineering and Research Institute

At present, the petroleum coke is mainly calcined by vertical shaft furnace or rotary kiln to meet the requirement of prebaked anode used for Aluminum reduction in China. Vertical shaft furnace for calcination is quite different from rotary kiln for calcination in the principle of calcination process, calcined coke quality, product availability, production capacity, energy consumption, operation cost, environmental issues and especially in the properties of calcined coke on real density, air reactivity, CO2 reactivity and ignition temperature which are closely related to the consumption of anode for aluminum reduction. Therefore, calcination of coke used for aluminum reduction by vertical shaft furnace is more competitive based on the existing quality of the green petroleum coke and the supply thereof.

3:05 PM
Prebaked Anode from Coal - Utilization of Coal Extract as a Coke Feedstock: *Maki Hamaguchi*[1]; Noriyuki Okuyama[1]; Nobuyuki Komatsu[1]; Jiro Koide[2]; Keisuke Kano[2]; [1]Kobe Steel, Ltd.; [2]Sumitomo Corporation

Preparation of prebaked anode utilizing coal solvent extraction technology will be reported. In the prebaked anode industry, it is recognized that the quality of anode coke has continuously declined, namely, higher sulfur and impurities, higher volatile matter content, and lower density, mostly due to the deterioration of crude oil quality.We at Kobe Steel, Ltd., have been developing a new non-hydrogenative solvent extraction process for coal with the aim of applying the coal-extract to metallurgical coke making. We named this process and the product (the ash-free coal-extract), a HYPER-COAL (HPC). In this paper, we will describe fabrication of prebaked anode test specimen using HPC as a feedstock of coke. It was demonstrated that prebaked anodes prepared from HPC coke have various advantages such as extremely low impurities such as Sulfur, Vanadium and Nickel, high apparent density, and low air- and CO2-reactivity compared to those from anode grade calcined petroleum coke.

3:25 PM
Charcoal in Anodes for Aluminium Production: *Bodil Monsen*[1]; Arne Ratvik[1]; Lorentz Lossius[2]; [1]SINTEF Materials and Chemistry; [2]Hydro Aluminium - PM Technology

Carbon anodes used in aluminium production are made from petroleum coke and coal tar pitch. Substituting some of the petroleum coke with charcoal have the potential to reduce the environmental footprint of the aluminium production, as CO2 from renewable sources are not considered a greenhouse gas. However, since charcoal is more porous and have a different reactivity towards air and CO2 than pet coke, charcoal may have adverse effects on anode properties. Pilot anodes, substituting part of the petrol coke with charcoal, have been produced and evaluated based on typical anode properties, e.g. apparent density, air-, and CO2-reactivity. Industrial charcoals, as used in the production of silicon, and pure charcoals, produced from maple and spruce, have been tested. In an attempt to reduce the effect of the porous charcoal, only the finer fractions of the anode dry mix were substituted. Substitutions based on both weight and volume are examined.

3:45 PM Break

4:00 PM
Ball-Milled Materials as Inert Anodes for Aluminum Production in KF-Alf3 Low-Temperature Electrolyte: *Sébastien Helle*[1]; Benoit Brodu[1]; Boyd Davis[2]; Daniel Guay[1]; Lionel Roue[1]; [1]INRS EMT; [2]Kingston Process Metallurgy Inc.

High-energy ball milling is well-known to be a powerful and simple technique to produce fine-grained and highly homogeneous materials with unique chemical, physical and mechanical properties for various applications. In this study, various nanostructured materials including Cu-Ni-Fe, Cu-Al-Ni-Fe based alloys and (Cu-Ni-Fe + MOx) composites were prepared by ball milling, consolidated to form dense electrodes and then evaluated as inert anodes for aluminum production in low-temperature (700°C) KF-AlF3 electrolyte. Their morphological, structural and chemical characteristics were studied at different stages during their preparation and after 20 h of electrolysis. Some of these ball-milled materials were identified as promising inert anodes for producing aluminum with a good purity.

4:20 PM
Corrosion Behaviors of NiFe2O4-NiO-Co3O4 Inert Anodes Materials in Na3AlF6-Al2O3 Melts: *Jilai Xue*[1]; Tao Zeng[1]; Jun Zhu[1]; [1]University of Science and Technology Beijing

Inert anodes for aluminum electrolysis were fabricated in laboratory by solid-state sintering of NiFe2O4-NiO-Co3O4 mixtures with varying composition. The microstructures of the materials, before and after electrolysis, were characterized using XRD, SEM-EDS and EPMA. The corrosion tests were systematicall performed in Na3AlF6-Al2O3 melts under different operating

parameters. It was found that the Co3O4 addition up to 3% in mass improved the material density and conductivity, and lowered its porosity as well, due to more chemically stabilized microstructure formed with the NiFe2O4-NiO-Co3O4 mixture than the NiFe2O4-NiO. The corrosion rate reduced with adding Co3O4, but increased again with the addition above 4%. The major contributing factor to the corrosion was the metallic Al dissolved in the melts, but this could be counteracted partly by oxygen gas generated in electrolysis with the appropriate current density about 0.4 A/cm2. The chosen materials show good corrosion resistance with potential for further application.

4:40 PM

Effect of Sintering Parameters on Properties of 18NiO-17(Cu-Ni)-65NiFe2O4 Composite Ceramic Anode: *Jia Ma*[1]; Yao Guang Chun[1]; Bao Li[1]; Zhang Xiao[1]; Ma Jun Fei[1]; [1]Northeastern University

Cold-pressing sintering is adopted to prepare 18NiO-17(Cu-Ni)-65NiFe2O4 composite ceramic inert anodes for aluminum electrolysis. The morphology, grain size and phases were investigated by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD), respectively. The effect of temperature and pressure on the relative density, porosity, grain size, electrical conductivity and bending strength was studied. The results show that temperature and pressure can significantly improve the properties of 18NiO-17(Cu-Ni)-65NiFe2O4 composite ceramic. The relative density of the specimens increases with temperature and pressure. The grain size increases with increase of the temperature, while it decreases with the increase of the pressure. The electrical conductivity increases with the decrease of grain size and porosity. The bending strength increasing with temperature and pressure then decreasing with temperature and pressure.

5:00 PM

Research on Preparation and Properties of 18NiO-NiFe2O4 Composite Ceramic Inert Anodes: *Jia Ma*[1]; Yao Guangchun[1]; Bao Li[1]; Zhang Xiao[1]; Ma Junfei[1]; [1]Northeastern University

In order to improve the properties of NiFe2O4 ceramic inert anodes, two-step sintering process was adopted to prepare and reinforce 18NiO-NiFe2O4 composite ceramic inert anodes by solid-state reaction, with 18wt% excess NiO. 18NiO-NiFe2O4 spinel matrix material was prepared firstly with NiO and Fe2O3 as the raw materials. The material prepared above was crushed to different sizes grains, and then blended together by cold-pressing sintering method. The effect of temperature on the relative density, porosity, electrical conductivity and bending strength was studied. The results show that temperature can significantly improve the properties of NiFe2O4 composite ceramic. The relative density of the samples increases from 69.59% to 98.28%, the porosity decreasing from 30.41% to 1.72% and the bending strength increasing from 14.62MPa to 71.94MPa, with temperature increasing from 1423 K to 1673K..

5:20 PM

Effect of Adding Ni-Fe on Properties of Inert Anodes of NiFe2O4 Based Cermet: *Zhongsheng Hua*[1]; Guangchun Yao[1]; Lei Wang[1]; Zhigang Zhang[1]; Lisi Liang[1]; [1]Northeastern University

To study the effect of metal addition on the properties of inert anodes of NiFe2O4 based cermet, Ni-Fe-NiFe2O4 cermet inert anodes were prepared by adding Ni and Fe powders into raw materials. The phase compositions, the mirco-structures, and the bending strength were investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM), INSTRON4206-006 electron mechanical experimental machine,respectively, and the bulk density and porosity were characterized by Archimedes drainage. The results showed that the cermets were consisted of NiFe2O4 and Ni-Fe, Ni-Fe distributed uniformly in the matrix. The bending strength increased with increasing Ni-Fe content, but the bulk density decreased, the porosity increased. Also the reasons leading to this phenomenon were analyzed.

Energy Conservation in Metals: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Energy Committee

Program Organizers: Cynthia Belt, Superior Industries International; Mark Cooksey, CSIRO; Donald Whipple, Bloom Engineering Co Inc; Russell Hewertson, Air Products and Chemicals Inc

Tuesday PM — Room: 310
February 16, 2010 — Location: Washington State Convention Center

Session Chairs: Mark Cooksey, CSIRO; Russell Hewertson, Air Products and Chemicals Inc

2:30 PM Introductory Comments

2:35 PM

Five Low Cost Methods to Improve Energy Efficiency on Reverberatory Furnaces: *Cynthia Belt*[1]; Ray Peterson[2]; Dave Bequette[1]; [1]Superior Industries International; [2]Aleris International

One important way to cut production costs in the metals industry is through implementation of energy efficiency improvements. Energy improvements can be found by reviewing furnace equipment systems and operating practices so as to operate the melting and holding process at the highest efficiency. A number of the changes can be implemented with minimal expense or capital investment. This paper will review several low cost methods to optimize energy efficiency on reverberatory melting or holding furnaces. These five methods (furnace utilization, idle procedures, fire rate, controls, and furnace pressure) will be explained along with information to implement these changes. Included are several rules-of-thumb and benchmarks for aluminum reverb furnaces.

2:55 PM

Energy Saving in the Foundry Industry by Using the CRIMSON Single Shot up Casting Process: *Mark Jolly*[1]; [1]University of Birmingham

Instead of using the traditional batch casting process, the CRIMSON (Constrained Rapid Induction Melting Single Shot Up-Casting) method employs a high-powered furnace to melt just enough metal to fill a single mould in a closed crucible. The crucible is transferred to a station for computer-controlled counter gravity filling of the mould for optimum filling and solidification. The CRIMSON method therefore holds the liquid aluminium for a minimum of time drastically reducing the energy losses attributed to holding the metal at temperature. With the rapid melting times achieved, of the order of minutes, there isn't a long time at temperature for hydrogen to be absorbed or for thick layers of oxide to form. The metal is never allowed to fall under gravity and therefore any oxide formed is not entrained within the liquid. Thus higher quality castings are produced leading to a reduction in scrap rate and reduced overall energy losses.

3:15 PM

Energy and Emissions with Oxyfuel: *Thomas Niehoff*[1]; [1]Linde Gas

Oxyfuel combustion systems are state of the art for many melting operations. Airfuel systems have been stepwise modified to oxygen enrichment, oxygen lancing and oxyfuel. Every change of the melting process will have consequences with regards to emissions and energy usage. The uniqueness of many operations requires unique skills to resolve potential issues. This paper will give some examples of how airfuel systems can be successfully converted to oxyfuel.

3:35 PM

Industrial Application Experiences with Microporous Calcium Hexaluminate Insulating Material SLA-92: *Dale Zacherl*[1]; Rainer Kockegey-Lorenz[2]; Andreas Buhr[2]; [1]Almatis, Inc; [2]Almatis GmbH

The microporous calcium hexaluminate insulating material SLA-92 has been introduced as an alternative to refractory ceramic fiber and other insulating refractory materials. Key properties associated with this product include: high chemical purity, long term high temperature stability up to 1500°C, low thermal conductivity up to 1500°C and high thermal shock resistance. The properties of this material and various applications in the steel and ceramic industries have been reported in previous papers. The current paper summarizes the industrial application experiences with innovative refractories based on SLA-

92 and discusses potential new applications in various manufacturing as well as energy-related industries.

3:55 PM
Identifying Some Potential Future Hydrometallurgical Processes in Treatment of Nickel Laterites: *Sarveswara Rao Katragadda*[1]; [1]Institute of Minerals and Materials Technology

Existing technologies that currently produce nickel worldwide utilize only about half of the nickel laterite deposits. So, there is a need for a new processing route to recover nickel and cobalt from the remaining laterite ores that are otherwise unutilized. Accordingly, an overview of different existing types of nickel resources and economical processes based on laterite horizons, consumption of energy and reagents and environmental concerns is made in this paper, along with a summary of metallurgical and engineering challenges to be overcome when implementing new technologies and concepts with regard to base metal extraction and sulfur removing efficiency. The paper also compiles and analyses a range of data concerning energy and greenhouse emissions pertaining to nickel technology.

4:15 PM Concluding Comments

Failure of Small-Scale Structures: Device Failure and Fatigue

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Marian Kennedy, Clemson Univeristy; Brad Boyce, Sandia National Laboratory; Reinhold Dauskardt, Stanford; Zhiwei Shan, Hysitron Inc

Tuesday PM Room: 206
February 16, 2010 Location: Washington State Convention Center

Session Chair: Molly Kennedy, Clemson University

2:00 PM Invited
Nitinol Fatigue – A Review: *Xiao-Yan Gong*[1]; Jixi Zhang[2]; Yanyao Jiang[2]; [1]Medical Implant Mechanics LLC; [2]University of Nevada, Reno

Stent is a metal mesh used as a medical implantable device to open the blocked artery or as a structural device supports the prosthetic flexible heart valve leaflets. Typical cross-section of a Nitinol self-expanding stent is on the order of several hundred microns. Recently, there is an increasing interest in understanding the fatigue of Nitinol owing to a hard lesson learned from using self-expanding stents treating the superficial femoral arteries. Fracture occurrences of up to 50% have been reported in some stents after one year follow-up. These stent fractures are due to in vivo cyclic displacements. As such, this presentation provides a review of Nitinol fatigue from over seventy published articles. It summarizes the key findings, identifies important challenges and provides a perspective for future work.

2:25 PM
Failure of Micron-Scale Polysilicon MEMS: Fatigue and Wear Mechanisms: *Daan Hein Alsem*[1]; Robert Ritchie[2]; [1]Lawrence Berkeley National Laboratory; [2]Lawrence Berkeley National Laboratory/University of California at Berkeley

Adhesion, wear, fatigue and fracture are all potential failure modes for MEMS devices. We focus on failure mechanisms in micron-scale polycrystalline silicon using on-chip testing and explain these phenomena in terms of the physical mechanisms using analytical transmission electron microscopy. With fatigue shown to occur via moisture-assisted sub-critical cracking within a cyclic stress-assisted thickened nano-scale oxide layer until the crack reaches a critical size to cause device failure, the sidewall morphology quantitatively determines fatigue and fracture behavior. Wear in ambient air is shown to involve a short adhesive wear regime, followed by an abrasive wear regime, where <100 nm silicon debris is created by fracture through silicon grains. These particles oxidize while plowing, leaving abrasive grooves associated with cracking rather than plastic deformation. Wear rates were orders of magnitude smaller than expected from macro-scale studies. This work illustrates that large surface-to-volume ratios in micron-scale systems can markedly alter mechanical behavior.

2:40 PM
Finite Element Simulation of Galvanic Corrosion in Silicon Microsystems: *Collin Becker*[1]; Conrad Stoldt[1]; David Miller[2]; [1]University of Colorado; [2]National Renewable Energy Laboratory

Many microsystems fabrication technologies currently employ a metallic overlayer, such as gold, in electrical contact with silicon (Si) structural layers. During postprocessing in hydrofluoric acid based solutions, a galvanic cell is created between the silicon and the metallic layer. As a consequence, autonomous corrosion (etching) of the silicon layer occurs and mechanical and electrical properties of structural Si are degraded. We present a finite element simulation to model electrochemical corrosion in MEMS. The model uses a heat transfer analogy to electrical conduction and predicts electrochemical kinetics with the Tafel equation. Focused ion beam milling of micron scale silicon resistive probes with metallic overlayers verifies the model accurately predicts corrosion in MEMS. Specifically, modeling results reproduce the current limited trend resulting from the surface area ratio (SAR) of metal to silicon.

2:55 PM
Size-Scale Effects in the Fracture of Polycrystalline Silicon for Microsystems: *Brad Boyce*[1]; E. David Reedy[1]; James Foulk[1]; [1]Sandia National Labs

In polycrystalline silicon used for microelectromechanical systems (MEMS), fracture is governed by the relationships between the size of the native flaws and the size of the microstructure, the size of geometric features (corners, notches, etc.), and the size of the device itself. In this study, we investigate the influence of these four length scales on resulting reliability using a recently developed high-throughput sequential tensile test structure which permits rapid collection of 1000's of microscale tests for robust extreme-value statistical analysis. Each of these four factors are shown to have a pronounced effect, sometimes reducing the strength by a factor of two or more. Cohesive zone and finite element modeling are employed to gain insight into these length scale effects.* Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract No. DE-AC04-94AL85000.

3:10 PM
Theta-like Specimens to Determine Tensile Strength at the Micro Scale: *Michael Gaither*[1]; Frank DelRio[1]; George Quinn[1]; Richard Gates[1]; Robert Cook[1]; [1]National Institute of Standards and Technology

Components of micro- and nano-electromechanical systems are typically formed via lithographic and etching processes that are known to leave residual surface features, stresses, and chemistry that ultimately control component strength and thus device performance and reliability. In order to measure how these surface characteristics interact with applied loads and deformations to induce failure and truncate lifetime, mechanical test structures are required to measure mechanical properties at ultra-small length scales. Here we describe a new "theta-like" geometry for micro-scale tensile strength measurement that allows for direct assessment of surface effects on strength. Specimens were formed from silicon-on-insulator wafers via deep reactive ion etching (DRIE) and tested with an instrumented indenter. The experimental results were interpreted via finite element models to extract fracture strength. Fracture strengths as great as 3 GPa were observed, with fracture initiating at DRIE etch pits. Statistical and fractographic analyses verified the existence of two processing-induced flaw populations.

3:25 PM
Fracture Behavior of Partially-Sintered Ceramics for Electrochemical Cell Applications: *Xiaoxing Liu*[1]; Christophe Martin[1]; Gerard Delette[2]; [1]Grenoble-INP; [2]CEA-Grenoble

The discrete nature of the fracture behavior of partially sintered ceramic powders is addressed using 3D Discrete Element Method simulations (DEM). Small samples, typically 30 µm thick, which are typical of electrochemical cell applications, are first prepared numerically by partially sintering around 20,000 micronic particles. Solid contacts formed during sintering are then modeled as brittle elastic bridges that may break under tension or shear. The numerical samples obtained from this sintering stage are submitted to tensile and compressive tests up to failure to obtain the fracture strength as a function of porosity and pertinent microstructural features at the particle length scale. Using these tests, we also investigate the effect of large pores on fracture. The formation and propagation of cracks originating from the thermal mismatch between the porous ceramic and a dense substrate is finally treated. Whenever

possible, simulation results are compared to experimental data from the literature.

3:40 PM

Failure Analysis of Audio Connectors Component Using X-Ray 3D Technology: *Daniele Rolim*[1]; Iramylson Freitas[1]; Idelcio Cardoso[1]; Ocileide Silva[1]; [1]Nokia Institute of Technology

This work proposes to investigate a critical assembly problem in audio connectors using non destructive method with both 2D and 3D X-Ray and destructive method with SEM. The main trouble when 2D analysis is used is the material overlay. This can induce to incorrect conclusions. Analysis in x-ray with 3D image can take a too long time during process of image acquisition but the result shows better precision to distinguish components interconnections. Each sample has taken around 40 minutes to acquire using 120kV and 60µA for tension and currency respectively. It was detected that proximity of some wires with solder of another connection can produce a friction between them, so the risk of short circuit arises, as some wires that composed one out of four connections are worn and/or broken. The complimentary analysis with SEM detected that either the wires insulating layer or most of the whole connection was damaged.

3:55 PM Break

4:10 PM Invited

Mitigation of Wear-Induced Failure of Microsystems by Vapor Phase Lubrication: *Michael Dugger*[1]; [1]Sandia National Laboratories

Since their inception about twenty years ago, microelectromechanical systems (MEMS) have been plagued with failures attributable to the interaction of moving surfaces. Designers have learned to avoid surfaces that touch or slide in order to maximize the reliability of their structures. While chemisorbed monolayers of organic molecules and non-liquid-based release processes have enabled successful fabrication of compliant structures, these approaches are not sufficiently robust to permit long term operation of devices with impacting and sliding surfaces. A recent advance in MEMS surface modification relies upon vapor phase lubrication. Using this approach, surface passivation is maintained via adsorption of a desired molecule from a low concentration source in the vapor phase. Unprecedented improvements in operating life result from maintaining surface passivation, and the elimination of wear particle generation. This presentation will discuss recent advances in MEMS lubrication and ongoing research aimed at developing a mechanistic understanding of vapor phase lubrication.

4:35 PM

In-Situ Microscale Fatigue Study to Determine the Effect of Microstructural Neighborhoods on Crack Initiation Mechanisms and Lifetimes: *Christopher Szczepanski*[1]; Sushant Jha[1]; Bob Wheeler[2]; James Larsen[3]; [1]UTC/AFRL; [2]UES/AFRL; [3]AFRL

Recent studies of fatigue crack-initiation mechanisms and their relationship to lifetime variability in bulk samples of Ti-6Al-2Sn-4Zr-6Mo has revealed that crack initiation results from a few different characteristic microstructural arrangements. These microstructural configurations, which vary in size and local crystallographic texture, can be ranked based on their frequency of occurrence and ultimately correlated with lifetime to failure. This paper will focus on a micro-scale fatigue study where micro-samples were machined in specific microstructural neighborhoods with textures that are believed to enhance their susceptibility to specific crack initiation mechanisms. A focused ion beam (FIB) microscope was used to machine micro-scale fatigue specimens containing microstructural arrangements of interest, as determined from the original study on bulk specimens. These specimens were then fatigued, in-situ, in a scanning electron microscope chamber. A study of deformation modes active in these specimens and correlation of microstructural arrangements to fatigue lifetime will be presented.

General Abstracts: Structural Materials Division: Fatigue and Fracture

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: Refractory Metals Committee, TMS: Titanium Committee
Program Organizers: Eric Ott, GE Aviation; Robert Hanrahan, National Nuclear Security Administration; Judith Schneider, Mississippi State University

Tuesday PM Room: 3A
February 16, 2010 Location: Washington State Convention Center

Session Chair: To Be Announced

2:00 PM

Fatigue Crack Growth Mechanisms of Long and Small Cracks in Structural Materials: *Anastasios Gavras*[1]; Christopher Lammi[1]; Diana Lados[1]; [1]Worcester Polytechnic Institute

Fatigue crack growth mechanisms of long and small cracks were investigated in cast and wrought aluminum and titanium alloys (as-cast A535, 6061-T6, and Ti-6Al-4V) with various microstructures. The effects of microstructure and bulk residual stresses on the fatigue crack growth response of each material were evaluated. Bulk residual stresses were introduced in the testing specimens through quenching. Long crack growth data were generated on compact tension specimens tested at low and high stress ratios (R=0.1 and R=0.7). Small crack growth testing was performed on surface flaw tension specimens at low stress ratio (R=0.1). Closure and microstructurally small crack growth mechanisms in samples with low and high residual stress levels were identified and related to the characteristic features of the materials. Corrective techniques compensating for residual stress, closure, and microstructural effects on fatigue crack growth data will be presented and discussed.

2:20 PM

Novel Methods for Microstructure-Sensitive Probabilistic Fatigue Notch Factor: *William Musinski*[1]; David McDowell[1]; [1]Georgia Institute of Technology

Traditional fatigue analysis schemes used for geometries with stress gradient fields (such as notches) have required extensive experimentation, estimation of fatigue failure distributions, and characterization of the notch effect via a notch root fatigue strength reduction factor, often called the fatigue notch factor, K_f. These experimental results are beneficial for life prediction of a given geometry and microstructure, but do not offer predictive insight into the underlying physical mechanisms that explain fatigue variability, size effects, and local gradient effects on fatigue damage. These notch effects are depicted through probability distributions of slip and small crack initiation processes that are informed by computational crystal plasticity simulations performed on realistic microstructures of materials used for aircraft gas turbine engines. Finally, the concept of a probabilistic, microstructure-sensitive fatigue notch factor, $K_f^{\backslash 956}$, is introduced that considers effects of nonlocal notch root plasticity, microstructure variability, and the probability of crack formation.

2:40 PM

Effect of Low-Temperature Overload on Fatigue Crack Growth Retardation: Sai Kumar[1]; Jyoti Mohanty[1]; Bipin Verma[1]; *Prabal Ray*[1]; [1]National Institute of Technology, Rourkela

Exponential model can be used to model fatigue crack growth predict the a-N curve to calculate residual life without integration of fatigue crack growth rate curve. In the present investigation an attempt has been made to estimate the fatigue life and retardation parameters of aluminium alloy components subjected to spike overload at subzero temperature using an Exponential Model. The specific growth rate in the model changes with loading condition and crack length. It has been expressed through dimensionless groups ($\Delta K/K_C$), (K_{max}/K_C) and (σ_y/E), and correlated with overload temperature.It is concluded that (i) an Exponential model can be effectively used to determine fatigue life,

(ii) overload at low temperature enhances retardation effect, (iii) presence of a large number of secondary cracks in the overload affected tear zone due to low temperature environment may be responsible for enhancing the retardation effect by reducing the available ΔK for primary crack extension.

3:00 PM

A Physical Interpretation of Basquin Relation: *Partha De*[1]; Wei Yuan[1]; Rajiv Mishra[1]; [1]Missouri University of Science and Technology

Basquin's empirical relation expresses σ_a the stress amplitude in a fully reversed fatigue test as an explicit function of N_f the number of load reversals to failure. The relation involves two parameters:'σ_f' the fatigue strength coefficient and 'b' the fatigue strength exponent. The fatigue strength coefficient strongly correlates to true fracture strength of the material in monotonic tensile test, while b generally varies within a small range of -0.05 to -0.12. Using friction stir processing a range of microstructures were produced with different combinations of strength leading to corresponding changes in S-N characteristics. The change of fatigue constants at different length scales of microstructure and variation in strengthening mechanism is utilized to give a physical significance to Basquin relation.

3:20 PM

Effect of Hydrogen on Crack Nucleation in 316 Stainless Steel under Rotating Beam Fatigue Loading Conditions: *Douglas Matson*[1]; Christian Skipper[1]; Gary Leisk[1]; Anil Saigal[1]; [1]Tufts University

Alloy 316 stainless steel samples were tested using a rotating beam fatigue test machine. Samples were either as-machined (uncharged) or precharged in hydrogen gas to obtain a concentration of 109 ppm. Cumulative damage was determined by counting the number and size distribution of cracks following sample fatigue failure using dye-penetrant and sample sectioning. Hydrogen precharged specimens have fewer and longer cracks than non-charged specimens for each alloy. The average number of cracks per specimen at the 500 MPa stress level was 14.67±2.08 for non-charged specimens and 5±1 for hydrogen-charged specimens. Total crack surface area was also determined by summing the cumulative area of all cracks in a sample for both precharged and non-charged condition and this damage area at failure was on the order of the gage area. These results are consistent with previous results showing hydrogen retards crack nucleation but accelerates crack growth for this alloy system.

3:40 PM Break

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S-N Fatigue and Fatigue Crack Propagation Behaviors of High Manganese Steels: *Jaeki Kwon*[1]; Sanguk Jin[1]; Sangshik Kim[1]; [1]Gyeongsang National University

Austenitic manganese steels with more than 13% manganese addition have been gathering a great interest particularly in ship-building and architectural industries due their excellent mechanical properties and weldability. Despite the interest, fatigue behavior of high manganese steels, including microstructural influences and fatigue crack initiation mechanism, have not been well established. In this study, uniaxial S-N fatigue and fatigue crack propagation (FCP) behaviors of high manganese steels were studied and the results were compared to those of TMCP steels with similar yield strengths. The present high manganese steels showed enhanced resistances to FCP in low stress intensity factor regime. Abnormally high resistance to S-N fatigue was observed for the high manganese steels in high and intermediate stress ranges probably due to extremely high strain hardening. Mechanism(s) associated FCP and S-N fatigue for high manganese steels were discussed based on detailed SEM micrographic and fractographic observations.

4:10 PM

Effect of Ca, Mg and Ti-Mg Addition on the Impact Toughness of Heat Affected Zone in Low Carbon Steel: *Jianghua Ma*[1]; Dongping Zhan[1]; Zhouhua Jiang[1]; Jin Yu[1]; Jicheng He[1]; Haijun Shen[2]; [1]Northeastern University; [2]Baoshan Iron & Steel Co., Ltd.

In order to understand the effects of deoxidants such as calcium, magnesium and titanium on the impact toughness of heat affected zone, three low carbon steels deoxidized by Ca, Mg and Ti-Mg were obtained respectively. After smelting, forging, rolling and welding simulation, the effects of Ca, Mg and Ti-Mg addition were studied. The inclusion characteristics of samples before welding and the fracture pattern of the specimens after the Charpy-type test were respectively analyzed using optical microscope and scanning electron microscopy. The following results were found. The density of inclusion is raised and the average diameter is decreased after adding alloys, especially when the Ca and Ti-Mg alloys were added, but the density adding Ca decreases 71.8% after 1 hour. The addition of Ti-Mg can enhance the impact toughness of the HAZ after welding simulation. The maximal value of the impact toughness is 66.5J/cm2. And the enhancing mechanism is discussed.

4:30 PM

Development of Layer-Integrated Steels with Superior Strength-Ductility Combination: *Shoichi Nambu*[1]; Masato Michiuchi[1]; Junya Inoue[1]; Toshihiko Koseki[1]; [1]The University of Tokyo

Variety of extraordinary high strength-ductility combinations, tensile strength above 1.3GPa and uniform elongation above 20%, could be achieved by layer-integration method using commercial steels.Multilayered (layer-integrated) steel composites consisting alternating layer of martensitic steel and austenitic steels were fabricated by controlling layer thickness and volume fraction of each component. Type 420J2 stainless steel, which shows ultra high strength in as-quenched condition was prepared for martensite layer, and three types of austenitic stainless steels, Type 304, 304N2 and 301 which have different work hardening exponent and yield strength, were prepared for austenite layer. The fracture toughness of Type 420J2 was controlled by heat treatment. It was demonstrated that variety of extraordinary high strength-ductility combinations could be obtained by controlling the geometrical and materials parameters, such as fracture toughness of martensite, work hardening exponent of austenite and strength ratio between components.

4:50 PM

Fatigue Crack Growth Behavior in a Monocrystalline Ni-Based Superalloy: *Clarissa Yablinsky*[1]; Katharine Flores[1]; Michael Mills[1]; James Williams[1]; [1]The Ohio State University

Historically, Ni-based monocrystal superalloy turbine blades have been limited by creep resistance. With modern airfoil designs and longer service times, fatigue resistance can also be a limiting factor. We examined the fatigue crack growth characteristics of monocrystalline Ni-based superalloy René N5. Test temperature was varied in order to examine the effects of deformation mode, plastic zone size, recovery, and other time dependent processes. The crack path observed at 550°C was highly faceted on the macro-scale, but the facets were smooth and mirror-like. In contrast, the crack path at 850°C was flat, consistent with an increase in crack growth rate with temperature, but with significant surface roughness. Scanning electron microscopy was used to determine crack path, fracture topography, and γ/γ' morphology along the crack wake and in the bulk material. Dislocation arrangements were examined via transmission electron microscopy in order to characterize the mechanisms of damage accumulation during fatigue crack growth.

5:10 PM

The Role of Non-Planar Deformation in Cyclic Softening Following Low Cycle Fatigue of a Ni-Based Superalloy: *Patrick Phillips*[1]; Raymond Unocic[1]; Libor Kovarik[1]; Dan Wei[2]; David Mourer[2]; Michael Mills[1]; [1]Ohio State University; [2]GE Aviation

Cyclic softening is a common material response during low cycle fatigue (LCF) over a wide range of testing temperatures. The polycrystalline Ni-based superalloy explored in this study exhibits this softening behavior, but only at elevated temperatures. This tendency is likely a result of activating new deformation modes depending on time and temperature. Using transmission electron microscopy characterization methods, the evolution of damage mechanisms was studied on specimens interrupted after a limited number of cycles. Both fine and coarse precipitate microstructures were examined, corresponding to a fast or slow cool, respectively, from the gamma prime solvus temperature. The operative deformation mechanisms are correlated with the precipitate structure, number of cycles, and testing temperature. The observed deformation modes include paired APB shearing, intense dislocation bands on {111} planes, and eventual cross-slipping processes between {111} and {100} planes. A rationale for the observed mechanisms in terms of time-dependent damage processes will be discussed.

6:00 PM

Computational Thermodynamics, Neural Networks and Genetic Algorithms: Tools to Design Creep Resistant and Weldable Superalloys: *Franck Tancret*[1]; [1]Université de Nantes

One drawback of many nickel-base superalloys is their poor weldability. In particular, cracking can occur in the mushy zone during solidification, in the so-called Brittle Temperature Range (BTR). Another type of cracking is due to the formation of intergranular liquid films, by the liquation of low melting point phases like carbides and/or intermetallics. In this work, computational thermodynamics (Thermo-Calc) is used to predict the occurrence of these types of cracking, and to design Ni base alloys by minimizing both the BTR and the risk of γ' liquation cracking, while keeping a good phase stability and good mechanical properties in the expected service temperature range. Optimized alloy compositions are determined by an automated maximization of stability and weldability, using genetic algorithms. Also, the creep rupture resistance is estimated through the regression of existing data, using neural networks. Predictions are then used to propose new weldable and creep-resistant superalloys.

Global Innovations in Manufacturing of Aerospace Materials: The 11th MPMD Global Innovations Symposium: Innovations in Primary and Secondary Forming - Titanium

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Shaping and Forming Committee, TMS: High Temperature Alloys Committee

Program Organizers: Deborah Whitis, General Electric Company; Thomas Bieler, Michigan State University; Michael Miles, BYU

Tuesday PM Room: 306
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Dan Sanders, Boeing Corporation; Lee Semiatin, AFRL-RX

2:00 PM Invited

Severe-Plastic Deformation and Superplasticity of Two-Phase Titanium Alloys: *Lee Semiatin*[1]; G. A. Sargent[2]; A. K. Ghosh[3]; G. A. Salishchev[4]; C. S. Lee[5]; [1]US Air Force Research Laboratory; [2]UES, Inc.; [3]University of Michigan; [4]Belgorod State University; [5]Pohang University of Science and Technology

A number of severe-plastic-deformation (SPD) methods have been developed to impart an ultrafine grain size during cold or warm primary processing of metallic materials. These techniques include high-reduction flat rolling, redundant or multi-directional forging, and multi-pass equal-channel angular extrusion. The microstructures developed in single-phase materials by such means and their effect on secondary superplastic (SP) forming will be summarized. With this as background, the balance of the talk will focus on the nature of the microstructure developed via SPD of two-phase titanium alloys, such as Ti-6Al-4V, and the effect of such microstructures on subsequent low-temperature SP deformation. The importance of controlling the size of alpha-particles (versus that of the alpha grains within each particle) in order to optimize low-temperature SP flow during forging or sheet forming will be discussed. It will be shown that microstructure evolution via static coarsening, dynamic coarsening, or dynamic spheroidization can each have a major effect on SP flow behavior. The specific role played by each mechanism depends on the microstructure developed in billet or sheet during primary processing via SPD.

2:30 PM

Processing, Structure and Properties of Safety Critical Titanium - An Engineering Perspective: *David Rugg*[1]; D. Furrer[2]; M. Glavicic[2]; [1]Rolls-Royce Plc; [2]Rolls-Royce Corporation

Advanced experimental techniques are available that allow scientists to routinely assess intergranular strain evolution, interface structures and mechanical properties at length scales of a few microns. Similarly, physically based models are reaching a level of maturity that allow use for niche industrial application. This paper reviews the industrial need for fundamental research of this type in the areas of processing, microstructural characterisation and mechanical behaviour. An overview of key future requirements in terms of measurement, representation and modelling will also be presented. Safety critical components will be used as a case study representing a combination of advanced manufacturing routes and complex load regimes in service.

2:50 PM

The Effect of Macrozone Formation during Thermo-Mechanical Processing on the Fatigue Response of Commercial Ti-6Al-4V Products: *M.R. Bache*[1]; C. Pleydell-Pearce[1]; [1]Swansea University

The development of regions of common crystallographic orientation during thermo-mechanical processing of α/ β titanium alloys is now recognised as a major controlling factor for subsequent mechanical properties. In particular, a process of stress redistribution driven by inhomogenous plasticity acts as a key mechanism behind fatigue crack initiation. Recent efforts have focussed on the identification of effective structural units (ESUs) in commercially processed products and their role during the fatigue process. Various product forms of the same alloy, Ti-6Al-4V, will be assessed to identify differences in texture and associated fatigue performance, utilising a variety of plain and notched specimen designs. Implications for service applications will be discussed.

3:10 PM

In Situ Observation of Texture Evolution during Rolling and Recrystallisation of Ti-6Al-4V: *Jonnathan Warwick*[1]; R. J. Talling[2]; M. Preuss[2]; D. Dye[1]; [1]Imperial College London; [2]Manchester University

A fine primary a microstructure is produced in bimodal Ti-6Al-4V by the recrystallisation of material that has previously been deformed in the a+b field, i.e. by recrystallisation (RX) of deformed a laths. Here we track the evolution of texture and microstructure during RX by using in situ hot stage backscatter electron imaging and in situ synchrotron X-ray diffraction, both with ~1s time resolution. These measurements are complimented by ex situ texture measurements and EBSD. The understanding generated of the evolution of these microstructures allows conclusions about process optimisation and the effect of RX ontexture to be drawn.

3:30 PM

The Effect of Beta Grain Growth on Alpha Variant Selection in Ti-6Al-4V: G. Obasi[1]; S. Birosca[1]; *Michael Preuss*[1]; [1]University of Manchester

In the current study, the texture evolution and the role of ß grain growth on variant selection in Ti-6Al-4V have been investigated. Three samples from different processing routes (Conventional Ingot Metallurgy (IM), Powder Metallurgy (PM) and Ti-6Al-4V with 0.39wt%Y) were uni-directionally rolled at 950ºC, followed by recrystallization for 8h and cooled at 1ºC/min to produce similar equiaxed microstructures and textures for the three alloys. The recrystallized samples were subsequently heat treated above beta transus temperature and then cooled at 1ºC/min to allow diffusional transformation. The microstructural analysis including beta grain reconstruction indicates the largest beta grain growths for the PM material followed by the IM and Yttrium containing alloy. Electron back scatter diffraction (EBSD) texture analysis shows that the major texture component in all recrystallized samples were the {11-20} <0001>. However, the PM material showed the most significant difference in terms of measured texture intensity compared to the calculated on a basis of equal variant probability followed by the IM material and the Ti-6Al-4V with 0.39wt%Y. The results suggest that variant selection can be related to grain growth and possibly the development of specific beta grain boundaries with a common (110) normal direction.

3:50 PM

The Relevance of Twinning during Deformation of Ti-6Al-4V: D.G. Leo Prakash[1]; R. J. Moat[1]; R. Ding[2]; I. Jones[2]; P. Withers[1]; J. Quinta da Fonseca[1]; *Michael Preuss*[1]; [1]University of Manchester; [2]University of Birmingham

A detailed study has been carried aimed at providing unambiguous evidence of twinning in Ti-^{6}Al-^{4}V during moderate (4 - 14%) uniaxial compression deformation at room temperature using a slow strain rate (10^{-6} s^{-1}). This is achieved by generating a strongly texture and recrystallised microstructure with strong basal texture components in the transverse and normal direction but none in the rolling direction. By subsequently compressing the material along the rolling direction dramatic texture changes were found after only 6% plastic deformation with a strong basal texture component in the rolling direction. Even though electron channelling contrast imaging and transmission electron microscopy were able to provide direct evidence of tensile and compression twins in the deformed material it was also evident that the fraction of twins

appeared far too low to explain the dramatic texture change during deformation. Detailed orientation mapping using EBSD identified an increasing number of entire grains having their c-axis aligned with the loading direction in the deformed material. The root cause for such complete grain rotation can only be speculated at the moment but it is suggested that individual grains require high activation energy for twinning but a low energy for the growth of the twin. It is further speculated that such twinning of entire grains might act as a recovery process of grains.

4:10 PM Break

4:20 PM
Dislocation Transmission through Interphase Boundaries in Ti-6Al-4V: R. Ding[1]; J. Gong[2]; A.J. Wilkinson[2]; *I. P. Jones*[1]; [1]University of Birmingham; [2]Oxford University

Dislocation transmission through interphase boundaries in Ti-6Al-4V has a significant effect on mechanical properties. Triangular cross-section micro-cantilevers have been machined using FIB from a commercially processed Ti-6Al-4V ingot and deformed using a nanoindenter. Each cantilever contains several alpha lamellae separated by thin fillets of beta. Slip occurs as well defined slip bands, which cross the beta fillets. TEM specimens in various orientations with respect to the cantilever (and slip bands) have been machined (again using FIB) from the deformed cantilever and the processes of slip nucleation and transmission examined.

4:40 PM
The Application of Fine Grain Titanium 6Al-4V for Superplastic Forming and Superplastic Forming and Diffusion Bonding of Aerospace Products: *Larry Hefti*[1]; [1]The Boeing Company

Superplastic Forming (SPF) of titanium 6Al-4V, with a standard grain size of about 8 µm, is typically performed at around 1650° to 1700°F (900° to 920°C). VSMPO in Russia has developed a fine grain version of the 6-4 alloy, with a grain size of about 1 µm, which is able to be superplastically formed at around 1425°F (775°C). Since this material diffusion bonds to itself as well as other standard grain size titanium alloys at this temperature, Superplastically Formed and Diffusion Bonded (SPF/DB) hardware can be produced. There are several advantages to using this lower forming temperature including a smaller amount of alpha case is developed on parts so there is less to remove, longer press platen and heater life, and less oxidation of the tool surface. In order to take advantage of these improvements, this material is currently being used in the production of SPF and SPF/DB aerospace components.

5:00 PM
Manufacturing of β-Titanium Ti-10V-2Fe-3Al Spin-Extruded Hollow Shafts for High Strength Power Train Applications in Aerospace and Automotive Industries: *Christian Machai*[1]; Dirk Biermann[1]; [1]Technische Universität Dortmund

The aerospace and automobile sector strives to increase the efficiency of turbines and internal combustion engines. Fuel efficient engines demand components with a good combination of low mass and appropriate mechanical properties of the material used. Components which consist of a spin-extruded hollow shaft made of β-titanium alloy offer high specific strength in addition to good fatigue and corrosion resistance. The presented process route for manufacturing the hollow β-titanium shaft consists of a complex incremental forming-process followed by a turning operation to remove surface damages induced by the preceding forming processes. The machining operation of the undeformed titanium raw material and the deformed near-net-shape hollow shaft is investigated to identify the optimum process route for manufacturing an undamaged workpiece. Therefore, for different tool materials and machining strategies the static and dynamic process forces, tool wear, surface quality and metallographic samples of the undeformed and deformed peripheral workpiece area are analyzed.

5:20 PM
Superplastic Forming and Diffusion Bonding Process Design for Aerospace Component: *Yong-Nam Kwon*[1]; [1]Korea Institute of Materials Science

Main interest in modeling superplastic forming(SPF) and diffusion bonding(DB) process is to predict an optimum pressurization schedule to control strain rate and to predict a resulting thickness distribution of forming parts. Finite element method(FEM) could be a useful guide to optimize SPF/DB processes. FEM is used to model the SPF/DB process of box and sandwich type structures to predict the pressure-time curve. Also, other process variables were investigated for process optimization. In order to eliminate defects of the part such as folding or excessive thinning, a new pressurization scheme is proposed. Contrary to the conventional one-step pressurization, which causes the folding, two-step pressurization can eliminate the folding. SPF/DB process was carried out with of Ti-6Al-4V sheet. SPF/DB formed part was cut down into pieces to check various features like dimensional accuracy, check thickness profile, hydrogen contamination, microstructure evolution and tensile properties.

5:40 PM
Application of Statistical Continuum Mechanics to Guide Processing of Aerospace Materials: *Dongsheng Li*[1]; Hamid Garmestani[1]; [1]Georgia Institute of Technology

To guide processing of aerospace materials, inverse materials design manage the information flow from design to properties to microstructure to processing. Using a new representation method to characterize statistical correlation in microstructure, a set of spherical harmonics weight is utilized to represent microstructures in our work. Multi scale modeling including statistical continuum mechanics is used to predict microstructure evolution during processing. We also developed statistical continuum mechanics models to calculate microstructure determined properties, including elastic, plastic, electronic, transfer and magnetic properties of aerospace materials. The relationship among microstructure, properties and processing is established using statistical continuum mechanics. Explicit mathematical solution may be calculated to solve detailed processing steps on how to achieve target microstructure with desired properties. Examples on titanium and magnesium alloys will be presented to demonstrate the capability of statistical continuum mechanics on manufacturing of aerospace materials.

Heterogeneous Nucleation and Initial Microstructure Evolution in Alloys and Colloids: Experiment II

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS/ASM: Phase Transformations Committee
Program Organizers: Rainer Schmid-Fetzer, Clausthal University of Technology; Heike Emmerich, RWTH Aachen University; Frans Spaepen, Harvard University; Martin Glicksman, University of Florida; John Perepezko, University of Wisconsin, Madison

Tuesday PM Room: 614
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Ma Qian, University of Queensland; John Perepezko, University of Wisconsin - Madison

2:00 PM Invited
Grain Formation: The Interdependence between Grain Growth and Nucleation: *David StJohn*[1]; Ma Qian[1]; Mark Easton[2]; [1]CAST CRC, University of Queensland; [2]CAST CRC, Monash University

A description of grain formation is presented that links the initial growth of a grain to its heterogeneous nucleation during the initial transient. It is proposed that, when potent nucleant particles are present, each nucleation event occurs very early in the initial transient. The grain size is determined by two components: (1) the distance a previously nucleated grain must grow in order to establish sufficient constitutional undercooling to be able to nucleate the next grain and (2) the additional distance to the most potent available nucleant particle that actually nucleates the new grain. The relative significance of these two components is illustrated by comparing magnesium and aluminium alloy systems. It will be further shown that the initial growth of a grain is spherical and that further nucleation events usually occur before growth breaks down to globular and dendritic morphologies. The factors affecting the limit of spherical growth are explored.

2:25 PM Invited
Modeling Transient Growth of Undercooled Solid Nuclei in the Melt: *Markus Rettenmayr*[1]; Marcel Fink[1]; [1]Friedrich-Schiller-University Jena

The growth conditions of undercooled solid nuclei in a liquid are hardly accessible experimentally due to the involved small length scale and the initial rapid growth velocity. However, some insight can be obtained through modeling. Standard solidification models applying continuum theory can describe growth only starting from the point where local equilibrium is reached, since off-equilibrium models generally assume a steady-state which is not applicable for the growth of undercooled nuclei. Early stages of growth can be described with a continuum model that incorporates recent progress in the description of interfacial thermodynamics. Such a model is applied in the present study on binary aluminum alloys. It is shown that depending on the initial undercooling a nucleus can theoretically start growing with velocities in the order of m/s. Local equilibrium is reached after a time span in the order of microseconds. Further details regarding growth conditions, nucleus size etc. will be discussed.

2:50 PM
Investigation of Heterogeneous Nucleation of the Beta Phase in the System Ti-Al: *Daniel-Hendrik Gosslar*[1]; Christian Hartig[1]; Robert Guenther[1]; Ulrike Hecht[2]; Ruediger Bormann[1]; [1]Hamburg University of Technology; [2]Access e.V.

The alloy system Ti-Al, in the composition range of 43 to 46 at.% Al, is suitable to study heterogeneous nucleation from the melt. Recent experiments proved directly heterogeneous nucleation of the alpha phase on in-situ formed borides. Additionally, from these experiments heterogeneous beta nucleation on ex-situ added borides is strongly suggested. The aim of this work is to investigate heterogeneous nucleation of the beta phase in more detail. For this purpose inoculation experiments are carried out for the Ti-45Al at.% alloy at different cooling rates. As appropriate inoculants titanium diboride particles are used. The resulting grain size in the as cast microstructures will be compared to free growth model calculations of the beta grain size. This comparison and atomic mismatch calculations in view of nucleation barrier are used to understand the microstructure evolution under the influence of heterogeneous beta nucleation.

3:10 PM
Heterogeneous Nucleation and Microstructure Formation in Peritectic Al-Ni Alloys: Evelyn Doernberg[1]; Ricardo Siquieri[2]; Hailin Chen[1]; Heike Emmerich[2]; *Rainer Schmid-Fetzer*[1]; [1]Clausthal University of Technology; [2]RWTH Aachen

The Al-Ni system is an important model system for investigating heterogeneous nucleation and microstructure formation during peritectic solidification. Peritectic reactions with both types II and III are systematically investigated by a close coupling of phase-field (PF) simulation, thermodynamic modeling, and various experimental methods. Microstructure evolvements during directional solidification were experimentally studied and parameters including the secondary dendrite arm spacing, the thickness of the peritectic envelope, and the volume percent of each phase were semi-quantitatively estimated. All the experimental results were reasonably reproduced by simulations. Using the internal droplet technique and precise calorimetric experiments, together with the PF calculation, nucleation undercooling, nucleation rates and mechanisms of nucleation will be preliminarily evaluated. The role of peritectic transformation and direct precipitation of the peritectic phase for both types of peritectic reactions is discussed. This work is supported by the German Research Foundation (DFG) in the Priority Program "DFG-SPP 1296".

3:30 PM
Heterogeneous Nucleation in Liquid Immiscible Alloys: *Markus Koehler*[1]; Lorenz Ratke[1]; [1]German Aerospace Center

Alloys being immiscible in the liquid state decompose into two liquid phases during cooling through the miscibility gap. In contrast to solidification where the solid phase is generally nucleated at inocculants or container walls, liquid immiscible alloys are thought to decompose via homogeneous nucleation. The process is badly investigated. Therefore we study heterogeneous nucleation in Al-base immiscible alloys (Al-Pb, Al-Bi) induced by inocculants like TiB2, TiC and ZrO2. We present experimental results on Al-Pb alloys cooled with different amounts of TiB2 and ZrO2 through the miscibility gap. The microstructrue is analysed in 2D with SEM and light microscopy and also in 3D using x-ray tomography. The experiments show a pronounced effect of the inocculants. The average Pb drop size changes as well as the size distribution. The experimental results are discussed with respect to classical theories of heterogeneous nucleation and newer models of nucleation and growth by Greer and co-workers.

3:50 PM Break

4:10 PM
Characterization of the Initial Stages of Phase Separation by Atom Probe Tomography: *Michael Miller*[1]; Ai Serizawa[1]; [1]ORNL

The changes that occur in the solute distributions during homogeneous or heterogeneous nucleation growth and during spinodal decomposition may be characterized by atom probe tomography in model and complex engineering alloys. Statistical analysis of the distribution of the solute atoms in the three-dimensional data with the maximum separation envelope method and simulations enables clusters and embryos as small as 5 atoms to be detected, and their size, composition, morphology and number density to be estimated as the microstructure evolves. In this study, the initial phases of phase separation of the remarkably stable Ti-O-Y nanoclusters in a nanostructured ferritic steel will be discussed.This research was sponsored by the U.S. Department of Energy, Division of Materials Sciences and Engineering; research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

4:30 PM
Nucleation of Strengthening Dispersions in a High-Strength Low-Carbon Steel: *Mike Mulholland*[1]; David Seidman[1]; [1]Northwestern University

Steels often rely on the precipitation of several phases to derive their mechanical properties, which results in very complex microstructures. Blast Alloy (BA) 160 is a high-strength low-carbon steel that relies on the precipitation of copper and M2C carbide precipitates in a martensitic matrix for its high yield strength of 160 ksi. Since the copper and M2C precipitates are less than 5 nm in radius, 3-D local-electrode atom-probe (LEAP) tomography is employed to study their precipitation as a function of time. The majority of the copper and M2C carbides are co-located spatially at peak aging times and beyond, suggesting the heterogeneous nucleation of M2C precipitates on copper precipitates. The nucleation behavior of both copper precipitates and M2C carbides is investigated to determine whether or not this is the case.

Hume-Rothery Symposium: Configurational Thermodynamics of Materials: Session IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Chris Wolverton, Northwestern University; Mark Asta, University of California, Davis; Gerbrand Ceder, Massachusetts Institute of Technology (MIT)

Tuesday PM Room: 212
February 16, 2010 Location: Washington State Convention Center

Session Chair: To Be Announced

2:00 PM Invited
Chasing Exotic Binary Alloy Compounds: The Necessary Synergy of Cluster Expansion and High-Throughput Methods: *Stefano Curtarolo*[1]; Gus Hart[2]; Ohad Levy[3]; [1]Duke University; [2]Brigham Young University; [3]NRCN

Predicting the stable crystal structures of alloys from their components is a major challenge of current materials research. Ab initio methods explore the phase stability landscape of binary alloys by calculating the formation enthalpies of a large number of structures, and identifying the minima at various component concentrations. Major methods of this type are cluster expansion (CE) and high-throughput ab initio calculations (HT). The CE explores structures on specific types of lattices while the HT method explores experimentally-known structures representing all crystal systems. The CE may find derivative superstructures missed by the HT but is not applicable off-lattice. Combining and reciprocally informing both methods resolve their respective drawbacks. We demonstrate this in a several technologically important Hf, Rh alloy systems. These results emphasize the complementary strengths of the CE and HT methods and the need for using both in searching for new stable compounds in metallic systems.

2:30 PM
Informatics Applications to Electronic Structure Calculations: *Krishna Rajan*[1]; Scott Broderick[1]; Tao Wang[1]; [1]Iowa State University

This presentation provides a demonstration of the types of classification and prediction possible when linking statistical learning methods with electronic structure calculations. Examples include the development of new methods to model the density of states and to explore methods of establishing correlations between crystal structure and crystal chemistry from data mining from DFT calculations.

2:50 PM
The Prediction of Crystal Structure by Combining Machine Learning Knowledge Methods with First Principles Energy Methods: *Gerbrand Ceder*[1]; [1]Massachusetts Institute of Technology (MIT)

De Fontaine's key work in cluster expansions enabled the ab initio prediction of structures with common underlying lattice. Finding ground states in topologically unconstrained space is still very difficult due to the complexity and high dimensionality of the coordinate space. As an efficient solution to this problem we have developed machine learning approaches that extract knowledge from a large set of experimental and computed information, and used these to rapidly direct accurate quantum mechanical techniques to the lowest energy crystal structure of a material. Knowledge is captured in a Bayesian probability network that relates the probability to find crystal structure at a given composition to structure and energy information at other compositions. We show that this approach is highly efficient in finding the ground states of binary metallic alloys and can be easily generalized to more complex systems. Using this approach we have already identified more than 400 new compounds.

3:10 PM Invited
Predicting Solid - Aqueous Equilbria for Materials Design: *Kristin Persson*[1]; [1]LBNL

Ab initio methods have proven very powerful in accurately predicting solid, surface and nano-scale properties. However, many technological problems involve materials in equilibrium with liquids, which are less straightforward to describe from first principles. We have addressed this issue for aqueous environments in an efficient manner by combining experimental data with ab initio calculations through a common point of reference system. We will briefly outline the method and show several examples of applications. In the first example we develop nanoparticle Pt Pourbaix diagrams to study size-dependent dissolution, relevant for fuel cell degradation mechanisms. Furthermore, we apply the formalism to the olivine LiFePO4 to predict the change of particle shape under different pH and potential conditions in order to facilitate targeted synthesis by hydrothermal methods. Finally, we map out solid state stability for oxides, in order to identify rules of stabilization under severe corrosive aqeuous conditions.

3:40 PM Break

4:10 PM Invited
Transfer Matrix Approach to Quasi-1D Nanostructures with Cluster Interatomic Interactions: Vasyl Tokar[1]; *Hugues Dreyssé*[2]; [1]Institute of Magnetism of NAS and MES; [2]Universite de Strasbourg, CNRS

Two types of epitaxial nanostructures (ENS) can be described in the framework of the lattice gas model: the ENS consisting of atoms of the same kind and filling some region on the surface in a submonolayer coverage regime or the ENS composed of a monolayer-thick binary alloy. ENS of this type include, in particular, such technologically important structures as nanowires (continuous quasi-1D ENS) and quasi-1D chains of magnetic islands for potential use in memory devices. Because of the quasi-1D character of the structures, they can be treated exactly within the transfer matrix approach (TMA) recently developed in the theory of protein folding and generalized by us to the theory of strained epitaxy. An important advance with respect to the traditional TMA is that cluster interactions within contiguous atomic chains of arbitrary length as well as the solid on solid restriction can be treated exactly within this approach.

4:30 PM
Towards a First-Principles Understanding of the Iron Phase Diagram: *Fritz Körmann*[1]; Alexey Dick[1]; Blazej Grabowski[1]; Tilmann Hickel[1]; Jörg Neugebauer[1]; [1]Max-Planck-Institut für Eisenforschung

Accurate tools for the theoretical prediction of material properties, such as the specific heat capacity or free energy, become increasingly important for a computational design of new materials. Density functional theory has emerged as one of the most powerful tools for such predictions. While originally developed and applied as a groundstate (T=0 K) theory, the combined concepts of DFT and finite temperature models evolved nowadays to a state making reliable and accurate predictions of a wide range of materials properties possible. In this talk we discuss the thermodynamic concepts capturing the vibrational, electronic, and magnetic excitations from DFT. To describe the magnetic contributions new methodologies based on the Heisenberg model were developed allowing for the first time an ab initio based quantitative prediction of the magnetic free energy [PRB 78, 033102 (2008)]. The predictive power of the proposed theoretical framework is verified by comparison with experimental data.

International Symposium on High-Temperature Metallurgical Processing: Smelting and Reduction Processes

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
Program Organizers: Jaroslaw Drelich, Michigan Technological University; Jiann-Yang Hwang, Michigan Technological University; Tao Jiang, Central South University; Jerome Downey, Montana Tech

Tuesday PM Room: 619
February 16, 2010 Location: Washington State Convention Center

Session Chair: Tao Jiang, Central South University

2:00 PM
Industrial Operation of JAE Nickel Smelting Technology at Jinchuan Nickel Smelter: Min Zhou[1]; Aidong Wan[1]; Guang Li[1]; *Ross Baldock*[2]; Harry Li[2]; [1]Jinchuan Non Ferrous Metals Corp.; [2]Ausmelt Ltd

A commercial nickel smelter using JAE nickel smelting technology has been successfully commissioned and put into the smooth operation at Jinchuan Non-ferrous Metals Corporation China since August 2008, after 2.5 years design and construction works. The paper provides details of this nickel smelting project, including the technical evaluation, demonstration plant investigation, engineering design, plant commissioning and operation. Emphasis has been given in comparison between the design targets and actual performance of the plant.

2:20 PM
Arc Plasma Smelting of Niobium Pentoxide towards Production of Nb Metal: *Bijan Nayak*[1]; Barada Mishra[1]; [1]IMMT

Smelting of niobium pentoxide was carried out in an extended arc plasma reactor using carbon as reductant.Carbon in the form of graphite was used as the reducing agent in the smelting. Niobium pentoxide powder with 98.5% purity and grain size below 63 micron, was taken as the starting raw material. Smelting of the charge (taken in the form of a mixture of oxide and carbon) was carried out in an arc plasma reactor at 100g scale for 15-20 min. The smelted product obtained in the form of chunk was characterized by XRD for identification of Nb and other impurity phases . Optical microscopy, SEM, EDS and micro Raman spectroscopy were employed to study the morphology and corroborate the occurrence of Nb. Chemical analysis of the product was done by ICPS and was found to contain 97.5% Nb with 86% maximum recovery.

2:40 PM
Carbothermic Reduction of Niobium Concentrate: *Joao Ferreira Neto*[1]; Flavio Beneduce Neto[1]; Cyro Takano[2]; [1]Institute for Technological Research - IPT; [2]University of Sao Paulo

Ferro Niobium is produced by aluminothermic reduction of the niobium concentrate. However, the niobium concentrate has impurities, such as phosphorus, lead and tin, which can contaminate the Ferro Niobium. Therefore, the niobium concentrate must be refined before aluminothermy. The carbothermic reduction of niobium concentrate was investigated as a method to promote the

impurities removal.The carbothermic reduction of niobium concentrate was investigated in self-reducing briquettes in temperatures between 1100 and 1250°C, aiming to promote the reduction of impurities, mainly phosphorus. The reduction was carried out using graphite as carbonaceous material. After reduction, the briquettes were evaluated in scanning electronic microscope.It was observed that the iron oxide of the niobium concentrate was simultaneously reduced with oxides of phosphorus, lead and tin, producing an alloy Fe-Sn-P, with phosphorus content up to 20%. Based on the results it was obtained an apparent activation energy of 296,3 +/- 29 kJ/mol.

3:00 PM

Volatilization of Antimonite in Nitrogen-Oxygen Atmospheres: *Rafael Padilla*[1]; Gustavo Ramirez[1]; Alvaro Aracena[1]; Maria Ruiz[1]; [1]University of Concepcion

The vaporization of antimonite (Sb_2S_3) was investigated in the temperature range 600-900°C in a conventional thermogravimetric apparatus. The study was conducted in inert (N_2) and oxidizing ($N_2 - O_2$) atmospheres under various gas flow rates. The results indicated a linear behavior of vaporization of the sample at temperatures above 700°C. Thus by using the vaporization kinetic constants, activation energy of 124 kJ /mol was determined for the vaporization in nitrogen gas. On the other hand, the weight loss due to volatilization of antimonite in the presence of oxygen occurs up to a certain extent depending on the temperature and partial pressure of oxygen. 20% of total vaporization occurred at 700°C while at 900°C the total vaporization was about 60%. The incomplete volatilization of antimonite is due to the formation of non-volatile oxides since the weight loss increases with a decrease in partial pressure of oxygen.

3:20 PM

The Optimization of the Coke and Agglomerte Quantity in Lead Production in "Water-Jacket" Furnace: *Ahmet Haxhiaj*[1]; Egzon Haxhiaj[2]; [1]University of Prishtina; [2]American University in Kosovo

Paper contains the analysis of technological process depending on the composition of lead (Pb) in agglomerate, and the theoretical and real rapport of coke consumption per tone of technical lead produced. The findings are based in the work of "Water-jacket" furnace in Trepça. Paper brings the results of theoretical and experimental studying, along with analytical and graphical analyses of regional thermal balance depending on the composition of load, and the coke quantity in load. While analyzing the technological process of technical lead production we have been searching for the growth of technical lead quantity produced depending on lead percentage in agglomerate and the air that is enclosed in the furnace. Moreover, we aimed to optimize the percentage of lead in agglomerate and the parameters of the process. Simultaneously, keeping the attention to the growth of technical lead produced, the minimization of energy consumption, and the minimization of environment pollution.

3:40 PM Break

3:55 PM

Influences of MgO on Roasting Properties of Iron Ore Oxidized Pellets: *Xiaohui Fan*[1]; Min Gan[1]; Tao Jiang[1]; Xuling Chen[1]; Lishun Yuan[1]; [1]Central South University

The roasting properties of iron ore oxidized pellets , such as the mechanical strength, the suitable roasting temperature and the remnant contents of FeO , are affected by adding MgO . As the contents of MgO increased, the strength of roasted pellets is decreased, the range of suitable roasting temperature is narrowed, and the remnant FeO of pellets roasted in high temperature is increased. According to the analysis of spectrum and microstructure, and combining the theory of solid reaction and the phase diagram, the main two reasons that affecting the roasting properties of pellets are that MgO dissolves in magnetite , and MgO reacts with Fe2O3 to form magnesium ferrite.

4:15 PM

Research on the Intensifying Reduction Technology Based on Mechanically Activated Ilmenite Ore: *Yufeng Guo*[1]; Hemei Liu[1]; Tao Jiang[1]; Guanzhou Qiu[1]; [1]Central South University

This text regarded Panzhihua ilmenite as the research object, systematically studied the influence law of reductive iron oxide affected by the ultra-fine ilmenite powder which was prepared from one part of raw material. The research showed that reduction process of ilmenite was accelerated by adding super-fines. The degree of metallization was investigated, and a metallization rate of 88.30% was obtained under the condition of superfine powder fineness 80%-0.010mm, Superfine powder /Ore 3:97, whereas no super-fines was 84.30%. The growth of reduction iron crystal in ilmenite iron oxide was investigated by light microscopy and scanning electron microscopy. Through the analysis, it was found that the metal iron content and micro-pore in reduced ilmenite which was added super-fines significantly increased compared with the control (without super-fines addition).

4:35 PM

Study on Reduction Roasting and Separation of Nickeliferous Laterite by Microwave Heating: Yi Lingyun[1]; *Huang Zhucheng*[1]; [1]Central South University

Nickeliferous laterite is difficult to achieve ideal indexes by conventional physical process. In this paper, the reduction roasting-magnetic separation of coal-containing nickeliferous laterite briquette was carried out by microwave heating and traditional roasting. The results show that traditional roasting has many difficulties such as needs higher temperature, longer roasting time and easily leads to lower reduction of the center. Reduction roasting by microwave radiation can heat the whole pellet simultaneously and give priority to heat coal particles and nickel minerals, which is conducive to the form of nickel-iron grains. The electromagnetic field effect of microwave can also improve the growing up and movement of Ni-Fe grains. The reduction roasting by microwave heating for 45min at power of 2kw can obtain the concentrate of 5.21% nickel and recovery of 72.01% while using traditional roasting for 150min at 1150°, the concentrate of 2.24% nickel and 30.96% recovery can be got.

4:55 PM

Study on Preparation of Titanium-Rich Material From Ilmenite by Reduction-Magnetic Separation Process: *Yufeng Guo*[1]; Dan Huang[1]; Guangzhou Qiu[1]; Tao Jiang[1]; [1]School of Minerals Processing and Bioengineering, Central South University

The preparation of Ti-rich material from Panzhihua-Xichang ilmenite in China was investigated by solid-state reduction-magnetic separation. The results indicated that high metallization of reduction product could be obtained by adding alkali metal additives under low reducing temperature, which realized the effective separation of iron and titanium. The ilmenite was added 3% additives and roasted at 1100° for 180 min, a Ti-rich material with 75% TiO2 and 90% recovery could be obtained by grinding-magnetic separation. Compared with no alkali metal additives, the reducing temperature was decreased from 1250°~1280° to 1100° and TiO2 grade increased by about 10 %. The mechanism research indicated that the additive had the function of strengthening solid diffusion and could reduce the effect of Mg2+ on the stability of Fe2TiO5, which had realized the reduction of iron oxidization in the ilmenite and the crystal grain growth of metal iron under low temperature.

5:15 PM

The Kinetics of Oxidation of Tellurium Sulfide Concentrate: *Edgar Blanco*[1]; [1]Western Utah Copper Company

A thermodynamic and kinetics investigation of the oxidation of tellurium in sulfide concentrate to TeO2 at high temperature was carried out as part of a new process to extract tellurium from its sulfide ore. The kinetics of the reaction was determined by measuring the weight change of a sample with time at temperatures between 700-1100ºC. The reaction rate followed the Shrinking-Unreacted-Core model under chemical reaction control. While most base metals remained with the calcine during the oxidation, almost all tellurium was volatilized in agreement with thermodynamic analysis.

Jim Evans Honorary Symposium: Metal Flow, Bubbles, and Inclusions Behavior in Refining and Reduction Vessels

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division
Program Organizers: Ben Li, University of Michigan; Brian G. Thomas, University of Illinois at Urbana-Champaign; Lifeng Zhang, Missouri University of Science and Technology; Fiona Doyle, University of California, Berkeley; Andrew Campbell, WorleyParsons

Tuesday PM Room: 620
February 16, 2010 Location: Washington State Convention Center

Session Chair: Stavros Argyropoulos, University of Torronto

2:30 PM Introductory Comments

2:40 PM
Plume Characteristics in Gas Stirred Ladles: *Piotr Scheller*[1]; Olena Volkova[1]; Dmitri Ryabov[1]; [1]Freiberg University

In our investigations trials in water models were performed as well as in the industrial process in the 30t ladles. In water models the phenomenology of plume formation and development was investigated and some characteristic parameter estimated. In the industrial trials the evolution of the geometry of the "eye" with gas flow rate was measured as a function of Ar flow rate. Using this basic information the flow velocity in the plume and it´s geometry as well as the bubble size were estimated for usual industrial process conditions. Using the dimension analysis the similarity criterion for the plume flow in the water model and in liquid steel is developed. The experimental results from water model are compared with the industrial one. The results obtained give a solid basis for the development of process models and establish the similarity criterion.

3:05 PM
Development of Online Sensors for Bubble Stirred Vessels: *Geoffrey Brooks*[1]; Xiaodong Xu[1]; William Yang[2]; [1]Swinburne University of Technology; [2]CSIRO Minerals

Bubbling is commonly used in pyrometallurgical vessels to enhance mass and heat transfer. These processes can be difficult to monitor because of the severe operating conditions associated with these processes. In this paper, we describe the development of techniques for simultaneously analysing image, sound and vibration signals associated with bubbling in vessels using multivariate statistical analysis to form new "latent" variables from the combined signals to control the process. This approach has been tested over a range of conditions for a bottom stirred cold model with two liquid layers (to simulate metal and slag) and the effect of combining different signals (e.g. sound and image, vibration and sound) has also been analysed. These results are examined in terms of what the signals tells us about bubbling but also with the view of developing practical online sensors for pyrometallurgical processes.

3:30 PM
Fluid Flow and Inclusion Behavior in a Continuous Billet Casting Tundish: *Qinglin He*[1]; Geoff Evans[1]; [1]University of Newcastle

The fluid flow and inclusion behaviour in a continuous billet casting tundish was studied by oil/water modeling. Oil content and droplet size distribution were measured at different locations in the tundish under various casting conditions, using high speed videoing technology in conjunction with a flow-through optical cell. Focus of this study was, in particular, placed on the behavior of macro size inclusions (>100µm), which are detrimental to the mechanical properties of steel. It was found that inclusion removal efficiency was affected by a number of factors, such as ladle stream condition, casting rate, initial inclusion concentration, tundish depth, ladle change, flow control device and shroud type. Tundish depth had the most significant effect on inclusion removal in the tundish. However, there is a critical tundish depth, beyond which it had no effect on inclusion removal.

3:55 PM
Physical Modeling of Slab Caster Tundish to Improve Yield and Quality of Steel: *Dipak Mazumdar*[1]; [1]Indian Institute of Technology-Kanpur

To minimize the volume of tundish skull (residual liquid left behind at the end of a sequence casting) in two different slab casting tundishes, an extensive water modeling investigation has been carried out. On the basis of such , appropriate modifications in the existing tundish designs (i.e. in terms of tundish floor, dam and pouring box) required to improve skull losses were derived and implemented in the shop floor. Full scale industrial trials show almost 60 to 70% reductions in tundish skull at the end of a sequence casting. Parallel to such, Residence Time Distribution measurements and microscopic examination of steel samples (collected from shroud, mono block regions and solidified slab) confirmed that modified and original tundish designs have similar inclusion floatation efficiency. Modified tundish designs have since been implemented in the two steel melt shops and practiced regularly in sequence casting.

4:20 PM Break

4:35 PM
Time Dependent MHD Models for Aluminium Reduction Cells: *Valdis Bojarevics*[1]; Koulis Pericleous[1]; [1]University of Greenwich

The time dependent MHD or stability problems for the aluminium reduction cells are typically restricted to the mathematical developments without the inclusion of the electrolyte channels. However, according to the well known Evans-Moreau model, the presence of electrolyte channels in the stationary case increases very significantly the interface deformation. The theory and numerical model of the reduction cell can be extended to the cases of variable bottom of aluminium pad and the variable thickness of the electrolyte with the presence of the channels. We present instructive analysis of different physical coupling factors affecting the magnetic field, electric current, velocity and wave development with animated examples for the high amperage cells.The results indicate that the 'rotating wave' instability is dominant in the absence of the channels, while the channels exert a stabilizing effect with a 'sloshing' parametrically excited MHD wave development in the aluminium production cells.

5:00 PM
Modeling on Multiphase Fluid Flow in Aluminum Electrolysis Cell: Yufeng Wang[1]; *Lifeng Zhang*[1]; [1]Missouri University of Science and Technology

Water modeling and mathematical modeling were established to study the fluid flow related phenomena in an aluminum electrolysis cell. The VOF multiphase fluid flow modeling was developed and validated by the water modeling - Laser Doppler Velocimeter (LDV) measurement in a full scale water model. Effects of the slot design and the tilted angle of the anode on the behavior of bubbles under the anode were studied. The slot under anode facilitates the escape of bubbles. With flat bottom anode, the slot is the main releasing path for bubbles. Without slot, a thicker gas film forms under the anode and this cause great energy consumption and induce violent fluctuation in the slot. By increasing the tilted angle of the anode, bubbles have multi-paths to escape, from the slot or moving along the tilted anode bottom and escaping from the curved end of the anode.

5:25 PM
Research and Development of Three-Dimensional Electrochemical Reactors at UC Berkeley and ICPF Prague – Overview: *Vladimir Jiricny*[1]; James Evans[2]; [1]Institute of Chemical Process Fundamentals, ASCR,v.v.i.; [2]University of California

The contribution is focused on the research and development of three-dimensional (fluidized and spouted bed) electrochemical reactors conducted at Berkeley and ICPF Prague. The overview of principles, experimental results on electrowinning of metals and organic synthesis and the reactor design will be presented. Some excursion to applications of these results will be shown.

5:50 PM
Importance of Microexothermicity in the Assimilation and Recovery of Additions in Liquid Metals: Zhi Li[1]; *Stavros Argyropoulos*[1]; [1]University of Toronto

Whenever exothermicity takes places during the course of assimilation of additions in liquid metals, two types have been identified, namely, Macroexothermicity and Microexothermicity. The former type of exothermicity is exhibited when a relatively small amount of addition is immersed into a large quantity of liquid metal; the latter type, on the other hand, is displayed when a powder mixture compacts consisted of different elements reacts exothermically to form intermetallics. The impact of Microexothermicity in the assimilation and recovery of compacted powder additions in liquid metals will be examined. Experimental work involving cylindrical compacted powder additions in liquid

Aluminum and liquid Magnesium will be presented. The additions used in this experimental work include Mn-Al,Zr-Zn and Zr-Al cylindrical powder compacts. The Microexothermicity, assimilation, and recovery data of these additions in liquid metals such as Aluminum and Magnesium will be compared. In addition, various ramifications of these comparisons in different liquid metal operations will be presented.

Magnesium Technology 2010: Fatigue, Failure, and Wear

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Sean Agnew, University of Virginia; Eric Nyberg, Pacific Northwest National Laboratory; Wim Sillekens, TNO; Neale Neelameggham, US Magnesium LLC

Tuesday PM Room: 613
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Michele Manuel, University of Florida; Anumalasetty Nagasekhar, The University of Queensland

2:00 PM
Effect of Shot Peening on High Cycle Fatigue Performance of Mg–10Gd–3Y–0.5Zr Magnesium Alloy: Wencai Liu[1]; *Jie Dong*[1]; Ping Zhang[2]; Li Jin[1]; Wenjiang Ding[1]; [1]Shanghai Jiao Tong University; [2]BTU-Cottbus

The effect of shot peening on high cycle fatigue performance of four states Mg–10Gd–3Y–0.5Zr magnesium alloy which named as as-cast, cast-T6, as-extruded and extruded-T5 GW103 were investigated. The glass bead with an average diameter of 0.35mm was adopted for shot peening and Almen intensity was arranged from 0.05 to 0.60mmN. The surface characteristics as roughness, microhardness and residual stresses produced by shot peening were measured. The microstructures of deformation layer of the peened specimens were observed by TEM and fracture surfaces were analyzed using SEM. The results show that the optimum Almen intensities of the four alloys are 0.40, 0.50, 0.10 and 0.10 mmN, respectively. The fatigue strengths of the four states peened specimens with the optimum Almen intensities were 30, 20, 65 and 75 MPa stronger than those of the reference unpeened specimens, the improvement of about 35%, 19%, 43% and 45% have been achieved, respectively.

2:20 PM
Monotonic and Multiaxial Cyclic Behavior of the Extruded AZ31B Magnesium Alloy: *Jafar Albinmousa*[1]; Hamid Jahed[1]; Steve Lambert[1]; [1]University of Waterloo

Recently, focus has been directed towards using magnesium alloys as a material for automobile vehicle structural components. In automotive applications, load bearing components are subjected to multiaxial fatigue loading. Thus, a better understanding of multiaxial fatigue behavior is a necessary step in the fatigue design of these components.This paper focuses on the monotonic and uniaxial and multiaxial cyclic behavior of extruded AZ31B magnesium alloy. Flat or tubular specimens were machined from large AZ31B extrusion sections. Two loading modes were considered for multiaxial tests: tensile and shear. All tests were performed at standard laboratory and in as-received conditions. Cyclic axial test results indicate that AZ31B exhibit asymmetrical cyclic behavior due to twinning. In contrast, cyclic torsional behavior was found to be symmetric. Energy, as a fatigue parameter, has been used to correlate the test results.

2:40 PM
Fatigue Evaluation of Friction Stir Spot Welds in Magnesium Sheets: *J. Jordon*[1]; M. Horstemeyer[1]; Jenna Grantham[1]; [1]Mississippi State University

In this study, the fatigue crack growth rate of magnesium AZ31 in friction stir spot welded is experimentally determined. Interrupted load control cyclic tests were conducted on single weld lap-shear coupons to determine fatigue crack growth rates. The coupons were stopped at regular intervals and the crack length was measured via an optical microscope. In addition, fractured coupons were examined under a scanning electron microscope with the intent to correlate striation spacing to the interrupted crack growth tests. The interrupted tests also revealed the percentage of initiation life associated with the friction stir spot weld joint. The crack growth rates conducted in this study were used to validate a long crack growth approach employed to predict the fatigue life of the friction stir spot weld coupons.

3:00 PM
Atomistic Simulations of Fatigue Crack Growth and the Influence of Temperature on Fatigue Behavior in Magnesium Crystals: *Tian Tang*[1]; Sungho Kim[1]; Mark F. Horstemeyer[1]; [1]Mississippi State University

The fatigue behavior of magnesium single crystals at various temperature and the fatigue crack growth in magnesium bicrystals has been computational simulated at nanoscale. The computational method used in this study is Embedded Atom Method (EAM), potentials. Five crystal orientation of initial crack, orientation C-(10¯10)[0001], orientation D-(1¯210)[0001]orientationF-(0001)[1¯210], orientation G (10¯11)[¯1012], and orientation I (10¯12)[10¯11] were analyzed for verifying the influence of temperature on fatigue behavior. The critical values of maximum strains at which the cracks start growing for different orientation increase with increasing temperature. The maximum strain \949max of cyclic loading was chosen as 0.8 of the critical value determined in uniaxial tension test. The fatigue crack growth rate of different orientation decrease with increasing temperature. In order to verify the effects of grain boundary, the mechanism of fatigue propagation was also investigated in several bicrystal model.

3:20 PM
Structure-Property Evaluation of Fatigue Damage in a Magnesium AM30 Alloy: *J. Bernard*[1]; J. Jordon[1]; M. Horstemeyer[1]; H. El Kadiri[1]; [1]Center for Advanced Vehicular Systems, Mississippi State University

The purpose of this study is to experimentally evaluate the fatigue characteristics of an extruded AM30 magnesium alloy in regards to structure-property relations. Fully-reversed, strain control fatigue tests were conducted in the extruded direction. Specimens were machined from locations with varying grain size and texture gradients. Using scanning electron microscopy, the fracture surfaces of the fatigued specimens were analyzed in order to develop relationships between the microstructure and the fatigue behavior of this material. Parallels between microstructural features such as particle size, grain size, Taylor factor, nearest neighbor distance and the number of cycles to failure were determined. These results were then used to develop a microstructure-based multistage fatigue model (MSF) employed to predict the variability of the fatigue results.

3:40 PM Break

4:00 PM
Very High Cycle Fatigue Property of Magnesium Alloy in Axial Loading and Rotating Bending: *Tatsuo Sakai*[1]; Yosuke Nakamori[1]; Noriyuki Ninomiya[1]; Mitsuji Ueda[2]; [1]Ritsumeikan University; [2]KS TECHNOS

Lightness and low value of Young's modulus are typical characteristics for Magnesium alloy when they are applied to the mechanical components in the engineering application. Fatigue property of this alloy is also important factor in order to ensure the reliability of the mechanical structures. Especially, the fatigue property in the long life region should be clarified for the fundamental design data to provide the safety of the mechanical structures in the long term use. From this point of view, fatigue tests were performed for the magnesium alloy(AMCa602) in axial loading and rotating bending. Such fatigue tests were carried out up to gigacycle regime to examine the very high cycle fatigue characteristics. S-N properties obtained in both loading types are compared to each other. Fracture surfaces of failed specimens were observed by means of SEM and fracture mechanisms of this alloy was discussed from view points of fractography and fracture mechanics.

4:20 PM
Numerical Modeling of Failure in Magnesium Alloys during Crush Simulations: Jonathan Rossiter[1]; *Kaan Inal*[1]; Raja Mishra[2]; [1]University of Waterloo; [2]General Motors R&D Center

Numerical modeling of failure of magnesium tubes during crush simulations is performed for commercial AZ31 magnesium alloy. LS-DYNA material model MAT_124 along with experimental stress-strain data from tensile and compression tests of the extruded tube have been used to capture the asymmetry in the tension and compression response over a range of strain and strain rates. A combination of three different failure criteria, consistent with observations of fractured components, has been implemented in the finite element simulations to accurately reproduce the experimental observations during axial

compression tests of round tubes. The presentation will include simulations of failure of magnesium tubes with both round and rectangular cross-sections and with different initial texture and tension/compression asymmetry. The predicted numerical responses of these tubes will be compared to experimental observations.

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Dry Sliding Wear Behavior of AE44 Magnesium Alloy Reinforced with Saffil Alumina Fibers: Bin Hu[1]; Liming Peng[1]; Bob Powell[2]; Michael Lukitsch[2]; Anil Sachdev[2]; *Xiaoqin Zeng*[1]; [1]Shanghai Jiao Tong University; [2]General Motors Corporation

The effect of 15 volume percent short alumina fibers for improving the wear resistance of the creep-resistant magnesium alloy AE44 was determined by pin-on-disk test under dry sliding wear conditions. The alumina fiber-reinforced AE44 composites were prepared by squeeze casting. Their wear behavior was determined for variations in sliding speed, applied load, and test temperature. Under all conditions the fibers improved wear resistance although the degree was influenced by fiber orientation and the nature of the binder (alumina or silica) used in the alumina fiber preforms.

5:00 PM

Influence of Cerium on Stress Corrosion Cracking in AZ91D: *Meredith Heilig*[1]; Daniela Zander[2]; David Olson[1]; Brajendra Mishra[1]; Norbert Hort[3]; Gerald Klaus[4]; Andreas Buehrig-Polaczek[4]; Joachim Gröbner[5]; Rainer Schmid-Fetzer[5]; [1]Colorado School of Mines; [2]TU Dortmund; [3]GKSS Research Centre; [4]Foundry-Institute of RWTH Aachen; [5]TU Clausthal

Applications of Mg-Al alloys are continually increasing, prompting the need to understand the corrosion behavior of these materials in loading conditions. This paper considers the effect of cerium additions on the stress corrosion cracking in the Mg-Al-Zn alloy AZ91D. The two dominant phases in the AZ91D microstructure are intermetallic Mg17Al12 precipitates interspersed within the Mg-rich matrix. The addition of cerium is thought to improve the corrosion performance of AZ91 alloys through microstructural modifications. Formation of Ce-Al intermetallics appear to minimize the volume fraction of Mg17Al12, thereby increasing the aluminum content of the Mg-rich matrix. Electrochemical impedance spectroscopy is used to characterize and compare the oxide films formed in AZ91D with varying amounts of cerium added. Immersion, potentiostatic polarization, and slow strain rate testing (SSRT) of AZ91D and AZ91D+Ce alloys were used to explore the influence of cerium on stress corrosion cracking behavior and passive film properties of AZ91D.

5:20 PM

In-situ Fracture Investigations of YAl_2 Reinforced Magnesium Matrix Composite: *Zhaohua Ling*[1]; Guoqing Wu[1]; Jianku Shang[2]; Sujie Wang[1]; Zheng Huang[1]; [1]Beihang University; [2]University of Illinois, Urbana-Champaign

A novel kind of magnesium matrix composites reinforced with YAl_2 intermetallic particles was prepared by stir-casting. The microstructure of the composite was observed by optical microscope and scanning electron microscope (SEM). The fracture process was in-situ investigated by the SEM during dynamic tensile test. The results show that the microcracks initiate both in the alloy matrix and interfaces, and then propagate continuously in the alloy matrix or along the interfaces. The YAl_2 particles can impede propagation of microcracks during the fracture process. The dominant crack mainly propagates along the boundary of the particle-dense area and particle-rare area and which finally leads to the failure of the composite. The matrix failure plays a significant role in the failure mechanisms of this kind of composite materials.

Magnesium Technology 2010: Magnesium - Rare Earth Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Sean Agnew, University of Virginia; Eric Nyberg, Pacific Northwest National Laboratory; Wim Sillekens, TNO; Neale Neelameggham, US Magnesium LLC

Tuesday PM Room: 612
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Sean Agnew, University of Virginia; Jian-Feng Nie, Monash University

2:00 PM

Development of High Ductility Magnesium-Zinc-Cerium Extrusion Alloys: *Alan Luo*[1]; Raj Mishra[1]; Anil Sachdev[1]; [1]General Motors Corporation

It has been shown in a previous study that the ductility of Mg-0.2%Ce alloy extrusion is higher than that of magnesium and other known magnesium alloys. However, the yield and tensile strengths of the Mg-0.2%Ce alloy remain low. In this paper, it is found that a small addition of zinc (around 2%) can significantly improve the strength of the Mg-0.2%Ce alloy (96% in yield strength and 32% in ultimate tensile strength) with only a slight reduction in elongation (27.4% vs. 31%). The microstructure of Mg-Zn-Ce alloys at various extrusion speeds is characterized using optical metallography, electron probe micro-analysis (EPMA), and electron backscattered diffraction (EBSD) techniques. It is concluded that while Zn provides strength through solid solution strengthening, Ce increases the ductility of the Mg-Zn-Ce alloys via improved texture.

2:20 PM

Effect of Extrusion Temperature on Microstructure and Mechanical Properties of Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr Alloy Solid State Recycled by Hot Extrusion: *Qudong Wang*[1]; Jie Chen[1]; Tao Peng[1]; Zheng Zhao[1]; Wenjiang Ding[1]; [1]National Engineering Research Center of Light Alloy Net Forming, Shanghai Jiao Tong University

The effect of extrusion temperature on microstructure and mechanical properties of Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr (wt.%) alloy solid state recycled by hot extrusion was investigated. The recycled specimens have very fine microstructure due to dynamic recrystallization during extrusion. Higher extrusion temperature tends to coarsen grains, especially when extruded at 773K. The age hardening response as well as UTS of recycled samples are remarkably improved with increased extrusion temperature. Better metallurgical bonding and reduced extrusion precipitation with increased extrusion temperature are responsible for the improvements. However, elongation decreases rapidly to 1.3% for the sample recycled at 773K probably because of coarsened grains and excessive cavitations. The sample recycled at 723K shows the best combination of strength and elongation after ageing treatment, whose TYS,UTS and elongation reach 346MPa,390MPa and 4.2%. Therefore, it is necessary to extrude at lower temperature while ensure fully metallurgical bonding of chips to obtain excellent strength and elongation of recycled samples.

2:40 PM

Structure of β_1 precipitates in Mg-Zn based alloys: Co-Existence of $MgZn_2$ and Mg_4Zn_7 Phases: *Alok Singh*[1]; Julian Rosalie[1]; Hidetoshi Somekawa[1]; Toshiji Mukai[1]; [1]National Institute for Materials Science

Although the the rod-like β_1 precipitate is among the most important in magnesium alloys, its crystal structure is not yet clear. Its structure has been believed to be based on the Lave's phase $MgZn_2$, but recently reported to be of monoclinic Mg_4Zn_7 phase. We have studied the structure of these precipitates by high resolution TEM in a Mg-Zn-Y alloy. It was found that domains of Mg_4Zn_7 phase coexist with Laves phase $MgZn_2$ within the precipitates. Structural units of the Mg_4Zn_7 phase characterized by icosahedral coordination rearrange to form the $MgZn_2$ structure with the orientation relationship with the matrix reported earlier. The crystallographic relationship between the phases is described. Axially, these phases are related to each other and the matrix as $[1\text{-}210]_{MgZn2} \| [010]_{Mg4Zn7} \| [0001]_{Mg}$. The {10-10} planes of $MgZn_2$ correspond to {003} and ½{023} planes of the Mg_4Zn_7 phase.

3:00 PM

Rheological Behavior of Semi-Solid Mg-Y Alloys: *Qiuming Peng*[1]; Yuanding Huang[1]; Norbert Hort[1]; Karl Ulrich Kainer[1]; [1]MagIC – Magnesium Innovation Centre

The knowledge of the rheological behaviour of Mg-RE suspensions is an important issue in semi-solid processes of Mg alloys congaing RE. Therefore, rheological experiments like step-change in shear rate tests, shear stress ramps and oscillation experiments have been carried out to characterise the flow behaviour of Mg-RE alloys exemplarily Mg-Y and Mg-Nd alloys. The microstructures, solid fraction and viscosity of the alloys at different solidified temperatures are investigated. The materials exhibit a yield stress and shows shear-thinning and thixotropic flow behaviour, which are strongly influenced by the solid traction, the particle shape and size and the degree of particle agglomeration. All experiments were carried out in a Couette rheometer. The experimentally gained data are similar to a model approach, which consists of a modified Herschel-Bulkley law. These databases provide an implication for the development of Mg-RE alloys.

3:20 PM

The Use of Computer Modeling for Producing DC Cast WE43 Magnesium Alloy Slab: *Mark Turski*[1]; John Grandfield[2]; Tim Wilks[1]; Bruce Davis[3]; Rick DeLorme[3]; [1]Magnesium Elektron; [2]Grandfield Technology Pty Ltd; [3]Magnesium Elektron North America

During direct chill casting, significant stresses can develop within the material leading to cracking within the cast slab. The situation is made worse for higher strength magnesium alloys, such as WE43, which also exhibits high strength at elevated temperatures. Consequently, the temperature and stress field must be well understood during the casting process.ALSIM is a fully coupled thermo-mechanical finite element modeling code capable of simulating the DC casting process. ALSIM was used to simulate vertical DC casting of 870 x 315 mm WE43 slab. Validation of the model was carried out by the use of thermocouples and post mortem sectioning of cast slabs containing cracks. Good agreement has been found between modeling predictions and thermocouple measurements and post-mortem examination of cast material. The use of this validated model has allowed optimized casting parameters to be developed to produce crack free WE43 slab.

3:40 PM Break

4:00 PM

Effects of Extrusion Conditions on the Microstructure and Properties of Mg-Zn-Y-RE Alloys: *Jonghyun Kim*[1]; Yoshihito Kawamura[2]; [1]Kumamoto Technology & Industrial Foundation; [2]Kumamoto University

Magnesium alloys are known for their light weight and specific stiffness which are greatly attractive to the automotive and aerospace industries. They are usually supplied as the flat sheets or profiled products processed by metal working. Extrusion process is a prevailing shape forming procedure of magnesium products. In the present study, the authors conducted hot extrusion of Mg-Zn-Y-RE alloys billets and investigated the effects of extrusion conditions on the microstructure and mechanical properties of the extruded products.The extrusion in the Mg-Zn-Y-RE alloys led to grain refinement through dynamic recrystallization. Extrusion at the lower ram speed of 3.7 mm*s-1 resulted in a larger average grain size and higher elongation than at 2.5 mm*s-1. The yield strength of the Mg-Zn-Y-RE alloys after extrusion at 3.7 mm*s-1 was lower than that of the alloy extruded at 2.5 mm*s-1.

4:20 PM

Effect of Cerium on the Deformation Behavior of Two Mg-Ce Alloys: *Lan Jiang*[1]; Xavier Quelennec[1]; John Jonas[1]; Raja Mishra[2]; [1]McGill University; [2]GM

The effect of cerium on the formability of Mg alloy tubes was investigated by means of uniaxial tension and uniaxial compression testing. The tests were carried out at ambient temperature and 200°C at strain rates of 0.1/s and 0.001/s. Samples were cut from extruded tubes containing two different levels of Ce addition. Specimens deformed to increasing strain levels were examined by optical microscopy and electron backscattered diffraction (EBSD) techniques. At both test temperatures, higher tensile ductilities were observed in the Mg-0.5%Ce material than in the Mg-0.2%Ce, especially at the higher strain rate. However, at 200ºC/0.001s-1, the Mg-0.2%Ce displayed better formability than the Mg-0.5%Ce. The differences can be attributed to the effects of initial texture, particle concentration and distribution, and grain size.

4:40 PM

Structural Relationships between Monoclinic and Laves Phase Precipitates in Mg-Zn-Y Alloys: *Julian Rosalie*[1]; Hidetoshi Somekawa[1]; Alok Singh[1]; Toshiji Mukai[1]; [1]National Institute for Materials Science

The response of textured Mg-Zn and Mg-Zn-Y alloys to controlled pre-ageing deformation has been examined in detail. Samples subjected to c-axis tension developed both deformation twins and some dislocations, while c-axis compression resulted in more extensive dislocation multiplication. Rod-like β_1' phase (Mg_4Zn_7) formed on dislocations while irregular precipitates of similar composition (and of the $MgZn_2$ Laves phase) formed on twin boundaries. The ageing response was accelerated by precipitation on both types of defect, but was more sensitive to the number of dislocations present. Higher dislocation densities also resulted in a more refined distribution of β_1' precipitates, which was not observed when the predominant defects were twins. The results highlight the sensitivity of Mg-Zn alloys to the nature of pre-ageing deformation and the importance of texture control in developing heat -treatments for wrought Mg alloys.

5:00 PM

Exploiting Low Levels of Rare Earth Addition in Mg Extrusion Alloys: *Matthew Barnett*[1]; A.G. Beer[1]; N. Stanford[1]; [1]Deakin University

The benefit of adding rare-earth alloying elements to wrought magnesium alloys is well known.How these elements act to alter the mechanical performance is less understood.The present article presents some experimental results that shed light on this effect.It is also shown that low levels of their addition can lead to notable improvements in performance without the deterioration in extrudability seen with other alloying elements.It is demonstrated that the texture changes rapidly with small levels of addition.However, there is a limited operating window within which favourable textures arise.It is argued-in agreement with previous suggestions-that the key to understanding the role of rare earth elements at low levels is twofold.One effect relates to the influence exerted on the homogeneity of the deformation, which is also manifest in dynamic strain aging phenomena.The other relates to the retardation of recrystallization, which allows a characteristic deformation structure to develop during hot working.

5:20 PM

Thermodynamic Database Development of Mg Alloys with RE Elements and Its Applications to Mg Alloy Design: *Youn-Bae Kang*[1]; Liling Jin[1]; In-Ho Jung[2]; Arthur D. Pelton[1]; Patrice Chartrand[1]; Carlton D. Fuerst[3]; [1]Ecole Polytechnique de Montreal; [2]McGill University; [3]General Motors

Rare earth (RE) elements are often added to Mg-based alloys in order to improve creep resistance and strength at elevated temperature by forming stable precipitates with Al (AlxREy) or Zn (ZnxREy). Thermodynamic models and databases can provide invaluable information to elucidate the complex precipitation behaviors involving RE. In the present study, recent progresses of Mg alloy database development with FactSage toward adding RE elements, including the systems of Mg-RE and Al-RE (RE = La, Ce, Pr, Nd, Y, Sc, …), and a number of Mg-Al-RE and Mg-Zn-RE (RE = La, Ce, Y, Sc), will be presented. The applications of the newly developed database to the Mg alloy design will be also presented.

Materials in Clean Power Systems V: Clean Coal-, Hydrogen Based-Technologies, Fuel Cells, and Materials for Energy Storage: SOFC I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Conversion and Storage Committee
Program Organizers: Xingbo Liu, West Virginia University; Zhenguo Yang, Pacific Northwest National Lab; K. Weil, Pacific Northwest National Lab; Mike Brady, Oak Ridge National Lab; Jay Whitacre, Carnegie Mellon University; Ayyakkannu Manivannan, National Energy Technology Laboratory; Zi-Kui Liu, Penn State University

Tuesday PM Room: 211
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Teruhisa Horita, AIST; Harry Finklea, West Virginia University

2:00 PM Invited
Degradation of SOFC Anodes and SOFC Performance in Coal Syngas Containing Phosphine: *Harry Finklea*[1]; Oktay Demircan[1]; John Zondlo[1]; Chunchuan Xu[1]; [1]West Virginia University

Coal syngas, a potential fuel for solid oxide fuel cells in large scale power generation, may contain ppm levels of phosphine as well as other impurities. In this presentation, we report studies of anode-supported solid oxide fuel cells (Ni/YSZ anode) exposed to 10 ppm levels of phosphine in a synthetic syngas mixture of hydrogen, water, carbon monoxide and carbon dioxide. Cell power degrades immediately at high rates (on the order of mW/cm2/hr) upon introduction of 10 ppm into the syngas. Operation for over hundred hours results in substantial migration of nickel to the surface of the anode and into voids in the anode as nickel phosphide phases. The rate of degradation is significant over a range of operating voltages of the SOFC. The mechanism of phosphine-induced degradation will be discussed, along with suggestions on how to mitigate the degradation.

2:40 PM
Effects of Phosphine on Solid Oxide Fuel Cell Performance and Related Anode Surface Temperature Measurement: Huang Guo[1]; Gulfam Iqbal[1]; *Bruce Kang*[1]; [1]West Virginia University

The degradation effects of phosphine (PH3) contaminant on the solid oxide fuel cells (SOFCs) performance are investigated under hydrogen and simulated coal syngas environment with and without steam. The related anode surface temperature measurements, as a function of applied current densities, are also carried out via our in-situ experimental technique. The experimental results indicate that the Ni-cermet-based SOFC anode is more susceptible to degradation due to PH3 in wet hydrogen than in dry hydrogen condition. Combining the results from post experiment characterization, such as SEM, EDX, XPS, the poisoning effect and degradation mechanism of PH3 on the SOFCs performance are discussed.

3:00 PM
Fabrication and Characterization of Thin Film $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) and $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ - $Ce_{0.8}Gd_{0.2}O_{1.9}$ (CGO) Composite Cathodes: *Bainye Angoua*[1]; Elliot Slamovich[1]; [1]Purdue University

Due to their high electrocatalytic activity for oxygen reduction, LSCF cathodes improve the performance of solid oxide fuel cells at intermediate temperatures (500-700°C). In this study, thin (<500 nm) dense LSCF and LSCF-CGO cathodes are produced by spray pyrolysis. Solutions of nitrate salts dissolved in a mixture of ethanol, butyl carbitol and methyl carbitol were atomized onto dense 8YSZ substrates between 230-240°C, followed by subsequent crystallization in air at 700-900°C. The effect of sintering temperature on film morphology and electrical conductivity was investigated to determine an optimum firing temperature with respect to cathode electrical resistance and formation of reaction products between 8YSZ and LSCF. Microstructure evolution of the raw films as a function of firing temperature was followed via SEM, FIB sections and EDS. Electrical characterization was performed by impedance spectroscopy on cathodes deposited symmetrically across the 8YSZ substrate. The effect of a thin CGO buffer layer was also investigated.

3:20 PM
Sintering Performance of YSZ Ceramics with Transition Metal Oxide Sintering Aid: *Stephen Sofie*[1]; M.L. Lifson[1]; C. Law[1]; [1]Montana State University

The effect of NiO additions in YSZ was examined to provide a means to tailor the sintering behavior of both fully and partially stabilized YSZ. YSZ powders of micro size scale were mixed with 0.1, 0.5,and 1.0 mol% NiO.Pellets were characterized from 1200C to 1400C with a 1 hour dwell. Undoped micro 8YSZ and 3YSZ powders sintered at 1300C for one hour yielded 79.5% and 67.6% theoretical density, respectively. At the same temperature profile, the 8YSZ powder with 1.0mol% nickel oxide reached 95% theoretical density and the 3YSZ micro powder with 1.0mol% nickel achieved 96.7% theoretical density. Dilatometry revealed that the onset of sintering decreased and maximum rate of sintering increased with increasing nickel oxide concentration.

3:40 PM Break

3:50 PM
Modeling of Total and Topologically Connected Triple Phase Boundaries in Composite Cathodes for Solid Oxide Fuel Cells: *Arun Gokhale*[1]; Shenjia Zhang[1]; [1]Georgia Institute of Technology

Analytical and numerical modeling of three-phase microstructures of LSM/YSZ composite cathodes is carried out for prediction of the geometric characteristic of the YSZ-LSM-Pore triple phase boundaries (TPB). The analysis predicts total and topologically connected TPB length per unit volume (LTPB) from volume fractions and characteristics of initial powder populations of YSZ and LSM. The parametric analysis reveals that (1) non-equiaxed plate-like, flake-like, and needle-like YSZ/LSM particle shapes can yield substantially higher LTPB; (2) mono-sized YSZ/LSM powders lead to higher LTPB as compared to the powders having wide size distributions; (3) LTPB is inversely proportional to the mean sizes of YSZ and LSM particles; (4) high value of LTPB is obtained at the lowest porosity volume fraction that permits sufficient connectivity of the pores for gas permeability; and (5) LTPB is not sensitive to the relative proportion of YSZ and LSM phases in the regime of interest in composite cathode applications.

4:10 PM
Modeling Solid Oxide Fuel Cells with Mixed Conducting Electrolytes and Anode Functional Layers: *Keith Duncan*[1]; Eric Wachsman[1]; [1]University of Florida

Recent research at the University of Florida has shown that the inclusion of an anode functional layer (AFL) significantly increases the maximum power density of ceria electrolyte solid oxide fuel cells (SOFCs). Here, results from the latest development of AFL-equipped SOFCs are presented. Accordingly, a continuum-level analytical model was developed to accurately describe and predict the performance of SOFCs, in general, and to better explain the electrochemical impact of the AFLs on SOFC performance. The model was derived by coupling the Nernst-Planck and Butler-Volmer equations, while employing defect thermodynamics for boundary values. This approach allowed the usual assumptions of reversible electrodes and linear potential gradients to be removed; thereby allowing a non-ohmic response in the mixed conducting electrolyte. The model was validated through successful fits to experimental data from SOFCs with acceptor-doped ceria electrolytes. Finally, the model was used to predict open-circuit voltage dependence on electrolyte thickness.

4:30 PM
Modelling the Effect of Dopant Concentration on Lattice Strain and Ionic Conductivity in Fluorite Oxides: *Keith Duncan*[1]; Eric Wachsman[1]; [1]University of Florida

Scientists have searched intensely for oxides with high ionic conductivity for use in SOFCs. In oxides, ionic conduction is by point defect migration, so management of point defect population is a key strategy for maximizing conductivity. Research on doping strategies revealed that, for each host oxide, there exists optimal dopants and dopant concentrations, which are largely determined by minimizing the tradeoff between mobile defects, defect association and lattice strain. Though increasing dopant concentration increases the concentration of defects, it also increases defect association while decreasing mobility—both are deleterious to ionic conductivity. In the presentation, this phenomenon will be articulated in a continuum-level framework to formulate the relationship between dopant concentration, lattice strain, defect association and ionic conductivity. Excellent fits of the model were obtained for (CeO2)1-

x(SmO1.5)x at various temperatures with only two adjustable parameters. Further analysis of the results reproduces empirical trends and relationships previously reported in the literature.

4:50 PM

Simulation of Oxygen Ion Transport in Mixed-Conducting Solid Oxide Fuel Cell Cathode with Complex Microstructure: *Hsun-Yi Chen*[1]; Hui-Chia Yu[1]; Cortney Kreller[2]; James Wilson[3]; Scott Barnett[3]; Stuart Adler[2]; Katsuyo Thornton[1]; [1]University of Michigan; [2]University of Washington; [3]Northwestern University

$LaSrCoO_3$ (LSC) is a mixed ionic-electronic conductor and is considered a promising material for solid oxide fuel cell (SOFC) cathodes. Understanding oxygen transport mechanisms in LSC can provide insights that could lead to the rational design of the cathode microstructure. Electrochemical impedance spectroscopy (EIS) measurements combined with theoretical modeling and simulation can be employed to elucidate oxygen reduction mechanisms. We developed a numerical approach to simulate the electrochemical response of a mixed-conducting cathode with complex microstructures. The method was demonstrated for a cathode with simple structures. Simulations were also performed using a three-dimensional LSC microstructures constructed using images obtained by focused ion beam-scanning electron microscopy (FIB-SEM). The current-voltage relation and impedance are calculated for various rate limiting cases.

5:10 PM

Reliability Model for Different Configurations of Planar-SOFC Anode under Syngas Contaminants: Gulfam Iqbal[1]; Huang Guo[1]; *Bruce Kang*[1]; [1]West Virginia University

An anode material reliability model developed for solid oxide fuel cell (SOFC) button cell is adapted for the planar-SOFC configurations: co-flow, counter-flow and cross-flow. The model accounts for thermo-mechanical and fuel gas contaminants effects on the anode material to predict its structural life. The temperature field and contaminant concentration on planar-SOFC anode are interpreted from DREAM-SOFC, a multi-physics solver. Due to larger active areas, planar-SOFC configurations exhibit greater spatial and temporal temperature gradients compared to button cells, which lead to higher thermo-mechanical degradation. For the co-flow configuration, anode thermo-mechanical degradation is severe at the anode-electrolyte interface and it starts near the fuel outlet whereas the fuel gas contaminants effects on the anode microstructure begin at the fuel inlet and propagate through the anode thickness and along the fuel flow. The knowledge obtained from this research will be useful to establish control parameters to achieve desired service life of SOFC stacks.

Materials Processing Fundamentals: Smelting, Refining, Aqueous and Liquid Processing

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: 601
February 16, 2010 Location: Washington State Convention Center

Session Chair: Prince Anyalebechi, Grand Valley State University

2:30 PM

A Comparative Electrochemical Study of Arsenic Removal from Organic and Inorganic Sources Using Various Sacrificial Electrodes: *Jewel Gomes*[1]; Sanoar Rahman[1]; Srikanth Varma[1]; Kamol Das[1]; David Cocke[1]; [1]Lamar University

Arsenic is considered as one of the toxic materials being controlled by Environmental Protection Agencies in several developed and developing countries. In nature, it occurs in the soil, minerals and in various organic forms. It enters the air, water, and land from wind-blown dust and gets into water from runoff, leaching, soil erosion and anthropogenically from chemicals used for wood preservation, insecticides, medicine, military purpose, pigments, and electronic circuitries. There are several techniques available for removal of arsenic, such as coagulation/filtration, ion exchange, reverse osmosis, and electrochemical. Electrocoagulation has been found as one of the most efficient techniques to remove inorganic arsenic with >99% efficiency. Here we present our work on electrochemical treatment of both inorganic and organic arsenic from water using various sacrificial electrode materials, i.e., iron, aluminum, copper, titanium and combination of them. The floc produced has been characterized using XRD, SEM/EDS, FTIR, and cyclic voltammetry.

2:50 PM

Characterization of Sodium and Bath Penetration in Industrial Graphitic and Graphitized Cathodes: *Jilai Xue*[1]; Liancheng Wu[1]; Gangqiang Jiang[1]; Qingren Niu[1]; Qingsheng Liu[1]; Hou Xin[1]; Jun Zhu[1]; He Hua[1]; [1]Unversity of Science and Technology Beijing

Experiments with various industrial cathodes ranging from 35% to 100% graphitic and fully graphitized carbons were carried out in a laboratory aluminum electrolysis cell. Sodium and bath penetration into the cathode samples was measured by XRD and SEM-EDS analysis. For a higher cryolite ratio, the cathodes with lower graphitic carbons exhibited more sodium penetration than the fully graphitized. XRD characterization shows that the increased graphitization (p002) in the cathode carbons can reduce the sodium penetration. SEM-EDS inspection reveals that the sodium and fluorides penetration within the cathode samples has different patterns for varying graphitization of the carbon materials. The porosity of the cathode materials did not demonstrate the importance in correlation with the penetration process as expected before. The results can provide useful information for quality evaluation of the industrial cathode products as well as for improvement in cathode performance.

3:10 PM

Analysis and Identification Minerals Present in Rock Samples: *Andrew Appaji*[1]; [1]Noorul Islam University

Abstract Rocks and Minerals are inseparable. The area of our investigation in placed at the southern horn of India – Velimalai Hills, where still no great mineralogical surveys are done. This study is the first of its kind of analyzing the presence of different minerals in these rocks. The Rocks samples are collected and they are subjected to X ray diffraction study and Fourier Transform Infra Red Studies. The minerals are analyzed. Their presence is confirmed. The study was carried out for 3 samples; other samples are also to be studied.

3:30 PM

Preparation and Degradation Orgnic of TiO2 Coated on Light Ceramic Surface: *Ju Hua*[1]; [1]Harbin Institute of Technology

TiO2 photocatalyst could degrade the organic pollutants from the surface. This process has many advantages, including low energy consumption, simple techniques, mild reaction conditions, and less secondary pollution. However, the granular TiO2 is so tiny that it is difficult to precipitate, it loses its active constituents, and difficult to recycle. Thus, the granular state greatly limits its practical application. In order to resolve these problems, in the thesis we studied and produced the TiO2-coated light weight ceramic material. Because the TiO2-coated light weight ceramic's specific gravity is close to the water's, it can be floated on the water. Also it increases the interface with water and illuminating area, which enhances the capability of photocatalysis.

3:50 PM

Fe-Ni Alloys Formation in Carbonthermal Reduction Process using Laterite Nickel Ore: *Jilai Xue*[1]; Luxing Feng[1]; Jun Zhu[1]; [1]Unversity of Science and Technology Beijing

Fe-Ni alloys can be used as raw material in making of stainless steel and laterite nickel ore is one of resources. In this work, a mixture of laterite ore and coal was set in a furnace at 900°C and then reduced to form Fe-Ni alloys at 1550-1620°C through direct carbonthermal reduction process. The targeted contents of Ni in the alloys were investigated against various parameters. The ratio of the laterite to coal was found as a major factor in forming the alloy with targeted nickel content. A higher temperature can make better separation between the metal and the slag, and is in favor of producing high content of nickel. The slag contained relative higher Mg and Si, and can make some processing difficulties.

4:10 PM Break

4:30 PM
Active Zinc Oxide Production from Waste Zinc Powder: *Cem Colakoglu*[1]; Onuralp Yucel[1]; [1]Istanbul Technical University

Active zinc oxide has a high surface area (approximately 35-60 m2/g) and also has 10 times less particle size than that of zinc oxide. In this study, various quality of active zinc oxides containing up to 98 wt.% ZnO were produced by hydrometallurgical process from waste zinc powder (blue zinc powder). In the first step of the experiments, waste zinc powder containing 95 wt.% Zn and 0.5 wt.% Fe was reacted with sulphuric acid solution then hydrogen peroxide was added to remove Fe from the system. In the second step, obtained zinc sulphate solution was reacted with the addition of sodium carbonate solution. Resulting zinc carbonate was first be dried at 100°C then calcined at different temperatures and time to set the alternative quality of active zinc oxide. Elemental analysis of the waste zinc powder and active zinc oxide was performed by using XRD, XRF, BET and AAS.

4:50 PM
Preparation of MgO Whisker from Magnesite Tailings: *Li Yue-yuan*[1]; Cui Hong-Xu[1]; Chen Min[1]; [1]Northeastern University

China has abundant magnesite resource, but the low-grade and powder ores have always been rejected, which influences the comprehensive utilization of resource and often causes circumstance problems. The present work investigated the preparation of magnesia whisker using magnesite tailings as starting materials by preparation magnesium carbonate whisker as precursor firstly in the route of calcination, hydration, carbonation and pyrolysis, follows by second calcination of the precursor. The influence of pyrolysis conditions and additives on morphology of magnesium carbonate crystal as well as the influence of calcination conditions on morphology of magnesia whisker was investigated. The results showed that thermal decomposition products was MgCO3•3H2O. Magnesium carbonate whisker with length of 10~60 μm and the length-to-diameter ratio of 10~20 was prepared when soluble magnesium salt was added. While temperature was elevated at 1°C•min-1, magnesia whisker with length of 10~60μm was obtained.

5:10 PM
Remediation of Chicken Processing Wastewater Using Electrochemically Produced Layered Double Hydroxides: *Jewel Gomes*[1]; Daniel Atambo[1]; Manish Rahate[1]; Kamol Das[1]; George Irwin[1]; Hector Moreno[2]; David Cocke[1]; [1]Lamar University; [2]Instituto Tecnologico de la Laguna

Green rust (GR) are Layered Fe(II)-Fe(III) Double Hydroxides (LDH), having a pyroaurite-type structure consisting of positively charged hydroxide layers and hydrtated anions in interlayers. This special feature of GR is very efficient for wastewater treatment as the pollutants are easily taken in or exchanged with the anions in the interlayers. It can be very simply and cost-effectively produced by electrocoagulation (EC). Chicken processing plant (CPP) produces large amount of wastewater containing variety of readily biodegradable organic compounds, fats and proteins. The possible re-use of properly treated CPP wastewater would be economic and environment friendly. In this study, we present our work on treatment of CPP wastewater using EC. Analysis of the EC-treated water for reuse in the same plant is discussed considering the U.S. EPA regulations. Two types of EC-reactors were used for this purpose. To better understand the treatment mechanism, EC-floc was also characterized using XRD, SEM-EDS, and FTIR.

5:30 PM
Carbon-Thermal Reduction Process for Making Al-Si Alloys from Al2O3-SiO2 Containing Industrial Wastes: *Jilai Xue*[1]; Yunxia Song[1]; Jun Zhu[1]; [1]University of Science and Technology Beijing

Industrial solid wastes containing 40%-50% Al2O3 and 30-50% SiO2 have potential to be an alternative resource for Al-Si alloys production. Spent potlining materials from aluminum reduction cells and coal flyash from electrical power plants were tested in laboratory to make Al-Si alloys using carbonthermal reduction process. A mixture of anthracite and petroleum coke was used as reducing agency. One of the most important operations was to prepare the briquettes made of different raw materials with varying contents of Al2O3 and SiO2. The effects of briquette porosity, Al/Si ratio, the amount of carbon reducing agency, on the reduction process and the resulting alloy quality were investigated. The results are useful for further development of an alternative technology for Al-Si alloy production, and for waste treatment in recycling metals from the industrial solid wastes containing Al2O3 and SiO2.

Mechanical Performance for Current and Next-Generation Nuclear Reactors: Microstructure and Nanostructure in Reactor Environments

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Nuclear Materials Committee
Program Organizers: Dylan Morris, NIST; Greg Oberson, Nuclear Regulatory Commission; Nicholas Barbosa, National Institute of Standards & Tech; Wolfgang Hoffelner, Paul Scherrer Institute

Tuesday PM Room: 201
February 16, 2010 Location: Washington State Convention Center

Session Chair: Wolfgang Hoffelner, Paul Scherrer Institute

2:00 PM Invited
Irradiation Effects in Thin Metal Films – Texture Control and Mechanical Properties: *Ralph Spolenak*[1]; [1]ETH Zurich

Thin metal films can serve as model systems for radiation effects in nuclear materials or are under study for application in fusion reactors. The current study focuses on the effects of the interaction between radiation-induced defects in thin metal films and their grain-boundaries. Frenkel pairs as well as stacking fault tetrahedra are induced by high energy ions (argon or self ions). Energy minimization leads to a selective grain-growth of the undamaged grains and converts a fiber textured thin films (driven by surface energy minimization) into a "single–crystal". The interaction of ions with grain boundaries renders the process nearly athermal. In spite of the growth of "undamaged" grains, ion-induced hardening is observed by local mechanical probes. In addition to observations by TEM and EBSD the interaction of defects and grain-boundaries is observed in-situ at the JANNUS facility at Orsay, Paris.

2:30 PM
Elevated-Temperature Compression Testing and Characterization of Deformation and Fracture in Sintered ZrN Pellets as Surrogates for PuN Fuels: *Kirk Wheeler*[1]; Pedro Peralta[1]; Kenneth McClellan[2]; [1]Arizona State University; [2]Los Alamos National Laboratory

ZrN was studied as a possible surrogate for PuN under the Global Nuclear Energy Partnership (GNEP) program. In particular, this work addresses the mechanical properties and fracture behavior of sintered ZrN pellets at elevated temperatures and relates this behavior to its initial microstructure. Uniaxial compression testing was performed in an ultra-high purity Argon atmosphere at both intermediate (800°C) and high (1200°C) temperatures. Post-mortem fractography was performed via scanning electron microscopy (SEM) on pyramidally shaped fracture pieces produced as a result of localized shear bands that developed during high temperature tests and lattice rotations typical of plastic deformation were discovered at the tips of cracks that were arrested at grain boundaries using Electron Backscatter Diffraction (EBSD). Applicability of the results to the understanding of the structural reliability of nitride fuel pellets at working as well as thermal overload temperatures is discussed. Work supported under DOE/NE Agreement # DE-FC07-05ID14654.

2:50 PM
High Temperature Oxidation Behavior of Grain-Refined Inconel 617 for VHTRs: Tae Sun Jo[1]; Jeong Hun Lim[1]; Dae-gun Kim[1]; *Young Do Kim*[1]; [1]Hanyang University

In this study, the effect of grain size on high temperature oxidation of Inconel 617 alloy was investigated by exposure at 950°C for 1000 h in air atmosphere. The grain-refined Inconel 617 was obtained by recrystallization after cold rolling of 50%. The grain size was refined from 71μm to 5.2μm. The as-received and grain-refined alloys were oxidized with forming external oxide layer as Cr_2O_3 and with forming internal oxide as $Al_2O_3$2O3 along the grain boundary. The formation of Cr_2O_3 caused the Cr-depleted zone below the external oxide scale. The Cr-depleted zone of grain-refined alloy was thicker than that of the as-received one, while the depth of internal oxide of grain-

refined one was shallower. By prolonging the exposure time, the area of internal oxide of grain-refined alloy was gradually decreased. However, as-received one was constantly increased. The internal oxide at the grain-refined alloy had a more homogeneous microstructure.

3:10 PM

Hot Steam Corrosion Behavior of Ni-Based Superalloys at High Temperature Steam Environments: *Donghoon Kim*[1]; Minu Kim[1]; Hyun Min Lee[1]; Daejong Kim[1]; Changheui Jang[1]; Duk-Joo Yoon[2]; [1]KAIST; [2]KEPRI

A high temperature steam electrolysis (HTSE) is one of the promising ways of the massive hydrogen production using the very high temperature gas cooled reactor (VHTR). During the operation of the HTSE-VHTR system, structural material such as intermediate heat exchanger (IHX) are exposed to high temperature steam condition. Thus the corrosion resistance of the heat exchanger materials is critical to the safety and performance of the system. In this study, the corrosion behaviors of several Ni-based superalloys were investigated in steam environments at 900° with and without hydrogen gas. Corrosion kinetics was studied by weight change per unit surface area and oxide layer and microstructure change were analyzed using a scanning electron microscope (SEM), energy-dispersive X-ray spectroscopy (EDX), and X-ray diffractometer (XRD). Also, the effects of alloying elements on steam corrosion resistance were investigated. Finally, the results were compared with those at high temperature air and helium conditions.

3:30 PM

The Cause of Dynamic Strain Aging in Zirconium Alloys: *Young Suk Kim*[1]; Sung Soo Kim[1]; [1]Korea Atomic Energy Research Institute

Tensile tests were conducted from RT to 400°C using the tensile specimens taken from the tangential (TD) and longitudinal directions (LD) of a Zr-2.5Nb tube with a strong tangential texture. The yield stress plateau (YSP), representing dynamic strain aging (DSA) of zirconium alloys, appeared from 150°C for the TD specimens but from 250°C for the LD specimens. To understand the cause of the YSP, the deformation mode was determined by measuring a textural change with temperature in the TD and LD specimens before and after the tensile tests using an X-ray method. It is found that the YSP for the TD and LD specimens is due to the operation of different deformation modes with orientation: twinning in the TD and <c+a> slip in the LD, respectively. Furthermore, negative temperature dependence of ductility from 200°C to 350°C is caused by the operation of <c+a> slip.

3:50 PM Break

4:05 PM

The Thermal Stability and Weldability of a Lean Grade of Duplex Stainless Steel: *Julie Tucker*[1]; George Young[1]; [1]Knolls Atomic Power Laboratory

Duplex stainless steels are desirable for use in power generation systems due to their attractive combination of strength, corrosion resistance, and cost. However, thermal embrittlement at high temperature (~1300°F) can complicate fabrication and embrittlement at low temperatures (≤800°F) limits the service temperature for many applications. New lean grade alloys may improve the manufacturing margin and potentially increase the upper service temperature of these alloys. The present work assesses the weldability and thermal stability of lean grade AL2003. Weldability was assessed by the transvarestraint test. Thermal stability was investigated via a series of isothermal agings between 550°F and 1300°F for times between 1 and 10,000 hours. The thermal stability of the aged samples was characterized by changes in hardness, Charpy impact energy, and elastic-plastic fracture toughness. Additionally, microstructural characterization of the aged samples via electron backscatter diffraction and transmission electron microscopy was performed to better understand the nature of the embrittlement.

4:25 PM

Microstructural and Mechanical Characteristics of Friction Stir Welded ODS Alloys: *Ramprashad Prabhakaran*[1]; J. Wang[2]; K. Chitrada[3]; W. Yuan[2]; I. Charit[3]; J. Cole[1]; R. Mishra[2]; [1]Idaho National Laboratory; [2]Missouri University of Science and Technology; [3]University of Idaho

Efforts are underway to examine the feasibility of using oxide dispersion strengthened (ODS) alloys for various high temperature applications including advanced nuclear reactors. Conventional fusion welding of ODS alloys causes various undesirable effects, such as coalescence of oxide dispersoids and significant porosity. In this study, MA956 and MA754 alloys were friction stir welded in a bead-on-plate configuration. Microhardness testing across the weld zone was performed to reveal the microstructural gradient in the weld. Sub-size tensile testing of the base material and stir zone were carried out to evaluate the tensile properties. Higher weld efficiencies have been achieved in both the alloys. Optical microscopy and transmission electron microscopy were used to study the changes in grain size and characteristics of the fine oxide dispersoids across the weld zone.

4:45 PM

Diffusion of Silver and Gold in Ion Irradiated Glassy Polymeric Carbon: Malek Abunaemeh[1]; *Ibidapo Ojo*[1]; Claudiu Muntele[1]; Daryush Ila[1]; [1]Alabama A&M University

In this study we investigate glassy polymeric carbon (GPC) as an alternative to PyC as a diffusion barrier for fission products in TRISO fuel. Silver diffusion is of particular interest. GPC can maintain dimensional and chemical stability in adverse environment and very high temperatures (up to 3000°C). In this work, we are looking at the diffusion of 5 MeV implanted Ag and Au in GPC. The implantation fluences were chosen such that they would produce an estimated 1 dpa. We used three types of GPC materials, pyrolyzed to 1000, 1500 and 2000°C. We sequentially heat treated and measured the metal implanted GPC, in order to create a series of diffusion profiles from which to extract diffusion coefficients in irradiation damaged and nonirradiated GPC. For measuring the metal distribution we used Rutherford backscattering spectroscopy and the simulation software RUMP.

5:05 PM

Late-Blooming Phase Investigation in an Ion Irradiated Fe-1wt.%Mn Alloy: *Estelle Meslin*[1]; Bertrand Radiguet[2]; Philippe Pareige[2]; Alain Barbu[1]; [1]CEA; [2]CNRS/University of Rouen

The mechanism at the origin of the formation of solute clusters in RPV steels is still subject to debate (irradiation enhanced or irradiation induced). Indeed, excepted for Cu, solute atoms usually present in solute clusters observed in neutron irradiated RPV steels are known to be under-saturated, at least considered separately, but solubility limits in more complex alloys remain unknown.In order to clarify this mechanism, an under-saturated FeMn binary alloy was self-irradiated within the multi-beam facility Jannus in Saclay at 400°C up to 2 dpa and then characterized by atom probe tomography. Experimental procedure and results will be presented.

5:25 PM

The Effects of Stress and Temperature on the Fatigue Crack Growth Behavior and Microstructural Evolution of Alloy 230: *Jatu Burns*[1]; Megan Frary[1]; [1]Boise State University

Alloy 230, a nickel-based superalloy, is a candidate material for heat exchangers in very high temperature reactors. The heat exchangers are expected to see a wide range of operating conditions, so it is important to understand the effects of both stress and temperature on mechanical behavior and microstructural evolution. We studied crack growth behavior under controlled stress intensity factor near the threshold regime between 650 and 800°C. The tests were performed in using static and cyclic loads with various frequencies and stress ratios. In the different temperature regimes, the damage mechanism is dominated by different phenomena. Electron backscatter diffraction was used to observe the cracking mode and to characterize microstructure and texture at the crack tip. Transgranular cracks were observed from fatigue cracking. In grains near the crack tip, the local orientation spread increased in to as much as 5°, an indication of local deformation.

5:45 PM

High-Temperature Corrosion of YSZ Plasma-Sprayed on Nickel-Alloys in Molten Chloride Salts: *Oscar Quintana*[1]; J. Ernesto Indacochea[1]; Mark Williamson[2]; Christine Snyder[2]; [1]University of Illinois; [2]Argonne National Laboratory

The objective of this work is to develop advanced structural materials that are strong and stable under conditions typical of pyrochemical processing systems, such as electrolytic reduction, designed for used nuclear fuel recycle. Two structural materials, 316L SS and HAYNES 214, were plasma sprayed with a NiCrAlY metallic bond coat layer, and an 8% Yttria stabilized zirconia top coat layer. One set of coated specimens was submerged for 72 hours in molten LiCl+6 wt.%Li_2O at 650°C with Ar+10%O_2 gas bubbling at two different flow rates (33ml/min and 3ml/min), and another set was exposed to the vapors, suspended above the molten salt. All tests were carried out in an

argon-atmosphere glovebox. The submerged coated specimens failed while the suspended ones did not. XRD, SEM-EDS detected Li_2ZrO_3 and Li_4ZrO_4 as well as other oxide phases on the corroded specimens.

Modeling, Simulation, and Theory of Nanomechanical Materials Behavior: Nanotubes, Soft Materials and Biomedical Applications

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Thomas Buchheit, Sandia National Laboratories; Sergey Medyanik, Washington State Univ.; Douglas Spearot, University of Arkansas; Lawerence Friedman, Penn State University; Edmund Webb, Sandia National Laboratories

Tuesday PM Room: 304
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Markus Buehler, Massachusetts Institute of Technology; Michael Chandross, Sandia National Laboratories

2:00 PM Invited
Molecular Scale Modeling of Polymer Nanolithography: *Michael Chandross*[1]; Gary Grest[1]; [1]Sandia National Laboratories

The production of surfaces with controllable/tunable nanostructures over large areas and at throughputs practical for commercial applications can be very difficult. Two processes of recent interest have been step-flash imprint lithography (SFIL) and nanoimprint lithography (NIL) in which nanoscale masks are imprinted into polymeric materials to create features with nm-scale resolution. Empirical approaches are currently the norm for industrial scale-up but are often prohibitively time-consuming and expensive. Modeling and simulation can decrease manufacturing process design cycle time enormously, as has been proven in many industry segments. Here we present our activities specifically with regard to nanopatterning by detailed large-scale simulations of nanolithographical processes in which rigid molds are imprinted into liquid oligomers that are subsequently hardened. We use a generic polymer model that can be applied to both SFIL, in which the oligomers are cross-linked by exposure to UV irradiation, and NIL, in which the liquid is hardened by lowering the temperature below the glass transition. Multiple stamps are inserted into melts of liquid oligomers at a temperature above the glass transition. The melts are either quenched or cross-linked and the systems are equilibrated. Stamps are then either removed at constant velocity to study the effects of stress and adhesion on resulting features, or simply deleted to study the effects in the limit of zero stress. We vary the size and pitch of the stamps in order to study the resolution limits of both methods.

2:30 PM
Atomistic Simulations of the Nanoindentation of Cyclotrimethylene Trinitramine (RDX) (001) Surfaces: *Marc Cawkwell*[1]; Kyle Ramos[1]; Daniel Hooks[1]; [1]Los Alamos National Laboratory

Recent nanoindentation studies of cyclotrimethylene trinitramine (RDX) single molecular crystals have provided the first convincing evidence for the homogeneous nucleation of dislocations around the theoretical yield stress of a molecular material [K.J. Ramos, D.E. Hooks, and D.F. Bahr, Phil. Mag (in press)]. Furthermore, the indentation of (001) surfaces suggests a previously unknown slip system is activated. We have undertaken a series of very large-scale molecular dynamics simulations of the nanoindentation of RDX surfaces using an accurate and transferable non-reactive molecular potential for nitramines. These simulations confirm that dislocation loops corresponding to the previously unknown slip system (001)[010] nucleate homogeneously beneath the indenter. We find that the location of the homogeneous nucleation events are best described by a Schmid-like criterion based on the location of the maximum resolved shear stress beneath the tip rather than Miller and Acharya's stress gradient criterion or commonly used Hertzian analyses.

2:50 PM
Atomistic Simulation Studies of Rotational Effects in Double-Wall Carbon Nanotubes: *Iman Salehinia*[1]; Sergey Medyanik[1]; [1]WSU

Carbon nanotubes are important building blocks in the design of novel nanomechanical devices. The weak van der Waals interaction between the walls of a double-wall carbon nanotube (DWCNT) allows for an easy slide and rotation of the tubes with respect to one another. This property provides a possibility to construct a new family of mechanical nanodevices where the nanometer scale motion is the relative sliding, rotation or helical bolt-nut motion of nanotube walls. In this work atomistic simulation method is applied to study the transmission between translational and rotational motions (helical motion) in double-wall carbon nanotubes. DWCNTs are classified in several cases and general rules regarding the possibility of bolt-nut behavior are found. It is found that the helical motion is dependent on both the chiralities of the inner and outer tubes. Types of DWCNTs that are the most applicable bolt-nut pairs for being used in nanomechanical devices are identified.

3:10 PM Invited
Insights into the Mechanical Behaviour of Glassy Polymers through Molecular Dynamic Simulations: *Sumit Basu*[1]; Dhiraj Mahajan[1]; [1]Indian Institute of Technology Kanpur

In this work we use Molecular Dynamics (MD) simulations of glassy polymers with a view to informing and enriching constitutive models for these materials. Firstly, we discuss effects of sample preparation, sample size and simulation parameters that influence the ability of Molecular Dynamics to simulate realistic deformation behaviour of glassy polymers. We further show that if proper simulation methodologies are employed, MD does provide important insights into various aspects of the mechanical behaviour of polymers. These include their ability to harden remarkably at large strains and aging and rejuvenation. We illustrate that the both these aspects of their mechanical behaviour have important bearing on the mechanics of fracture initiation in these materials. Finally, our MD simulations, coupled with primitive path analyses show that stress induced dis-entanglement takes place in glassy polymers during deformation. Moreover, formation of voids during deformation affect the local entanglement densities in a significant manner.

3:40 PM Break

4:00 PM Invited
Nanomechanical Properties of Human Vimentin Intermediate Filaments: *Markus Buehler*[1]; Zhao Qin[1]; [1]Massachusetts Institute of Technology

Intermediate filaments (IFs), in addition to microtubules and microfilaments, are one of the three major components of the cytoskeleton in eukaryotic cells. Here we present the development of a full atomistic molecular model of the vimentin dimer and tetramer, based on a bottom-up molecular dynamics simulation approach. We report an analysis of the behavior of IF dimers and tetramers under mechanical stress, including studies of changing the pulling velocity and a detailed analysis of the deformation and rupture mechanisms. We observe a transition of alpha-helices to beta-sheets under mechanical deformation, as has been observed indirectly in earlier experimental studies in similar materials, enabling the protein filaments to sustain large tensile strain in excess of several hundred percent strain at very large failure strength approaching several nN. We compare our results quantitatively with AFM results of IF stretching, showing good agreement. We also discuss the mechanics of IF networks in cells.

4:30 PM
Multi-Scale Model for the Extreme Piezoresistivity in Silicone/Nickel Nanostrand Nanocomposites: Oliver Johnson[1]; *George Kaschner*[2]; Thomas Mason[2]; David Fullwood[1]; Brent Adams[1]; George Hansen[3]; [1]Brigham Young University; [2]Los Alamos National Laboratory; [3]Conductive Composites Company, LLC.

Extreme piezoresistivity was discovered in a novel Silicone/Nickel Nanostrand/Nickel Coated Carbon Fiber (Si/NiNs/NCCF) nanocomposite. The three-dimensional structure of this nanocomposite system has been investigated using Focused Ion Beam (FIB) and Scanning Electron Microscopy (SEM). The inter-nanoparticle distance distribution has been established. A novel technique was developed to study the charge transport phenomena responsible for the extreme piezoresistivity in the Si/NiNs/NCCF system using conductive nanoindentation. Using this information, finite element simulations have been developed to investigate the evolution of the inter-nanoparticle distance distribution with bulk material deformation. A quantitative quantum mechanical

tunneling (QMT)/percolation model has been developed based upon first principles, which bridges the gap between quantum effects at the nanoscopic scale and bulk material response at the macroscopic scale. The predictions of this model are compared to experimental measurements conducted under various environmental conditions.

4:50 PM Invited
Nanomechanical Energy Exchange and Dissipation: *Jeffrey Grossman*[1]; P. Alex Greaney[1]; [1]Massachusetts Institute of Technology

We review our recent work employing classical molecular dynamics simulations to understand the details of vibrational energy transfer at the nanoscale, and ways in which the unique behaviors of nanomechanical energy exchange could be utilized for the development of completely novel detection strategies. In addition, we present results from the simulation of dissipation in carbon nanotube resonators, where anomalous dissipation is observed during the ringdown of a flexural mode, leading to variations in the quality factor of more than an order of magnitude.

5:20 PM
Study of Thermo Mechanical Behavior of Plasma Nano Coated TiNi Shape Memory Alloy (SMA) for Biomedical Applications Using Finite Element Method: *Payodhar Padhi*[1]; Ramakanta Behuria[2]; [1]Hi-Tech Medical College and Hospital; [2]Konark Inistitute of Science and Technology

Shape-memory alloys (SMAs) are metals that at a certain temperature revert back to their original shape when being strained. The combination of good biocompatibility, good strength and ductility with the specific functional properties of SMA such as the shape memory effect, damping capacity and superelasticity create a smart material for biomedical applications. Some of the most innovative SMA devices include: the self expanding stents, shape memory staples, vascular filters, orthodontic arch wires and porous implants. In present study the temperature distribution induced during plasma coated was calculated using a two-dimensional finite element model (FEM) in ANSYS, finite element solver. To reduce computation time, a part of the NiTi-coated side affected by the plasma will be modeled. The size of the model could be further reduced using axis of symmetry. The film was simulated using 5-node rectangular shell. The paper studies the thermo mechanical effect of SMA for an expanding stents.

5:40 PM
Spatial Nonlocality and the Viscosity of Polymer Melts toward their Glassy State: *Ruslan Puscasu*[1]; Billy Todd[1]; Peter Daivis[2]; Jesper Hansen[1]; [1]Swinburne University of Technology; [2]RMIT University

Once the confinement of fluids approaches molecular dimensions, classical theory must be generalized to allow for local position dependent coefficients. It has been recently shown that for flow fields with high gradients in the strain rate over the with of the real-space kernels nonlocality plays a significant role [Todd et al., Phys. Rev. Lett. 100, 195901 (2008)]. An extended analysis of the exact nonlocal viscous kernel for undercooled polymer melts is presented. We compute the k-space and r-space kernels calculated from the stress and transverse momentum density autocorrelation functions. Functional forms have been found to fit the kernel data. The results show that the kernels have a width of a few molecular diameters which means that the generalized transport coefficients must be used in predicting the flow properties of fluids on length scales where the gradient in the strain rate is of the order of these dimensions (eg in nanofluidics).

Neutron and X-Ray Studies of Advanced Materials III: Applications of Line Profile Analysis

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Titanium Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Jaimie Tiley, Air Force Research Laboratory; Erica Lilleodden, GKSS Research Center; Peter Liaw, University of Tennessee; Yandong Wang, Northeastern University

Tuesday PM Room: 303
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Emil Zolotoyabko, Technion, Israel; Peter Liaw, University of Tennessee Knoxville

2:00 PM Keynote
X-Ray Line Profile Analysis – An Ideal Tool to Quantify Structural Parameters of Nanomaterials: *Michael Zehetbauer*[1]; Erhard Schafler[1]; Michael Kerber[1]; Sigrid Bernstorff[2]; Tamas Ungar[3]; [1]University of Vienna; [2]Sincrotrone Trieste S.C.p.A.; [3]Eötvös Lorand University Budapest

For a long time the shift and broadening of Bragg profiles have been used to evaluate internal stresses and coherent domain sizes, i.e. the smallest crystalline region without lattice defects. Modern technology provides both enhanced detector resolution and high brilliance X-ray sources thus allowing measurements with high resolution in space and time. In parallel to the hardware, also diffraction theories have been substantially improved so that the shape of Bragg profiles can be quantitatively evaluated not only in terms of the crystallite size and -distribution, but also in terms of the density, type and arrangement of dislocations, twins and stacking faults. Thus state-of-the-art X-ray line profile analysis enables the thorough characterization especially of nanostructured materials which also contain lattice defects. The method can be also used to prove the existence of dislocations in crystalline material. Examples with nanostructured metals, ceramics, polymers and even molecular crystals like fullerenes are given.

2:30 PM Invited
In-Situ Synchrotron and Neutron Diffraction Studies of Deformation Behaviors at Small Length Scales: *Xun-Li Wang*[1]; Sheng Cheng[2]; Alexandru Stoica[1]; Joe Horton[1]; C. T. Liu[3]; Peter Liaw[2]; Liang Zuo[4]; [1]Oak Ridge National Laboratory; [2]University of Tennessee; [3]Hong Kong Polytechnic University; [4]Northeastern University

There have been considerable debates over the deformation mechanisms at small length scales. In-situ synchrotron and neutron diffraction were used to study deformation mechanisms in Ni over a broad range of grain sizes, under both monotonic and cyclic loading conditions. The experimental data show that unlike in coarse-grained metals, where the deformation is dominated by dislocation slip, plastic deformation in nanocrystalline Ni is mediated by grain-boundary activities, as evidenced by the lack of intergranular strain and texture development. For ultrafine-grained Ni, although dislocation slip is an active deformation mechanism, deformation twinning also plays an important role, whose propensity increases with the grain size. This research was supported by U.S. Department of Energy (DOE), Office of Basic Energy Sciences, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC. LZ and XLW thank Natural Science Foundation of China for supporting their collaborative research (No. 50528102).

2:50 PM Invited
Neutron Diffraction and Micromechanics Studies of the Fatigue Crack Deformation Behavior: *Yanfei Gao*[1]; Rozaliya Barabash[2]; Lili Zheng[1]; Sooyeol Lee[1]; Hahn Choo[1]; Peter Liaw[1]; [1]University of Tennessee; [2]Oak Ridge National Laboratory

The general deformation characteristics near a fatigue crack tip include a plastic zone in front of the crack tip and a plastic wake left behind, where the cyclic loading and fatigue crack growth lead to a compressive strain. The magnitude and distribution of the compressive strain in this plastic wake depend on the stress multiaxiality, material properties, and crack growth increment in each loading cycle. We compare lattice strain measurements by the neutron diffraction technique and simulations by an irreversible and hysteretic cohesive-

interface model, which is developed to simulate the fatigue crack nucleation and growth. Crystal plasticity simulations have been conducted to relate the macroscopic and lattice strains. Good agreement between micromechanical models and neutron strain measurements has been reached.

3:10 PM Invited
Diffraction Line Profile Analysis for the Study of Transformation Kinetics in Nanocrystalline Materials: *Paolo Scardi*[1]; [1]University of Trento

Diffraction techniques in kinetics studies are mostly employed to identify and quantify phase changes. However, further information can be obtained from the study of the line profiles. This is especially useful when studying the evolution of nanocrystalline and of heavily deformed materials during high temperature kinetics. Modern methods of diffraction profile modeling provide a detailed picture of the nanostructure in terms of crystalline domain size/shape distribution and density of lattice defects, together with the usual information on phase composition. This information can be collected also during the kinetics, by in situ measurements. As data quality is an important issue in line profile studies, the high brilliance and focusing of synchrotron beams are a valuable support to follow fast kinetics. This contribution will show results of line profile analysis in kinetics studies of grain growth in different nanocrystalline systems. Recent advancements in line profile analysis will also be reviewed.

3:30 PM Invited
Single-Grain Microstructure from Polycrystalline Specimens: *Tamás Ungár*[1]; [1]Eötvös University Budapest

X-ray line profile analysis proves to be a powerful method to characterize the microstructure of crystalline materials. In its conventional application it provides data about the average properties of large aggregates of crystallites. Synchrotrons facilitate to extract single-grain or single-crystallite microstructure properties in powder- or polycrystalline specimens. The lecture will deal with the method itself, first results provided by the method and the scientific possibilities offered by the procedure.

3:50 PM Invited
Evolution of Microstructure during Tensile Deformation of TWIP Steel: X-Ray Line Profile Analysis: *Hahn Choo*[1]; Tamas Ungar[2]; Yang Ren[3]; Sang-Ho Han[4]; [1]University of Tennessee; [2]Eotvos University; [3]Argonne National Laboratory; [4]POSCO

The evolution of average grain size, dislocation density, twin density, and crystallographic texture was investigated during the tensile deformation of twinning-induced plasticity (TWIP) steel using high-energy synchrotron x-ray diffraction and line profile analysis. TWIP steel, recently developed by POSCO for automotive applications, exhibits an excellent combination of high strength (about 1 GPa tensile strength) and ductility (about 65% total elongation). In this study, we investigated the volumetric evolution of twin volume fraction and dislocation density as a function of the tensile plastic deformation to further the understanding of the interaction between the twin and dislocation and its influence on hardening and ductility of the TWIP steel.

4:10 PM Invited
Neutron Diffraction and EPSC Modeling: *Bjørn Clausen*[1]; Donald Brown[1]; Carlos Tomé[1]; Laurent Capolungo[2]; Sean Agnew[3]; [1]Los Alamos National Laboratory; [2]Georgia Tech-Lorraine; [3]University of Virginia

In-situ neutron diffraction measurements provide volume averaged bulk information about the evolution of internal strain, texture and defect concentration during deformation. The information contained in each diffraction peak originates solely from the sub-set of grains within the polycrystal that has the given lattice plane normal parallel to the scattering vector for the detector. This level of detail is well matched by the EPSC model that is based upon a discrete set of single crystals with an orientation distribution representative of the actual texture of the material. By selecting sub-sets of grains based upon their orientation is it possible to generate average values of elastic strains, weight fractions and dislocation densities from the model that can be directly compared to the peak shifts, peak intensities and peak widths from the diffraction measurements. The two techniques are frequently used in combination to elucidate material properties not probable by other means.

4:30 PM Break

4:40 PM Invited
Phase Transformation and Tensile Behavior of a Bainite Steel Studied by In Situ Neutron Scatteing and Dilatometry: *Yo Tomota*[1]; Min-Seo Koo[1]; [1]Ibaraki University

Bainitic transformation was monitored simultaneously by a dilatometer test and neutron scattering. The changes in lattice constants and volume fractions of bainitic ferrite and the retained austenite were measured by high angle diffraction while the size distribution of bainite lath by small angle scattering. In both cases, the transformation kinetics was estimated from the specimen length change with the dilatometer. These three data show good agreements and hence the detailed mechanism of the banitic transformation at a very low temperature like 573K resulting in nano-size scaled layer structure with ferrite and carbon enriched austenite was totally discussed. Then, the tensile behavior of the bainite steel was studied by neutron diffraction during tension test. The stress partitioning between ferrite and austenite, i.e, phase strains and among differently hkl oriented grains., i.e., intergranular strains and stress-induced martensitic transformation were found to change with work-hardening.

5:00 PM Invited
Line Broadening Analysis of High Resolution X-ray Data: *I. Noyan*[1]; Andrew Ying[1]; Conal Murray[2]; [1]Columbia University; [2]IBM Research Division

We report thickness values measured from a set of ideal samples (semiconductor grade silicon-on-insulator thin films) using the Scherrer equation (FWHM and integrated-breadth variants), rocking-curve modeling, thickness fringe analysis, Fourier analysis, the Warren-Averbach method (single peak and multiple-order variants), the Williamson-Hall method as well as from x-ray reflectivity and cross-sectional transmission electron microscopy. We will compare these results and discuss the applicability of these techniques to various types of samples.

5:20 PM
Characterization of Pt Nanoparticles by Debye Function Analysis of the X-Ray Diffraction Pattern: *Kenneth Beyerlein*[1]; Paolo Scardi[2]; Bob Snyder[1]; [1]Georgia Institute of Technology; [2]University of Trento

One of the most promising and immediate applications of nanoparticles is their use as highly efficient, energy saving catalysts for industrial chemical synthesis. Their efficiency is dependent on the small particle size and in some cases the particle shape. A whole pattern Debye Function Analysis technique of extracting this important microstructure information from the x-ray diffraction pattern will be presented. Platinum nanoparticles prepared by a salt reduction process with sizes ranging from 1-20nm were the focus of this study. Synchrotron diffraction patterns from these particles were analyzed by this Debye approach with the size distribution, particle shape abundance, and faulting densities determined. The results of this analysis will be compared with the quantities obtained by Transmission Electron Microscopy image analysis.

5:30 PM Invited
Protein Powder Diffraction and Materials Science: *Robert Von Dreele*[1]; [1]APS/Argonne National Laboratory

Protein crystallography and materials science might appear to be non-intersecting fields of science, however powder diffraction provides insight into the materials science aspects of crystalline proteins. Surprisingly, proteins provide extremely sharp powder diffraction patterns that are sensitive to sample environment and composition (pH, salt concentration, etc.). Amongst other things, these changes provide a means of extracting sufficient information to partially overcome the extreme peak overlap problem inherent in protein powder patterns. Protein powder patterns also allow examination of protein crystallization, the effect of radiation damage and crystalline phase changes. This talk will review the current status of these and other aspects of this new field.This work was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under contract number DE-AC02-06CH11357.

5:50 PM
Quantifying the Evolution of Lattice Strain due to Cyclic Loading in 7075 Aluminum Alloy: *Jay Schuren*[1]; Matthew Miller[1]; Alex Kazimirov[2]; [1]Cornell University; [2]Cornell High Energy Synchrotron Source

X-ray diffraction experiments during in-situ cyclic mechanical loading were used to quantify the lattice strain distributions within a polycrystalline

aluminum alloy (AA7075-T6) at various points in the cyclic loading. By isolating the changes between the lattice strain distributions at different points in the sample's cyclic life we investigated the evolution of the lattice strain distributions as the sample progressed toward fatigue failure. The focus of this presentation will be on a new method for quantifying the image specific experimental resolution, the requirement of a statistically significant population of crystals in each measurement, and the evolution of lattice strain distributions due to cyclic loading in AA7075-T6 undergoing zero-tension macroscopic loading.

Pb-Free Solders and Emerging Interconnect and Packaging Technologies: Reliability (II)

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Kwang-Lung Lin, National Cheng Kung University; Sung Kang, IBM; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Iver Anderson, Iowa State University; Fu Guo, Beijing University of Technology; Thomas Bieler, Michigan State University; Andre Lee, Michigan State University; Rajen Sidhu, Intel Corporation

Tuesday PM Room: 204
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Rajen Sidhu, Intel Corp.; Alexandre Kodentsov, Eindhoven University of Technology

2:00 PM
Impact of Isothermal Aging on Long Term Reliability of Fine Pitch Ball Grid Array Packages with Sn-Ag-Cu Solder Interconnects: *Tae-Kyu Lee*[1]; Weidong Xie[1]; Kuo-Chuan Liu[1]; Thomas R. Bieler[2]; [1]Component Quality and Technology, Cisco Systems; [2]Chemical Engineering and Materials Science, Michigan State University

The interaction between isothermal aging and long term reliability of fine pitch ball grid array packages with Sn-3.0Ag-0.5Cu (wt%) solder interconnects are investigated. In this study, 0.4mm fine pitch packages with 300\956m diameter Sn-Ag-Cu solder balls are used. Two different die sizes and package substrate surface finishes are selected to compare the internal strain impact and alloy effect during thermal cycling. The samples are isothermally aged and thermal cycled from 0 to 100°C with a 10minute dwell time. Based on weibull plots for each condition, the lifetime of the package reduced approximately 45% with 150°C aging precondition and with longer time for 100°C aged samples. The microstructure evolution is observed during thermal aging and thermal cycling, focused on Sn grain single to multi grain transformation. Different mechanisms after aging in various conditions are observed and their impact on lifetime reduction is discussed.

2:15 PM
Pb-Free Process Development for Microcircuit Package Assemblies – A University/Industry Design Project Collaboration: *Mike Powers*[1]; Jim Shackelford[2]; Derek Fong[2]; Zi Gwen Kwan[2]; Tammy Leung[2]; Kit-Ying Mak[2]; Enrique Pedron[2]; Raminderdeep Sidhu[2]; Hong-Siang Wei[2]; [1]Agilent Technologies; [2]University of California Davis

A team of engineering students in the Department of Chemical Engineering and Materials Science at the University of California Davis collaborated on a design project investigation to develop a cost effective process for Pb-free solder assembly of hybrid microcircuit package assemblies employed in test and measurement instruments (a "real" materials design project). The students considered the required materials properties, as dictated by the component assembly design, and how the properties are affected by materials selection and the joining process. The team conducted solder joint strength, Au embrittlement and solder wetting experiments at UC Davis in order to evaluate the process design. The team also conducted process development, including furnace profiling and reliability testing, at Agilent facilities in Santa Rosa, California. Through applied research and testing the student team developed a reliable Pb-free process for solder assembly of the hybrid microcircuit package assembly project vehicle.

2:30 PM
Reliability Evaluation of Cu-Cored Solder Joints: *YunSung Kim*[1]; Eunsik Kim[2]; Heylim Choi[1]; Jungtak Moon[2]; Heeman Choe[2]; [1]Kookmin University; [2]MK Electron Co

Recently, copper-cored solder balls have attracted attention in microelectonic packaging community for two main reasons; first, they are known to have enhanced resistance to electromigration failure. Second, Cu-cores in the solder balls can serve as a spacer during reflow and prevent the solder balls from touching each other when the ball size and pitch is very small as in high-density BGA or CSP packages. As little data is available in the literature primarily on the shear strength of Cu-cored solder joints, an extensive study is required to evaluate fully the reliability of Co-cored solder joints both at package- and board-levels. In this study, we are assessing the physical and mechanical properties of the Cu-cored solder joints using several test methods, such as thermal cycling and high-speed ball pull tests to compare to those of conventional Sn-Ag-Cu solder joints.

2:45 PM
Roles of Service and Materials Parameters on Reliability of Pb-Free, Sn-Based, Solder Joints: *K.N. Subramanian*[1]; Andre Lee[1]; [1]Michigan State University

Isothermal mechanical testing of single shear-lap specimens of Pb-free, Sn-based, solder joints at various temperatures ranging from -55°C to 150°C indicate different modes of deformation to be dominant in different temperature ranges. These observations also indicated the role of strain-rate on the mode of deformation. Importance of these findings on thermomechanical behavior of high Sn-based solder joints will be discussed along with their implications on testing parameters employed in accelerated thermal cycling tests. Results from repeated thermal shock, thermomechanical fatigue (with different dwell-times at temperature extremes, different heating- rates, and in different temperature ranges), and iso-thermal stress relaxation studies will be presented to illustrate these implications. These findings bring out the critical issues that need to be taken into consideration in designing accelerated thermomechanical test methods for long-term reliability of Sn-based solder joints.

3:00 PM
Thermal and Mechanical Fatigue Reliability of TiW/Cu and Al/Cu Underbump Metallizations on Glass Substrate: *Yong Jun Oh*[1]; Chul Min Choi[1]; Jae Ho Kim[1]; [1]Hanbat University

Direct formation of interconnect lines and underbump metallization (UBM) on glass substrates is a promising technique that enables simple and cost-effective sensor chip packaging. In this study, the joint reliability of SnAg solder/UBM(TiW/Cu or Al/Cu)/glass under thermal and bending fatigue cycles is evaluated. The TiW/Cu UBM is fabricated by sputter deposition of TiW onto glass and electroplating of Cu, while the Al/Cu UBM is formed by anodic bonding of glass to Al and electroplating of Cu onto Al. SnAg solder bumps are electroplated on both the UBMs and reflowed at 260°C. The samples are exposed to temperature cycles between -20 and +120°C. The three-point bending test is conducted for rectangular specimens with a dimension of 10×20 mm^2 (LxW) at ambient temperature. The failure mechanisms under thermal and mechanical fatigue are presented from the viewpoint of the strength of each layer and its bonding strength with the joined materials.

3:15 PM
Accelerated Life Prediction of Electrochemical Ion Migration on ENIG Surface Finish Circuit: *Won Sik Hong*[1]; No Chang Park[1]; [1]Korea Electronics Technology Institutue(KETI)

Electrochemical metallic ion migration(ECM) can be generated by electrochemical reaction between anodic and cathodic electrode on electric circuit in case of existing temperature, humidity and applied voltage, and finally induce to malfunction of electronics due to precipitation of metallic ion in cathode. Thus we have studied failure mechanism and accelerated life prediction method of ECM occurrence. Modified Eyring model which is combined stress model(temperature, humidity, voltage) was utilized to accelerated life prediction model. To obtain temperature and humidity coefficient factors of ECM failure, accelerated life test was conducted by more than f(50%) failure. Failure criterion of insulation resistance was 10E7O. From these results, we have deduced temperature and humidity coefficient factors of acceleration model for predicting ECM failure life. Also electrochemical oxidation and reduction mechanism of ECM were examined by physics-of-failure based

failure analysis. These results would be helpful to predict the life of electronic circuit due to ECM failure.

3:30 PM Break

3:45 PM

Space: The Final Frontier for Pb-Free Electronics?: *David Witkin*[1]; [1]The Aerospace Corporation

While military and aerospace electronics are currently exempt from RoHS prohibitions on Pb in electronics, the Pb-free transition in the consumer electronics industry has influenced exempt industries. Long-term costs of Pb-free interdiction and sourcing of Sn-Pb parts may soon exceed the costs to find an acceptable Pb-free solution for high-reliability systems in harsh usage environments. Space electronics systems are low-volume and high cost. In these systems, solder joints are subjected to multi-year storage times and extensive ground testing before launch, and are not accessible for inspection or repair after launch. The question arises as to whether Pb-free solders well suited to the unique assembly, test, storage and orbit environments are the same as those being pursued for other industries. Using previous reliability testing programs as a guide, Bi-containing Sn-Ag solders were selected for characterization. The microstructural evolution and mechanical properties of Bi-bearing solders were compared to SAC305, exhibiting significant differences.

4:00 PM

Creep Property of Sn-3Ag-0.5Cu-xNi/Au/Ni Joints after Aging: *Chung-Nan Peng*[1]; Jeng-Gong Duh[2]; Tae-Kyu Lee[3]; Kuo-Chuan Liu[3]; Michael Tsai[3]; [1]Department of Materials Science and Engineering, National Tsing Hua University; [2]Department of Materials Science and Engineering, National Tsing Hua University; [3]Interconnect Technology Team Reliability Engineer, Manufacturing Technology Group, CISCO

In BGA packages and flip-chip packages, SnAgCu solder alloys have been regarded as the most promising Pb-free substitutes for the SnPb solder. However, flip chip package solder joints suffer thermomechanical fatigue and creep failures due to the CTE mismatch between silicon die and substrate. In this study, creep tests were conducted on Sn-3Ag-0.5Cu-xNi/Au/Ni (x = 0-0.1 wt.%) joints at 100°C and 150°C, respectively. Various creep parameters, such as global and localized creep strain, variation of creep strain and strain-rate for creep were determined. It was demonstrated that the observed behavior could be well-fitted using creep constants for Sn-3Ag-0.5Cu-xNi/Au/Ni joints in the modified creep power law. In addition, the effect of Ni content in the solder on the grain-orientation-dependent elastic deformation state was also discussed. Microstructure characterization, including EBSD and FE-EPMA analysis, were used to describe the creep behavior of the Sn-3Ag-0.5Cu-xNi/Au/Ni joints.

4:15 PM

Interface Design of Lead-Free Electronic Interconnects: *K.N. Subramanian*[1]; Deep Choudhuri[1]; Andre Lee[1]; [1]Michigan State University

Service reliability of lead-free electronic components is significantly affected by the thermal excursions encountered during service. Catastrophic crack that develops from such thermal excursions due to coefficient of thermal expansion mismatches are invariably at the interfaces, especially at the tin/tin grain boundaries or tin/(intermetallic-compound) interfaces, near the solder/substrate interface regions. Keying such interfaces to retard sliding of mating regions, or stitching such mating regions to prevent de-cohesion and dissipate stresses by promoting plastic deformation in tin grains, enhances the reliability of the lead-free electronic interconnects. Reinforcing lead-free solders containing significant amounts of tin with Nano-structured Polyhedral Oligomeric Silsesquioxane (POSS), consisting of Si-O cages with surface active radicals, provides such attributes to enhance the service reliability of interconnects made with them.

4:30 PM

Study of the Impact Performance of Solder Joints by High-Velocity Impact Tests: *Ning Zhang*[1]; Yao Shi[2]; Fu Yang[3]; [1]Beijing University of Technology and University of Kentucky; [2]Beijing University of Technology; [3]University of Kentucky

The impact behavior of solder joints were studied using three different high-velocity impact tests of the U-notch Charpy impact test, the No-notch Charpy impact test and a lab-designed drop test. The solder joints were made of five solder alloys, Sn37Pb, Sn3.8Ag0.7Cu, Sn2.0Ag0.7Cu, Sn1.0Ag0.7Cu and Sn0.7Ag0.7Cu (in weight percent), in which the traditional Cu/solder/Cu butt joint was used. All three impact tests gave the same trend of the impact behavior of the solder joints with the Sn37Pb joints having the highest impact resistance and the Sn3.8Ag0.7Cu joints having the lowest impact resistance. For the lead-free joints, the Sn1.0Ag0.7Cu joints had better impact resistance than the Sn2.0Ag0.7Cu, and the Sn2.0Ag0.7Cu joints better than the Sn0.7Ag0.7Cu. The impact behavior was correlated to the fracture morphologies from the SEM micrographs. The comparison of three cases showed that the No-notch Charpy impact test is a promising method for evaluating the drop performance of solder joints.

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Investigation and Effects of Wafer Bow in Different 3D Stacking Schemes: *Kuan-Neng Chen*[1]; Y. Zhu[2]; W. W. Wu[1]; R. Reif[3]; [1]National Chiao Tung University; [2]IBM T J Watson Research Center; [3]Massachusetts Institute of Technology

3D stacking technology has become an attractive candidate for future packaging and semiconductor applications. The reliability of this technology is definitely a significant consideration before it can be fully used for products. The reliability of 3D stacking, which is related to the quality of stacking, is highly dependent with wafer bow. Wafer bow, also as known as "wafer warpage", is contributed by materials on the wafer and process steps. When the wafer bow is too large, the stacking quality is affected and thus a reliability issue is occurred. This paper investigated the effects of wafer bow in different 3D stacking schemes, including metal bonding, oxide fusion bonding, and SOI-based layer transfer technology. Fabrication considerations and material selections are discussed for minimizing wafer bow in 3D stacking applications.

5:00 PM

Shear and Bending Fatigue Failure of Lead Free Solder Joint and Fracture Mechanics: *Huili Xu*[1]; Woong Ho Bang[1]; Choong-Un Kim[1]; Tae-Kyu Lee[2]; Hongtao Ma[2]; Kuo-Chuan Liu[2]; [1]University of Texas at Arlington; [2]Cisco System Inc.

With continuing miniaturization of packaging structure for electronic devices, the reliability failure at solder joint is becoming of major concern in microelectronic industry. Among many mechanisms leading to solder joint failure, the fracture by cyclic bending, shear, and shock load is particularly concerned. Conventionally, those load conditions rarely contribute to the joint failure; however, with an expansion of mobile electronics combined with compact packaging structure, they are becoming equally problematic to more conventional cyclic thermal load. Consequently, there are growing numbers of research conducted on this subject in recent years, yet understanding on the involved fracture mechanics is still unclear. This paper presents the result of our investigation, both theoretical and experimental, that aim to identify the fracture mechanics active in cyclic bend and shear fatigue and the contributing factors to the fracture at each fatigue mode including type of solder alloy, mechanical constraints, and fatigue conditions.

5:15 PM

Investigating Defects in through-Silicon via (TSV) Chains by Three Dimensional Imaging Reconstruction: *Alphonse-Marie Kamto Tegueu*[1]; Robert Morris[1]; Gregory Thompson[1]; Susan Burkett[1]; [1]The University of Alabama

Metal filled through-silicon vias (TSVs) allow electronic devices to be connected using a three dimensional approach. This technology is being optimized to address the need for increased functionality and performance. Vias with tapered sidewalls are formed in our laboratory, lined with silicon dioxide, titanium, and copper as insulation, barrier, and seed films, respectively. Vias are then filled with copper by a pulsed electroplating process. The process wafer is attached to a carrier before mechanically thinning the process wafer back side. The base of the vias is exposed from the opposite side and contact pads are formed for testing the electrical resistance of vias chained together. Discontinuity in the via chains can be investigated by a procedure involving multiple polishing and imaging steps along both the diameter and depth of the vias. Three dimensional imaging reconstruction will be described for TSVs as useful method for investigating possible defects or failures.

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials IX: Session IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee
Program Organizers: Chih-ming Chen, National Chung Hsing University; Srinivas Chada, Medtronic; Sinn-wen Chen, National Tsing-Hua University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-ho Lee, Hongik University; Kejun Zeng, Texas Instruments; Yee-wen Yen, National Taiwan University of Science and Technology; Wojciech Gierlotka, AGH University of Science and Technology; Chao-hong Wang, National Chung Cheng University

Tuesday PM Room: 203
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Wojciech Gierlotka, AGH Univeristy of Science and Technology; Clemens Schmetterer, University of Vienna

2:00 PM Invited
High Temperature Lead-Free Solder: Solidification Behavior of (Cu,Ni)-Sn-Zn: *Hans Flandorfer*[1]; Clemens Schmetterer[1]; Matthieu Froger[1]; Herbert Ipser[1]; [1]University of Vienna

In order to obtain solder joints for e.g. die-attach and BGA (Ball Grid Array) solder spheres, chipscale package (CSP) and multi-chip modelling (MCM), solder materials with a higher melting regime (> 230 °C) are necessary. The hitherto used high Pb solders (up to 85 % Pb) need to be substituted by lead-free materials. Promising candidates for such lead-free solders are e.g., alloys of the systems Cu-Sn-Zn and Ni-Sn-Zn. Knowledge about these systems is highly important as it provides detailed information to the solidification behavior and formation of intermetallic phases. This is a crucial point for the control of the microstructure and texture of alloys and alloy interfaces. The systems Cu-Sn-Zn and Ni-Sn-Zn have been investigated by means of XRD, DTA and metallography including EPMA techniques.Isothermal sections of Cu-Sn-Zn and Ni-Sn-Zn at various temperatures will be presented here. The resulting phase diagrams are also based on a careful literature assessment.

2:25 PM Invited
Phase Diagrams in Lead-Free Soldering: *Clemens Schmetterer*[1]; Hans Flandorfer[1]; Herbert Ipser[1]; [1]University of Vienna

During soldering layers of intermetallic compounds (IMCs) are created, which influence the mechanical joint-stability. Knowledge of these IMCs, required to understand their formation, is conveniently summarized by the phase diagram containing the constituents of the solder and the contact material. Within COST Actions 531 and MP0602 emphasis has been placed on the experimental and thermodynamic study several intermetallic systems, like Ag-Cu-Ni-Sn and its subsystems and Ni-P-Sn for conventional soldering, or Sn-Zn and Sn-Au based systems for HT-soldering. Other systems have been studied as well in order to explore many promising systems, but some of these may only find use in niche-applications, e.g. In-Ni-Sn, due to the high price of In.Examples from several systems will be presented in this contribution. The experimental phase diagrams will be compared to thermodynamic assessments based on the COST 531 database. Their relevance for soldering and solder related processes will be shown.

2:50 PM
Phase Equilibria in the Sn-Ni-Zn Ternary System: *Jaewon Chang*[1]; Sun-Kyoung Seo[1]; Hyuck Mo Lee[1]; [1]KAIST

In the electronic packaging field, the minor addition of Ni and Zn in Sn-based Pb-free solders has been recommended to enhance the mechanical properties of solder joint. In order to thermodynamically evaluate the proper composition level of Ni and Zn as well as to analyze the reaction with the Ni substrate, the thermodynamic data are needed for the Sn-Ni-Zn ternary system. In this study, the phase equilibria of the Sn-Ni-Zn ternary system have been investigated at 473, 773, and 1073K (less than 60 at% Ni). To identify the equilibrium phases, the scanning electron microscopy (SEM), X-ray diffraction (XRD) and electron probe microanalysis (EPMA) were used. On the basis of the experimental data and thermodynamic parameters, the isothermal sections of the Sn-Ni-Zn ternary system have been modified in consideration of the ternary solubility in the binary compounds.

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Interfacial Reaction between Pure Sn and Cu Foil with Different Cu Grain Sizes: *Jo Mei Wang*[1]; Jenq Gong Duh[1]; [1]National Tsing Hua University

Cu6Sn5 and Cu3Sn were easily formed at the interface between Sn and Cu during reflow and aging process. Thick Cu-Sn compound and Kirkendall voids at the interface would reduce the mechanical strength of solder joints. The IMC growth rate in solder was related to the grain size of Cu foil. In this study, the pure Sn solder was reflowed with the Cu foils with different grain sizes at 250 oC for 1-20 min. It was found out that the Cu foil with small grain size can reduce the IMC growth. In addition, Cu6Sn5 and Cu3Sn growth rates were calculated for samples with various Cu grain sizes. The correlation between the grain size and the distribution of Kirkendall voids were also discussed and proposed.

3:30 PM
Interfacial Reactions in the Sn-In-(Zn)/Ag and Sn-In-(Zn)/Ni Couples: *Ching-feng Yang*[1]; Sinn-wen Chen[1]; [1]National Tsing-Hua University

Sn-In alloys are important low melting-point solders. Ni and Ag are commonly used in electronic products. The dissolution behaviors and intermetallic compound formation of Ag and Ni substrates in the Sn-20In solder with Zn addition up to 5 wt.% upon dissolution are examined at 230°C. The Zn addition in the Sn-20In alloy lowers the solidus and liquidus temperatures, effectively reduces the undercooling, and retards the dissolution rate of Ag substrate. The reaction product in the Sn-In-(Zn)/Ag couple is the ξ phase when Zn is less than 1 wt.%. When the Zn addition is higher than 1 wt.%, three different Ag-Zn compounds are formed. The reaction product is the Ni_3Sn_4 phase in the Sn-In-(Zn)/Ni couples when Zn is lower than 2 wt.%. The growth rate of the Ni_3Sn_4 phase is reduced with Zn addition. When the Zn addition is higher than 2 wt.%, the reaction product changes to the Ni_5Zn_{21} phase.

3:50 PM Break

4:00 PM Invited
Enhancement of Heterogeneous Nucleation of β-Sn Phases in Sn-Rich Solders by Adding Minor Alloying Elements with Hexagonal Closed Packed Structures: Moon Gi Cho[1]; Hyun You Kim[1]; Sun-Kyoung Seo[1]; *Hyuck Mo Lee*[1]; [1]KAIST

The measured undercooling of pure Sn was about 30°C due to the difficulty of nucleating a solid β-Sn phase from a liquid phase. To promote the heterogeneous nucleation of β-Sn phases, the addition of impurity elements to the solders was suggested. Among the impurity elements, alloying elements with hexagonal closed packed (HCP) structures, such as Co, Zn, Ti and Mg, were found effective to enhance heterogeneous nucleation of β-Sn phases in Sn-rich solders. Calculations of the density functional theory indicate that the interfacial energy between β-Sn and Zn was relatively low; furthermore, the surface of Zn (10-11) was estimated to have the lowest interfacial energy with β-Sn phases. Minor alloying elements with HCP crystals are expected to provide more favorable heterogeneous nucleation sites for β-Sn phases.

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Phase Diagram and Thermodynamic Properties of Ag-Cu-In-Sn Quaternary System: *Wojciech Gierlotka*[1]; Dominika Jendrzejczyk-Handzlik[2]; [1]Yuan-Ze University; [2]AGH University of Science and Technology

The Sn-Ag-In alloys are promising lead-free solders. Copper is commonly use a substrate material and in this case phases relationships and phases stabilities in Ag-Cu-In-Sn quaternary system are very important for electronic industry. The CALPHAD approach was used for thermodynamic modeling of Ag-Cu-In-Sn quaternary system as well as for all the constituents binaries and ternaries systems. Good agreement between calculations and available thermodynamic data was found.

4:45 PM
Interfacial Reaction Effect on Mechanical and Electrical Reliability in Cu/Solder/Cu Bump: *Myeong-Hyeok Jeong*[1]; Jae-Won Kim[1]; Byunghoon Lee[2]; Kiwook Lee[3]; Jaedong Kim[3]; Hoo-Jeong Lee[2]; Young-Bae Park[1]; [1]Andong National University; [2]Sungkyunkwan University; [3]Amkor Technology Korea

Cu/Solder/Cu bump makes large amount of intermetallic compound and Kirkendall void which can degrade electrical and mechanical reliability. Therefore, it is essential to understand the fundamental growth mechanisms

of IMC and Kirkendall void. And also, their effects on mechanical reliability should be systematically investigated. In this work, we performed kinetic studies on the Cu/Sn/Cu bump structure in order to quantify the amount of intermetallic compound and Kirkendall void by using in-situ annealing and electromigration test in a scanning electron microscope chamber during current stressing conditions with current density of 2.0 x 104 A/cm2 at 125~175oC. Cu6Sn5 and Cu3Sn were already formed at interface between Cu bump and solder after reflow. Intermetallic compound growth was significantly enhanced by current stressing where the growth rate follows a linear relationship with stressing time. Finally, their effect on the mechanical reliability of Cu/Sn/Cu bump will be discussed in detail.

5:05 PM

Interaction of Sn-Based Solders with Electroless Nickel Substrates: The Ni-P, Sn-P, and Ni-Sn-P Phase Diagrams: Clemens Schmetterer[1]; Rajesh Ganesan[1]; Adela Zemanova[2]; *Ales Kroupa*[2]; Herbert Ipser[1]; Alan Dinsdale[3]; [1]University of Vienna; [2]Institute of Physics of Materials, ASCR; [3]National Physical Laboratory

Electroless nickel substrates contain considerable amounts of phosphorus due to their deposition from aqueous solutions of nickel salts by means of chemical reduction using hypophosphite. In order to understand their reaction with Sn-based solders, a reliable knowledge of the ternary Ni-P-Sn phase diagram is necessary. CALPHAD-type calculations can provide consistent phase diagram information. In the present case experimental phase diagram information was provided for the three title systems. In addition, experimental partial thermodynamic data were obtained for the systems Ni-P and Sn-P, and they were combined with calorimetric measurements of the enthalpy of formation as well as with ab-initio calculations. All these experimental data served as an input into a CALPHAD optimization of the two binary systems Ni-P and Sn-P, and together with available data on the Ni-Sn system, a first attempt was done to extrapolate the ternary Ni-P-Sn system and compare it with the experimental version.

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The Interfacial Reactions of Snagin Pb-Free Solders on Cu Substrates: *YuYan Jieng*[1]; Albert T. Wu[1]; [1]National Central University

Pb-free SnAgIn solder system is an appropriate candidate for its low liquidus temperature that is close to eutectic SnPb solder. Various solder compositions of SnAgIn system were alloyed while the amount of Ag was fixed at 3 wt%. Solders of different compositions were reflowed on Cu substrates at various temperatures for different period of times. Samples were also annealed at 120°C, 150°C and 180°C for prolonged hours for the investigation of solid state aging. The thicknesses of the interfacial compounds, Cu6(Sn,In)5 were calculated, and the kinetics of the growth of compounds is discussed in this paper. ζ-phase particles, solid solution of Ag4Sn and Ag3In, formed in the solders during reflow and solid state aging. Thermodynamic equilibrium of these phases at various test conditions will be presented.

Processing Materials for Properties: Light Weight Materials Processing

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division
Program Organizers: Brajendra Mishra, Colorado School of Mines; Akio Fuwa, Waseda University; Paritud Bhandhubanyong, National Metal and Materials Technology Center

Tuesday PM Room: 617
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Rachman Chaim, Technion - Israel Institute of Technology; Raman Singh, Monash University

2:00 PM Keynote

Recent Developments in Processing/Structures/Properties Relationships of Titanium Alloys: *Greg Oberson*[1]; Sreeramamurthy Ankem[2]; [1]U.S. Nuclear Regulatory Commission; [2]University of Maryland, College Park

While titanium has long been used as a structural material, the 21st century will see increasing demand for advanced titanium alloys to meet technological challenges for applications in the aerospace, biomedical, and energy fields, among many others. Variation of alloying elements and processing conditions of titanium alloys can be used to tailor the microstructures and hence properties such as strength, fracture toughness, creep resistance, corrosion resistance, and biocompatibility. This paper discusses recent findings regarding the effect of processing on microstructure and low-temperature ($<0.25*T_m$) creep behavior of single-phase and two-phase titanium alloys. In particular, the role of alloy chemistry, interstitial impurities, and phase morphology will be considered. These findings are useful to predict the creep behavior of presently used titanium alloys and to develop new processing techniques for advanced titanium alloys with enhanced creep resistance.

2:30 PM

Effect of Casting Mould on Mechanical Properties of 6063 Aluminium Alloy: *Wasiu Ayoola*; Samson Adeosun[1]; Olujide Sanni[1]; F. Ochulor[1]; [1]University of Lagos

There are various modern processes usually employed in the production of castings vis-a-vis; sand-mould, permanent-mould, die and centrifugal castings. Sand-mould process is known to induce peculiar microstructures depending on average grain size, grain size distribution, grain shape and chemical composition. These affect specific properties such as surface finish, permeability and refractoriness. In this study, the effect of using CO2 process, permanent mould, cement- bonded sand mould and natural sand mould on the hardness, tensile strength, impact strength and microstructure of cast 6063 Aluminum alloy is examined. The results show that there is significant increase in hardness (33.7BH) and strength (131.23Mpa) of the alloy when natural sand mould is used for its production. Superior impact strength is exhibited in the alloy when permanent mould is used. Keywords: aluminum, casting, mould, mechanical properties, microstructure

2:50 PM

Ignition Characteristics of Aluminum-Nickel Heterostructures Produced by Ultrasonic Powder Consolidation: *Dinc Erdeniz*[1]; David Colanto[1]; Gokce Gulsoy[1]; Teiichi Ando[1]; [1]Northeastern University

Al-Ni heterostructures were consolidated from elemental powders and nanoflakes by the application of ultrasonic vibrations to compacts kept under uniaxial loading. This ultrasonic powder consolidation (UPC) technique can produce powder consolidates at room to warm temperatures within a fraction of a second. Consolidation conditions were studied with Al and Ni powders and nanoflakes. The Al-Ni consolidates produced by UPC may be used as local heat sources in different fields of manufacturing. Therefore, their ignition characteristics and heat outputs were also investigated by differential thermal analysis and continuous-heating ignition tests. Results indicate that ignition is preceded by the solid-state reaction Al + Ni = Al_3Ni which then catalyzes ignition through the formation of eutectic liquid between Al_3Ni and Al.

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Improved Formability of Normalized Cold Rolled Aluminum 1200 and 1230 Alloys: *Samson Adeosun*[1]; Sanmbo Balogun[1]; [1]University of Lagos, Akoka

Annealing of cold rolled aluminium 1xxx alloys at 480°C for 6 hours and stepping down to 420°C for 2 hours produced cups short of the desired height during cupping. Aluminum 1200 and 1230 alloys sheet of 1.2mm thickness have been used for this study. Test samples were subjected to temperatures in the range 400°C – 530°C at 30°C interval with hold time varying from 2 hours to 8 hours and then normalized in natural air. Tensile, cupping and microstructural analyzes were carried out on these samples. In both alloys presence of dendritic structure of Al3Fe, fine crystals of α-aluminum and occurrence of well dispersed fine precipitates of Al12Fe3Si in the matrix caused improved ductility. Good strength with low ear propensity in drawn cups is achieved when AA1200 alloy is normalized at 460°C for 6hrs and AA1230 alloy at 400°C for 6hrs.

3:30 PM

Studies on the Microstructure, Mechanical and High Temperature Wear Behviour of A356 Alloy with Minor Additions of Copper and Magnesium: *Kori Shivaputrappa*[1]; [1]Visvesvaraya Technological University

A356 alloy has wide applications in automotive, marine and other sectors due to its excellent combination of properties such as good fluidity, low coefficient of thermal expansion, high strength-to-weight ratio and good corrosion resistance. In the present study, the effect of copper (Cu) and magnesium (Mg) addition on the microstructure and mechanical behavior of A356 alloy were investigated. Results indicate that, the minor additions of Cu in the range of 0.1-0.5%

improves the mechanical properties. This is due to the partial refinement of a-Al dendrites and solid solution strengthening and precipitation hardening. Also, addition of Mg (0.3-0.7%) results in improvements in strength and hardness at the expense of ductility. This improvement is probably due to the conversion of unmodified acicular silicon to lamellar and possibly even a fibrous structure. Similarly, the worn surface study shows that the change in microstructure leads to improvement in tribological properties of A356 alloy.

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Nanoscratch Behavior of Fine and Ultrafine-Grained Bulk Alumina Fabricated by Spark Plasma Sintering: *Lin Huang*[1]; Yuhong Xiong[1]; Zhihui Zhang[1]; Yonghao Zhao[1]; Wenlong Yao[1]; Amiya Mukherjee[1]; Julie Schoenung[1]; [1]University of California, Davis

Understanding the response of ceramic materials to applied surface scratches is of significant importance for enhancing the finishing fabrication (e.g., grinding and polishing) and the wear resistance of structural ceramic components. Previous studies have indicated that the response is sensitive to grain size. In an effort to systematically evaluate this potential effect, bulk alumina ceramics, with fine and ultrafine-grained microstructures, were fabricated via spark plasma sintering (SPS) and were subjected to variable scratch loads using a Berkovich nanoindenter. The microstructures of residual scratch tracks were characterized using scanning electron microscopy (SEM) and atomic force microscopy (AFM). The effects of different scratch velocities were also investigated. Typical features for both plastic deformation and brittle deformation were observed, and they were further studied using transmission electron microscopy (TEM). Keywords: Alumina; Scratch; Plastic Deformation; Brittle Fracture

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Microstructural Development during Thermo Mechanical Processing of Pipeline Steel: Kawunga Nyirenda[1]; *Hara R. S. Yotam*[1]; [1]The Copperbelt University

Pipeline steels operate under severe conditions such as high pressure and low temperatures and hence require to have high strength and toughness. Good properties can be obtained through understanding of microstructure during thermal mechanic processing. For industrial application, the major changes in microstructure are grain growth and recrystallization. This work looked at recrystallization with a view of determining the upper (T95%) and lower (T5%) limits of recrystallization for an X70 micro-alloyed steel. This was achieved by determining percentage volume of recrystallization and grain aspect ratio.

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Enhanced Performance of Anti-Reflection Functionality by Nano-Sized Structures Fabricated Using Nano-Imprint Lithography: *Kang-Soo Han*[1]; Ju-Hyun Shin[1]; Heon Lee[1]; [1]Korea University

As an effective method of increasing its conversion efficiency, the protective layer of solar cells was patterned with a nano-sized moth-eye structure. This pattern with its conical shape and size serves as an anti-reflective layer. To form a pattern on thermoplastic polymer films and glass plates, the hot-embossing method and nano-imprinting method were used, respectively. Moreover, each substrate patterned on both sides was also fabricated to improve the performance of anti-reflection functionality. Due to the enhanced anti-reflection functionality, the transmittance of patterned films and plates were increased. As a result, a solar cell with patterned polymer films and glass plates as a protective layer exhibited higher quantum efficiency and total conversion efficiency than a solar cell with a bare polymer film and a glass plate as a protective layer.

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From Fantasy to Reality: The Development of Silver Bullet Ammunition: *Kevin Jaansalu*[1]; Michael Briggs[1]; [1]Royal Military College of Canada

The silver bullet is an important part of American folklore. Yet when author Patricia Briggs uses a cast silver bullet in her book "Moon Called", fans cry that there is no such thing. There are two parts to developing silver bullet ammunition: first, casting the bullet, and second, loading and firing the bullet. The substitution of silver for lead in bullet casting requires only minor changes in the casting methods to accommodate the higher casting temperatures and heat load. Two interior ballistic codes were used to model the peak chamber pressure and velocity. Two importance factors for small arms are primer performance and bullet resistance forces in the bore. In spite of the scant literature data, modelling captured the essential performance of the silver bullet.

5:10 PM

Research on Microstructure and Mechanical Properties of Low Alloy Cast Steel with High Strength/Low Yield Ratio by TMCP Technology: Chi Yu[1]; *Haitao Zhou*[1]; [1]Qinhuangdao Branch, Northeastern University

The microstructure of an acicular ferrite/bainite dual-phase steel with high strength/low yield ratio was investigated by TMCP technology, with its mechanical properties tested. The results indicate that bainite can be formed in a wide range of cooling rates from 2°X /s to 25°X /s. The different microstructures are obtained under two kinds of cooling rate of as rolled steel, and bainite ferrite microstructure by accelerated cooling has higher strength than granular bainite microstructure after air cooling. Meanwhile, the yield strength of the steel can be up to 635 MPa through TMCP, the ratio of tensile strength to yield strength is 0.78, and the ferrite is benefit to improve the mechanical properties of low carbon microalloyed steel.

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Microstructure Characterization of Joining Dissimilar Materials: *Oleg Barabash*[1]; Zhili Feng[1]; Mike Miles[2]; [1]Oak Ridge National Laboratory; [2]Brigham Young University

We present the microstructural analysis results of the joints between the steel sheet DP980 and either aluminum alloy AA5754 or magnesium alloy AZ31B. The joining method is based on the principles of the friction spin welding (SW). Lately this method was widely used for joining different plastic details. Fast rotation of the bit is provided with special equipment. During several second the bit is quickly inserted into the metal with low melting temperature (Al or Mg). Then the top of the bit surface is touching the surface of the steel sheet. Friction forces generate extremely fast heating of the touching surfaces up to hypomelting temperatures and form a high perfection joint. Formation of the joint takes place several seconds.Joint structure was examined by means of optical and electron scanning microscopes. The joint between DP980 and the bit contains extended TMAZ and relatively narrow SZ with intense plastic deformation.

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Low Stress Viscous Creep in a Ti3Al2.5V Tubing Under Internal Pressurization: Srikant Gollapudi[1]; Indrajit Charit[2]; *Korukonda Murty*[3]; [1]Defence Metallurgical Research Laboratory; [2]University of Idaho; [3]North Carolina State University

Hoop creep behavior of a thin-walled Ti3Al2.5V tubing studied under closed-end internal pressurization at low stresses of interest to in-service conditions revealed viscous creep behavior. An activation energy close to that for grain boundary diffusion suggested Coble creep mechanism commonly observed in small grain size materials. However, the predicted data were 3 orders of magnitude lower than the experimental strain-rates. The slip bands observed in TEM have a mean spacing of 250 nm and the predictions based on the Spingarn and Nix model of the climb of dislocations at the grain boundaries in the slip bands were noted to be close to the experimental results. This is the first time that the slip band model was found to be operating in the viscous creep regime and further work is needed to find this mechanism in other materials. This work is supported by the National Science Foundation grant #DMR-0412583.

Solid-State Interfaces: Toward an Atomistic-Scale Understanding of Structure, Properties, and Behavior through Theory and Experiment: Diffusion, Radiation Damage, and Interaction with Point Defects

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee

Program Organizers: Michael Demkowicz, Massachusetts Institute of Technology; Douglas Medlin, Sandia National Laboratories; Emmanuelle Marquis, University of Oxford

Tuesday PM Room: 602
February 16, 2010 Location: Washington State Convention Center

Session Chair: Y. Mishin, George Mason University

2:00 PM Invited

Interface Enabled Defects Reduction in Helium Ion Irradiated Cu/V Nanolayers: *Xinghang Zhang*[1]; Engang Fu[1]; Amit Misra[2]; [1]Texas A&M University; [2]Los Alamos National Laboratory

In nuclear reactors,radiation induced void swelling can cause significant dimensional instability of structural materials and degrade their mechanical properties in the form of embrittlement.Nanostructured composites offer unique opportunity to significantly reduce void swelling via effective annihilation of radiation induced opposite type of point defects. Sputter-deposited Cu/V nanolayer films with individual layer thickness,varying from 1 to 200nm were subjected to helium(He)ion irradiation at room temperature. At a peak dose level of 6 displacements per atom (dpa),the average helium bubble density and lattice expansion decrease significantly with decreasing h.The magnitude of radiation hardening decreases with decreasing layer thickness,and becomes negligible when is 2.5 nm or less.This study indicates that nearly immiscible Cu/V interfaces spaced a few nm apart can effectively reduce the concentration of radiation induced point defects. Consequently, Cu/V nanolayers possess enhanced radiation tolerance, i.e. reduction of void swelling and suppression of radiation hardening after He ion irradiation, compared to monolithic Cu or V.

2:30 PM

Effects of Solute and Vacancy Segregation on Antiphase Boundary Migration in Fe_3Al with Stoichiometric and Off-Stoichiometeric Composition: *Yuichiro Koizumi*[1]; Samuel Allen[2]; Masayuki Ouchi[1]; Yoritoshi Minamino[1]; [1]Osaka University; [2]Massachusetts Institute of Technology

Effects of solute and vacancy segregation on migration of 1/2<100> antiphase domain boundaries (APDBs) in Fe_3Al with Al-concentrations of 24-28at% have been studied by a phase-field method in which inhomogeneous vacancy distribution is taken into account. Fe- and Al-atoms segregate to the APDBs where Al-concentration is respectively smaller and larger than 26at%. Boundary mobilities of APDBs were evaluated by measuring the boundary velocity of shrinking circular APDs at 673 K. The Fe- and Al-segregations both decreased the mobility by solute drag. The more the composition deviates from Fe-26at%Al, the more significant the segregation and the decrease in the boundary mobility are. As the APDs shrink, the APDBs break away from the segregation atmospheres and the mobilities increase. After the breakaway, vacancies segregate at APDBs at lower Al-concentration (<26at%) whereas they are depleted at higher Al-concentration (>26at%). As a result, APDBs exhibit lower mobilities in the case of higher Al-concentration.

2:50 PM

A First Principles Study of Hydrogen Trapping at Carbides in Steels: *Sanket Desai*[1]; Neeraj Thirumalai[1]; Peter Gordon[1]; [1]ExxonMobil Research and Engineering

Hydrogen embrittlement of steels is an important problem for the oil and gas industry. Several studies have reported that trapping of hydrogen by strong binding sites in steel can help mitigate hydrogen embrittlement. One type of trapping site suggested by prior studies is the interface between ferrite and carbide (e.g. cementite). Recent experimental studies have indicated that the bulk of the carbides may also trap hydrogen. We examine the atomistic mechanism of hydrogen trapping at carbides through density functional theory. In this talk, we will discuss our findings on hydrogen trapping in the bulk of carbides and share preliminary studies of trapping at the carbide-steel interface.

3:10 PM

Mechanisms of Point Defect Migration in CuNb Interfaces: *Kedarnath Kolluri*[1]; Michael Demkowicz[1]; [1]Massachusetts Institute of Technology

Cu-Nb multilayer nanocomposites exhibit high resistance to radiation damage because Cu-Nb interfaces are strong sinks for radiation-induced defects as well as sites for efficient Frenkel pair recombination. In this presentation, we describe atomistic modeling studies of the mechanisms by which point defects absorbed at Cu-Nb interfaces diffuse. We find that vacancies and interstitials delocalize into jog pairs on Cu-Nb interface misfit dislocations. These defect configurations diffuse by the hopping of individual jogs between misfit dislocation intersections. Defects migrate preferentially along one set of misfit dislocations. The implications of these insights for interface diffusivity and Frenkel pair recombination models are discussed.

3:30 PM

Molecular Dynamics and Molecular Statics Studies of Cascade Damage in Twist Grain Boundaries in Copper: *Xian-Ming Bai*[1]; Richard Hoagland[1]; Michael Nastasi[1]; Arthur Voter[1]; Blas Uberuaga[1]; [1]Los Alamos National Laboratory

Molecular dynamics simulations are used to model the defect production due to cascade damage in sigma 5 twist grain boundaries in copper. We have found that the spatial translations result in several different structures for the twist grain boundary. Molecular statics calculations show that these structures have significantly different defect thermodynamics properties (e.g., vacancy and interstitial formation energies) near or at the grain boundaries. For each grain boundary structure, we have performed multiple molecular dynamics simulations to study the statistically averaged number of defects produced as a function of the original distance between the primary knock-on atom and the grain boundary. The correlation between defect thermodynamics and the produced damage is examined. Comparison of radiation damage between twist and tilt grain boundaries is also performed.

3:50 PM

Radiation Damage and He Solubility at Semi-Coherent Cu/Nb Interfaces: *Dhriti Bhattacharyya*[1]; Michael Demkowicz[2]; Igor Usov[1]; Richard Hoagland[1]; Amit Misra[1]; [1]Los Alamos National Laboratory; [2]Massachusetts Institute of Technology

Thin films of pure Cu, pure Nb and Cu/Nb multilayered nanocomposites were irradiated with He+ ions at 33 keV, with a total dose of 10^{17}/ cm^2. Implanted He depth profiles were then assessed using Nuclear-Reaction Analysis and radiation damage structures imaged using Transmission Electron Microscopy (TEM). The radiation damage was visible as He-filled bubbles and dislocation loops- both in the 1-2 nm size range. In the case of Cu-Nb multilayers with semicoherent interfaces, it was found from TEM studies that the number of radiation induced defects was much lower, and that the critical He concentration at which bubbles are resolved in TEM was ~5-8%, while this was found to be below the detection limit in the case of pure Cu and Nb. This excellent radiation resistance is attributed to the Cu/Nb interfaces, which act as strong sinks for radiation-induced defects.Research funded by DOE, Office of Basic Energy Sciences.

Solid-State Interfaces: Toward an Atomistic-Scale Understanding of Structure, Properties, and Behavior through Theory and Experiment: Microstructural Evolution

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee

Program Organizers: Michael Demkowicz, Massachusetts Institute of Technology; Douglas Medlin, Sandia National Laboratories; Emmanuelle Marquis, University of Oxford

Tuesday PM Room: 602
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Nathan Mara, Los Alamos National Laboratory; Michael Demkowicz, Massachusetts Institute of Technology

4:10 PM Invited

Towards Improved Representations and Maps of the Grain Boundary Character Distribution: *Christopher Schuh*[1]; Srikanth Patala[1]; [1]MIT

This talk will summarize our recent efforts to devise new representations of grain boundary misorientation distributions. First, the topology of the rotation group space of misorientations will be discussed, and the minimum number of variables needed to uniquely color the group space determined. This opens the door to a new, more intuitive type of grain boundary character map, in which the grain boundaries in a micrograph can be colored uniquely by their disorientations. Such maps permit a more direct visualization of connectivity among boundaries of various types. Second, a method will be introduced to describe grain boundary misorientation statistics as a continuous function over the fundamental zone of misorientations. This approach resembles that used in the distribution of grain orientation statistics in texture analysis, and makes the representation of grain boundary character distributions both more rigorous and intuitive.

4:40 PM

Grain Boundary Properties in Three Dimensions: *Anthony Rollett*[1]; Gregory Rohrer[1]; Jia Li[1]; Sukbin Lee[2]; Moneesh Upmanyu[3]; Michael Groeber[4]; Michael Uchic[4]; Robert Suter[1]; [1]Carnegie Mellon University; [2]Purdue University; [3]Colorado School of Mines; [4]Air Force Research Laboratory

Current efforts to characterize grain boundary networks in three dimensions are reviewed for both serial sectioning and synchrotron analysis. Results on boundary populations from stereological analysis are compared with analysis of true 3D networks. Correlations of boundary type at triple lines are discussed, including both misorientations and boundary normals. The boundary networks are also analyzed to extract dihedral angles and calculate grain boundary energy as a function of the five-parameter type. The energy anisotropy is compared to theoretical results, where available. Although the energy is generally inverse to the population, there are non-trivial variations from this rule. The measured microstructures also provide instantiations for simulations of properties. The heterogeneity of plastic deformation provides an example of such a microstructure-property relationship.

5:00 PM

Quantification of Microstructure Variability in Surrogates for Oxide Nuclear Fuels and Its Effects on Local Mechanical Properties: *Karin Rudman*[1]; Pedro Peralta[1]; Chris Stanek[2]; Kirk Wheeler[1]; Manuel Parra[1]; Darrin Byler[2]; Kenneth McClellan[2]; [1]Arizona State University; [2]Los Alamos National Laboratory

Grain boundaries (GBs) play an important role in the retention and release of fission gas in oxide fuels. In general, intragranular bubbles tend to grow near boundaries and then move faster across GBs. As shown by recent atomistic simulations, certain GB structures facilitate the movement and release of the gas, while others retain it. This study aims to experimentally characterize misorientation distributions in samples of depleted Urania and Yttria Stabilized Zirconia mixed with Ceria. The samples are characterized using Electron Backscattering Diffraction (EBSD) to determine grain size, grain orientation, and GB misorientation, and Energy Dispersive Spectroscopy to map composition. Microindentation is used along with EBSD to establish correlations between GB strength and crystallography. The results are used to draw conclusions on the use of these materials as surrogates for mixed oxide (MOX) fuels, and will also inform multiscale simulations of fission gas behavior by providing realistic microstructural information.

5:20 PM

Fundamental Derivation of Phase Field Equations for Microstructural Evolution in Metals with Defects: *Santosh Dubey*[1]; Anter El-Azab[1]; [1]Florida State University

The phase field approach has been extensively used to model mesoscale morphological and microstructural evolution in materials. This approach uses space and time evolution of conserved and non-conserved field variables (order parameters) coupled through the material free energy, to describe nucleation and growth of the microstructure in materials. Most phase field models are phenomenological in scope and they lack a physical justification for using non-conserved order parameters. An attempt has been made to derive the continuum scale kinetic equations for both conserved and non-conserved order parameters from atomistic details using statistical mechanics principles. All model parameters are shown to be linked to the atomistic details of the material. We apply this approach to the problem of void formation in irradiated metals by condensation of vacancies from the matrix, where we also account for the role of interstitials on the processes of voidnucleation and growth.

Surface Engineering for Amorphous-, Nanocrystalline-, and Bio-Materials: Session IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Surface Engineering Committee

Program Organizers: Sandip Harimkar, Oklahoma State University; Arvind Agarwal, Florida International Univesity; Sudipta Seal, University of Central Florida; Narendra Dahotre, University of Tennessee

Tuesday PM Room: 604
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Sandip Harimkar, Oklahoma State University; Arvind Agarwal, Florida International University

2:00 PM Introductory Comments

2:05 PM Invited

Studies on Plasma Surface Nitriding of Interstitial Free Steel: Manoj Debnath[1]; Jyotsna Dutta Majumdar[1]; *Indranil Manna*[1]; [1]Indian Institute of Technology Kharagpur

In the present study, attempts have been made to enhance the surface dependent mechanical (hardness, wear resistance) and electrochemical (corrosion resistance) properties of IF steel by plasma nitriding. Plasma nitriding has been carried out at an applied pressure of 5 mbar using a gas mixture of nitrogen (20 %) and hydrogen (80 %) at 450°C for 1-5 h. The microstructure of the nitrided surface consists of uniformly dispersed globular precipitates of nitrides in the ferrite matrix. The size and volume fraction of the precipitates increases with increase in nitriding time. While γ'-Fe4N is a common micro-constituent, ε-Fe3N and ζ-Fe2N also form at high nitriding time. Similarly, prior deformation (20% - 25%) seems to aid formation of ε-Fe3N, besides γ'-Fe4N. A significant improvement in microhardness and resistance to wear and corrosion has been obtained by plasma nitriding, particularly at 450°C for 5 h after 25% prior cold deformation.

2:25 PM

Nucleation and Growth of Diamond Thin Films Deposited by a CO_2 Laser-Assisted Combustion-Flame Method: *Travis McKindra*[1]; Matthew O'Keefe[1]; [1]Missouri University of Science and Technology

The deposition of diamond thin films using a CO_2 laser-assisted combustion-flame CVD process was investigated by comparing different wavelengths for deposition times of less than 5 minutes. The laser wavelength was varied based on the resonant absorption of laser energy by gaseous precursors (O_2/C_2H_2/C_2H_4), in particular 10.591 µm that did not match a resonant absorption (untuned) and 10.532 µm that matched a C_2H_4 molecule resonant frequency (tuned). The film morphology and microstructure was characterized by scanning

electron microscopy (SEM) and scanning transmission electron microscopy (STEM). After one minute of deposition the films were discontinuous. The amount of faceting and particle size was dependent on the laser wavelength. X-ray diffraction (XRD) and micro-Raman spectroscopy were used for phase identification. The diamond component in the films varied with the deposition time and laser wavelength. The largest diamond component and most faceted grains were obtained using the tuned CO_2 laser regardless of time.

2:45 PM

Stress-Driven Surface Instabilities in Ionic Solids Containing Charged Point Defects: *Steven Henke*[1]; Anter El-Azab[1]; Peter Chung[2]; [1]Florida State University; [2]U.S. Army Research Lab

Oxides exhibit a wide range of functional characteristics that make them suitable for numerous technological applications. In many of these applications, the oxides phases form epitaxial systems with the underlying support structure, giving rise to internal stresses that affect both surface mass rearrangement and bulk point defect distributions. In oxides, however, point defects can be charged, and their concentrations are also influenced by space charge formation and the associated internal electric fields. We present a continuum model for ionic solids containing charged point defects coupled with elasticity and electrostatics. Using a 3D finite element scheme, we demonstrate the effects of these defects on the film's morphological stability and characterize incipient instabilities in terms of model parameters.

3:05 PM

The Effect of Frequency of Microarc Oxidation on Surface Properties of 7075 Aluminum Alloys: *Serkan Bozkus*[1]; Murat Baydogan[1]; Huseyin Cimenoglu[1]; Eyup Kayali[1]; [1]Istanbul Technical University

In this study, thick ceramic coatings were fabricated by microarc oxidation on 7075 aluminum alloy in a KOH, Na2SiO3 solution. Micro arc oxidation was performed by using an AC power supply operating in variable frequency between 100 Hz - 167 Hz. Mechanical and physical properties were examined on the surface of the oxide film including hardness, wear resistance and surface roughness. Rockwell C testing was used to compare the relative adhesion characteristics of the oxide film. The unlubricated tribological performance of the coatings was examined using wear system with recipirocating motion against sintered Al2O3 ball. The surface morphology and structure was examined by SEM and X-ray diffractometer. The studied properties of the samples as a function of frequency were discussed.

3:25 PM

Study of the Nanocomposites for Superalloy Thermal Barrier Coatings: *Shiqiang Qian*[1]; [1]School of Materials Engineering, Shanghai University of Engineering Science

High temperature alloy can be plated on metal bonding coat layer, by magnetron sputtering or electrophoresis, and thermal barrier layer, by high-speed electric or electrophoresis. We can get nanocomposites on alloy by plating metal bonding coat layer first and then thermal barrier layer. The surface morphology,phase composition and element of these two layers can be observed through OM, XRD, SEM. We placed the K17 high temperature alloy into melting sodium chloride at 900° for 1h then air-cooled for 10 minutes and did 15 cycles hot corrosion test. For metal bonding coat layer, magnetron sputtering is better than electrophoresis while for thermal barrier, electrophoresis better than high-speed electric. We found that it has better anti-hot-corrosion performance for nanocomposites through plating NiCrCoAlY, a metal bonding coat layer, by magnetron sputtering and then YSZ, a thermal barrier layer, by electrophoresis on K17 alloy.

3:45 PM Break

4:00 PM

Laser Assisted Deposition of AgInSe2 Films on Si(100): *Dinesh Pathak*[1]; [1]GNDU, Amritsar, Physics

Laser ablation has attracted special interest for the formation of thin films Compared with other formation technique.A distinctive feature of laser ablation is that it allow high quality and stochiometry of films of even very complex element material. In this presentation laser ablation of AgInSe2 chalcopyrite semiconductor will be discussed in which it is difficult to maintain stochiometry by conventional method. High Quality AgInSe2 (AIS) films were grown on Si(100) substrates by the ultra-high-vacuum pulsed laser deposition technique from the AIS target synthesized from high-purity materials. The X-ray diffraction studies of the films show that films are textured in (112) direction. .Increase in substrate temperature results in more order structure. It is observed that compositional stochiometry is maintained to the more extent by PLD technique than other traditional methods like thermal evaporation. The optical studies of the films show that the optical band gap is about 1.20 eV.

4:20 PM

A Novel High Throw Bright Acid Tin Plating of Printed Circuit Boards: *Xiao Faxin*[1]; SHEN xiaoni[1]; [1]Henan University of Science and Technology of China

A novel acid tin plating process produces a bright deposit with excellent throwing power from a sulfate system. Particular attention is placed on the effects of benzylidene acetone, formaldehyde and OP emulsifier on the tin coating and the appropriate concentration of these additives is 0.6 mg/L, 10 mL/L and 15mL/L, respectively. The bright smooth tin layer may be deposited at 1-4 A/ dm^2 and 20-45°X in this solution. The throwing power can reach 90% and the deepening plating ability of hole with L/d of 5 is 100%, which shows that this process may be used for rack plating applications of printed circuit boards. The XRD results show that the crystal face is mainly assigned to the (112) face on the deposits. The SEM results show that the appearance is uniform and the grain is superfine. It was also found that vanadium pentoxide greatly enhances the stability of the plating solution.

4:40 PM

A High Speed Electroless Copper Plating Process of Printed Circuit Board from EDTA•2Na-Containing Solution: *Xiao Faxin*[1]; Shen Xiaoni[1]; Niu Fei[2]; [1]Henan University of Science and Technology of China; [2]Central South University of China

Electroless copper deposits were plated on printed circuit boards in EDTA•2Na-containing solutions. Particular attention is placed on the effects of a-a'-bipytidine, triethanolamine (TEA)and L-arginine on the copper depositing and the appropriate concentration of these additives is 10 mg/L,1mL/L and 0.1mg/L,respectively. The smooth and uniform copper coating may be deposited in the pH value of 12.5-13.0 at 20-50°. The depositing rate of this solution,which is up to 6.7μm/h, is much faster than that previously reported. The use of TEA as additive greatly promotes the depositing speed of coating. The XRD results show that the crystal face is mainly assigned to the (111) and (220) face on the deposits. The SEM results show that the appearance is uniform and the grain is superfine. The backlight level of coating in hole with diameter of 1mm achieves 10th grade after plating for 15min.

5:00 PM

Influence of Process Parameters for Electroless Plating Nickel Alloy Nanoparticles on Carbon Fibers: *Jia Ma*[1]; Yao Guangchun[1]; Bao Li[1]; Zhang Xiao[1]; Ma Junfei[1]; [1]Northeastern University

Carbon fibers have been coated with nickel alloy nanoparticles by electroless plating process, with sodium hypophosphite as a reducing agent in an alkaline bath. The effects of process parameters such as catalyzation time,coating time and nickel sulphate concentrations were investigated. The morphology, coating thickness, growth rate, particle-size, phases and element content in the coating layer of the nickel-coated carbon fibers were investigated by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction(XRD) and ICP, respectively. It has been confirmed that the growth rate and nickel content increase with an increase of nickel sulphate concentrations and coating time. The catalyzation time for 45min, coating time for 20min, bath temperature of 70°, pH of 9,and nickel sulphate concentration of 40g/L is good to get a continuous and uniform coating of nickle alloy nanoparticles on carbon fiber. The average diameter of the nickel alloy nanoparticles has been estimated as 10 nm.

5:20 PM

Synthesis of Nano Porous CO2 Absorbent for Recycling: *Sachi Kanta Kar*[1]; Payodhar Padhi[2]; [1]Central Tool Room and Training Centre; [2]R&D Centre, Hitech Medical College and Hospital, Bhubaneswar

The absorption of carbon dioxide is a chemical reaction, not a physical one. Carbon dioxide reacts with the sodium hydroxide based absorbent and undergoes a complete chemical change. This change is irreversible; therefore the absorbent cannot be regenerated for reuse. A novel method is used to develop ceramic nano absorbent, which is porous in nature. The material can be reused in cyclic manner. Further it is observed from DSC that the absorbent can withstand high temperature. shows the CO2 absorbing behavior of this

absorbent in various CO2 concentration atmospheres, as measured with a Thermo gravimetric/Differential Thermal Analyzer (TG/DTA). The absorbent's weight increased around 354°C due to CO2 absorption, and the reaction was remarkably accelerated around 736°C.

Sustainable Materials Processing and Production: Measuring Sustainability

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee, TMS: Education Committee

Program Organizers: Christina Meskers, Umicore; Randolph Kirchain, Massachusetts Institute of Technology; Diana A. Lados, Worcester Polytechnic Institute; Markus Reuter, Ausmelt Limited

Tuesday PM Room: 2B
February 16, 2010 Location: Washington State Convention Center

Session Chair: Markus Reuter, Ausmelt Ltd

2:00 PM Introductory Comments

2:05 PM Invited

Metrology Needs in Sustainability and Materials Performance: *Richard Ricker*[1]; [1]NIST

Sustainability is an attractive approach to environmental stewardship because it recognizes the importance of continued profitability in maintaining the environment and advancing society. However, the ability of this approach to actually provide benefit will depend entirely on the predictive capability of the full life cycle impact analysis of alternatives. The ability of these models to predict outcomes with acceptable levels of uncertainty will depend on the data used in these models and the appropriateness of the corresponding measurement methods. This presentation will review the on-going effort at NIST to evaluate sustainability metrology needs and examine the role of measures of materials performance and lifetime predictions in these models.

2:30 PM

Lighweight Materials for the Automotive: Environmental Impact Analysis of the Use of Composites: *Karel Van Acker*[1]; Ignaas Verpoest[1]; Wim Dewulf[1]; Joost Duflou[1]; [1]K.U.Leuven

The automotive is a sector where energy consumption during the use phase prevails over the production and the end-of-life phase. Therefore, a lot of research and innovations at replacing classical steel parts by lighter materials like light metals and polymer composites. While composites are very attractive for the use phase of cars, their introduction suffers from the limited end-of-life options for composite structures. An extensive life cycle analysis for a reference car design was conducted to study the effects of replacement of conventional steel structures by lightweight carbon fibre composite alternatives. The study also takes second order effects in the design of the car into account. The opportunities and treats of the trend towards more intensive use of carbon-fibre composites in car design are identified. A discussion on the potential of other types of composites for the automotive in terms of sustainability considerations is added.

2:55 PM

The Challenge of Allocation in LCA: The Case of Open-Loop Recycling: *Elsa Olivetti*[1]; Anna Nicholson[2]; Jeremy Gregory[1]; Randolph Kirchain[1]; [1]MIT; [2]BIO Intelligence Service

Life cycle assessment (LCA) is used increasingly as a tool to provide quantitative metrics of environmental burden and inform early stage materials selection decisions. One common challenge in LCA stems from allocation of burden when multiple products share some of the same processes. A critical example of this is the partitioning of benefit or burden at product disposition in open-loop recycling scenarios. This work investigates whether the various methods proposed for allocating end-of-life burden lead to different results and explores the trends in allocation method bias for or against particular materials classes. Stylized analyses across a range of materials are presented and allocation methods explored include both economic or value-based as well as mass-based approaches. Results indicate that some methods are more averse to materials with a high ratio between the required secondary and primary production impact while others are more averse to materials with high primary production impact.

3:20 PM

Screening-Level Environmental Burden Assessments for Metals Use in Electronics: A Case Study on the U.S. Printed Wiring Board Industry: *Carl Lam*[1]; Seong-Rin Lim[1]; Oladele Ogunseitan[2]; Julie Schoenung[1]; [1]University of California, Davis; [2]University of California, Irvine

Simple screening-level metric indicator methods have been traditionally used to help identify high environmental burden materials present in electronic product systems for further detailed assessment, development of pollution prevention countermeasures, and general materials selection decision-making with limited environmental information. For this study, metrics for global resource consumption, ecological and human health toxicity (cancer and non-cancer categories) are considered for evaluating the United States printed wiring board (PWB) industry's metals use. Leachable metal quantities are used for the ecological and human health toxicity screening assessments with U.S. EPA's Tool for the Reduction of Chemical and Other Environmental Impacts (TRACI) and U.S. EPA's Risk-Screening Environmental Indicators (RSEI) methodologies. Global resource consumption indicators consider the ratio of PWB metal usage relative to world metal reserve statistics. Sensitivity analyses are performed for variable changes in PWB recycling rate, dynamic reserve quantities and metal leaching ratios.

3:45 PM Break

3:55 PM Invited

Agent Based Modeling of Large-Scale Socio-Technical Metal Networks: *I. Nikolic*[1]; Andrew Bollinger[1]; C. Davis[1]; [1]Delft University of Technology

Metals production and consumption networks are complex Large Scale Socio-Technical Systems, consisting of many technical installations, companies operating them, at a global scale, with large environmental impacts. Understanding their dynamics and behavior requires models that are capable of capturing these multiple dimensions. Modeling in the metals domain has traditionally focused on technical and metallurgical aspects. Agent Based Modeling (ABM), a relatively new technique in the metals domain is a tool from the Complex Adaptive Systems field that allows investigation of the change of metals production and consumption networks structure, as they evolve towards a more sustainable state. This paper presents two examples of ABMs of metals networks that illustrate how the modeling technique can be used and the types of insights it offers.

4:20 PM

Toxicity and Resource Depletion Potentials of Light-Emitting Diodes (LEDs): *Seong-Rin Lim*[1]; Daniel Kang[2]; Oladele Ogunseitan[2]; Julie Schoenung[1]; [1]University of California, Davis; [2]University of California, Irvine

Light-emitting diodes (LEDs) are regarded as environmentally friendly light sources due to their high energy efficiency and nonuse of mercury. However, various environmental consequences should be additionally investigated to validate the true environmental performance of LEDs because LEDs utilize the III-V semiconductors with toxic and rare materials, such as gallium, indium and arsenic. The objective of this study is to evaluate toxicity and resource depletion potentials of a variety of color LEDs. Leachability tests are performed to investigate whether the LEDs meet waste regulations and to analyze heavy metal contents. Toxicity and resource depletion potentials of the LEDs are evaluated by using the toxicity and resource depletion characterization factors from Life Cycle Impact Assessment (LCIA) methodologies. This study can contribute to identifying diverse environmental impacts of LEDs and providing valuable information for design for environment (DfE), green purchase, and policy making.

4:45 PM

The Many Aspects of Measuring Sustainability - An Industry Perspective: *Christina Meskers*[1]; C. Hagelüken[1]; [1]UMICORE Precious Metals Refining

Metals will play a key role in meeting the challenges for the future. The metallurgical industry is an important actor in the transition towards closed product and material life cycles, one of the aspects of a sustainable society. In this role the industry needs to develop "sustainable" production, manufacturing and recycling processes, and to assist material selection. It requires the combination of a holistic, collaborative approach and suitable tools. The former includes looking at the entire life cycle, each stage and the interactions within on

different levels. Furthermore it should include technical, economic, legislative and societal aspects. Suitable tools consider the detail and complexity necessary for analyzing and quantifying phenomena at the macroscopic as well as the microscopic level. Such a transition demands creativity, critical rethinking of existing practices, and possibly radical change or completely new business models. This contribution will explore the practical implications for a materials technology company.

5:10 PM
Substance Flow Analysis of Cobalt in China: *Xiao Caimei*[1]; Zhong Juya[1]; Guo Xueyi[1]; Tian Qinghua[1]; [1]Central South University

The method of Substance Flow Analysis (abbreviated as SFA) provides a helpful tool for the study of the industrial metabolism of a certain metal within a regional level. Cobalt is one of the important strategic commodities of national economic development in China, the resource utilization and recycling of which are important for the sustainable development of China's economic construction. In this work the flows and inventory of cobalt in China, 2008, were traced with the STAF model, and the situations of production, fabrication and manufacturing ,use and waste management in China were introduced. The result revealed that the resource of cobalt in China was so poor that a large amount of cobalt material or cobalt products needed to be imported. Based on the result, several suggestions were proposed in the paper, aiming to contribute important reference information for the industrial metabolism in China.

5:35 PM Concluding Comments

The Vasek Vitek Honorary Symposium on Crystal Defects, Computational Materials Science and Applications: Mechanical Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Mo Li, Georgia Institute of Tech; David Srolovitz, Institute for High Performance Computing, Agency for Science, Technology and Research, Singapore; Adrian Sutton, Imperial College London; Vaclav Paidar, Institute of Physics AS CR vvi; Jeff De Hosson, University of Groningen

Tuesday PM Room: 603
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Takeshi Egami, University of Tennessee; Shao-Ping Chen, Los Alamos National Laboratory

2:00 PM Invited
Rethinking Continuum Plasticity Theory for Crystalline Solids: *John Bassani*[1]; [1]University of Pennsylavnia

Constitutive equations for plastic flow are generally assumed to be associative in the sense that the flow potential is taken to be the yield function. G. I. Taylor recognized as early as 1926 that BCC iron and brass behave quite differently than FCC aluminum and copper. Ample experimental evidence now exists, but not until atomistic simulations became sufficiently refined have we been in a position to rigorously address issues of plastic flow in crystalline solids. Professor Vitek pioneered atomistic simulations of dislocation core structures and their mobility that has led to new understanding. In this lecture, issues that arise at various scales are discussed. Atomistic simulations are used to construct multislip continuum models. Using homogenization models, the effective behavior of polycrystals is found to generally be of the non-associated flow type. The latter is shown to significantly affect macroscopic deformations and failure mechanisms including cavitation instabilities, sheet necking, and fracture.

2:25 PM Invited
A Comparison of Coulombic and Plastic Shear Faults in Ice: Narayana Golding[1]; *Erland Schulson*[1]; Carl Renshaw[1]; [1]Dartmouth College

New experiments at -10 °C on both equiaxed and columnar-grained polycrystals have established unambiguously that ice exhibits two kinds of compressive shear fault. One kind, termed a Coulombic (C) or frictional fault, is oriented at ~ 30° to the direction of shortening and is comprised of a narrow band of microcracks that nucleate prior to terminal failure and then link up to create the fault. The other, termed a plastic (P) or non-frictional fault, is oriented at ~ 45° (i.e. sub-parallel to planes of maximum shear stress) and is comprised of a band of re-crystallized grains. P-faults form once the degree of triaxial confinement is sufficient to suppress frictional sliding. Both C and P faulting are accompanied by localized heating. These processes appear to operate in rocks and minerals as well, and possibly in ceramics. Their nature will be discussed, and their role in ice-structure interactions will be noted.

2:50 PM Invited
Kinetics of Martensitic Phase Transformation: Molecular Dynamics of Martenstic Phase Transitions: *Graeme Ackland*[1]; Oliver Kastner[1]; [1]University of Edinburgh

In martensitic phase transformations a crystal phase changes to lower symmetry, typically on cooling. These transitions underlie mechanical behaviour including superelasticity, transformation induced plasticity and shape memory. "Phenomenological theory" relates two crystal structures geometrically, and determines the interfacial "habit" plane while energy minimisation is used to predict microstructures. These approaches are contradictory: the microstructure is determined at the transformation boundary, and cannot "know" whether it will be a global minimiser. Neither theory considers where the atoms go. We introduce interaction potentials which reproduce a martensitic group-subgroup transformation without unphysical metastable states (most previous studies have not). We then model the phase transformation process in 2D and 3D, showing that the "habit" plane is not sharp, and pre-transformation rotations both eliminate the angular momentum problem and trigger the twinning process. Finally, we investigate cycling between two phases, showing how the defect microstructure nucleates subsequent transformation, allowing a reverse shape-memory effect.

3:15 PM
Atomistic Simulations of Hydride Formation and Fracture at the Crack Tip: *Jun Song*[1]; William Curtin[1]; [1]Brown University

A hydrogen atmosphere is adversely present in many applications involving metals (e.g., Ni). Specially, hydrogen can achieve high concentration at the crack tip. The local crowding of hydrogen at the crack tip may lead to continuous hydride development and consequently greatly influence the fracture behavior. A Coupled Atomistic/Discrete-Dislocation (CADD) multiscale model is then used to explicitly investigate the development of hydride and subsequent fracture process at the crack tip. We clarify both the chemical and mechanical driving forces that lead to the hydride formation. The resultant hydride inhibits the dislocation emission from the crack tip as well as prevents the incoming of dislocations towards the crack tip. Consequently the crack tip is restricted from plastic blunting and brittle cleavage may occur.

3:30 PM
First Principles Calculations of Uranium and Uranium-Zirconium Alloys: *Benjamin Good*[1]; Benjamin Beeler[1]; Chaitanya Deo[1]; Sergey Rashkeev[2]; Maria Okuniewski[2]; Mike Baskes[3]; [1]Georgia Tech; [2]Idaho National Lab; [3]Los Alamos National Lab

Uranium zirconium (U-Zr) alloys, when used as the fuel matrix in sodium cooled fast nuclear reactors, have a body centered cubic structure that shows a miscibility gap at the temperature of operation. The alloys exhibit a variation in composition under operation with zirconium atoms migrating against the temperature gradient. We examine several systems of U and U-Zr alloys within a density functional theory framework utilizing the Vienna Ab-initio Simulation Package (VASP). We implement a methodology that applies two separate generalized gradient approximations of the exchange-correlation and compare results obtained from the two pseudopotentials. Bulk properties analyzed include the elastic modulus, lattice constant, and the Birch-Murnaghan equation of state for the α-U, γ-U, and γ-UZr phases. Defect parameters calculated include formation and migration energies of vacancy, interstitial, and substitutional defects for the γ-U and γ-UZr phases. These values are compared to computational and experimental results documented in the literature.

3:45 PM Break

4:05 PM Invited

Application of Vitek's Relationship between the Plastic Dissipation and Work Expended on Brittle Decohesion to the Understanding of Hydrogen-Induced Intergranular Cracking: Paul Novak[1]; Rong Yuan[2]; Moshen Dadfarnia[1]; Brian Somerday[3]; *Petros Sofronis*[1]; Robert Ritchie[2]; [1]University of Illinois; [2]University of California-Berkeley; [3]Sandia National Laboratories

Development of a lifetime prediction methodology for failure of materials used for hydrogen containment components is of paramount importance to the planned hydrogen economy. Arguably the most devastating mode of hydrogen-induced degradation is the hydrogen embrittlement of high-strength steels. We present an approach to quantify the effect of hydrogen on the fracture toughness of a low alloy martensitic steel through the use of a statistically-based micromechanical model for the critical local fracture event—the nucleation of a microcrack at a carbide/ferrite interface. We use Vitek's analysis of the relationship between the plastic work accompanying brittle decohesion to calculate the effect of hydrogen on the strength of a carbide/matrix interface. The result is a quantitative description of the interfacial strength in terms of the carbide size and the local hydrogen concentration. The model is used to predict the macroscopic fracture strength as a function of the nominal concentration of hydrogen.

4:30 PM Invited

Breakdown of Relationship between Chemical Bonding and Deformation Behavior in Crystalline Materials: *Peter Panfilov*[1]; Yuri Gornostyrev[2]; A. R. Kuznetsov[2]; [1]Ural State University; [2]Institute of Metalphysics of the Ural Branch of RAS and CJSC Institute of Quantum Materials Science

According well known paradigm the directed interatomic bonds cause brittleness and poor plasticity, while metallic type of bonds automatically means high plasticity and rupture. This statement is true for majority of crystalline solids, where bonding energy and shear resistance are isotropic and intrinsically coupled. However, there are some cases that do not obey this rule. Refractory FCC-metal iridium exhibits both high plasticity and transgranular cleavage. On the other hand, covalent crystals of titanium dichalcogenides (TiX2, X=S, Se, Te) demonstrate brittle fracture together with dislocation plasticity, too. Using complex atomistic approach based on first principle modeling of deformation and fracture mechanisms we examine this paradox behavior. This work is supported by the U.S. Civilian Research and Development Foundation (CRDF) (# RUXO-005-EK-06/BG7305) and the Ministry of Education and Science of the Russia (# 2.2.2.2/5579).

4:55 PM

Transitions of Dislocation Glide to Twinning and Shear Transformation in Shock-Deformed Tantalum: *Luke Hsiung*[1]; Geoffrey Campbell[1]; James McNaney[1]; [1]Lawrence Livermore National Laboratory

Shock-induced twinning and alpha (bcc) --> omega (hexagonal) phase transition in polycrystal and single-crystal tantalum, which exhibits no clear solid-state phase transformation under hydrostatic pressure conditions, have been observed. Since the domains of deformation twin and omega phase were frequently found in regions containing evenly distributed high-density dislocations without cellular dislocation structures, we suggest that the shock-induced twinning and shear transformation occur as alternative deformation mechanisms to accommodate insufficient dislocation flow resulting from the exhaustion of dislocation multiplication when dynamic recovery processes become largely suppressed under dynamic-pressure conditions. A novel mechanism is proposed to rationalize the transitions of dislocation glide to twinning and shear transformation in shock-deformed tantalum. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DEAC5207NA27344.

5:10 PM

Shock Induced Deformation Substructures and Damage in a Copper Bicrystal: *Ellen Cerreta*[1]; Fang Cao[1]; Irene Beyerlein[1]; Frank Addessio[1]; Carl Trujillo[1]; George Gray[1]; [1]Los Alamos National Laboratory

Controlled shock recovery have been conducted to determine the role of shock pressure and crystal orientation on the substructure and damage evolution of a [100]/[01-1] copper bicrystal. Optical metallography, electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) were utilized to quantify damage and characterize orientation variation and substructure evolution of post-shock specimens. Dislocation cell structures were displayed in both grains and the average cell size decreases with increasing shock pressure. Twinning has been observed in [100] grain. The stress and directional dependence of twinning is analyzed in considering the energetically favorable dissociation of dislocations into Shockley partials and the stress-orientation effect on the partial width. The calculated propensities for twinning in the [100] and [01-1] crystals are in good agreement with the experimental observations. Differences in dislocation cell development and twinning based on crystal orientation are linked to observed differences in the damage evolution of incipiently spalled specimens.

5:25 PM

Dislocation Dynamics, Static Shocks and Size Effects: *Amine Benzerga*[1]; P. J. Guruprasad[1]; [1]Texas A&M University

The plasticity of single crystals of varying size, orientation and initial dislocation content is investigated under compression using discrete dislocation dynamics. Coupling with finite element methods enables rigorous boundary-value problem solutions to be obtained while the physics of dislocation interactions is enhanced through additional rules for dislocation junctions and dynamic sources. By monitoring the evolution of Nye's tensor, maps of geometrically necessary dislocations (GNDs) are generated at desired resolutions and strain levels. The GND contours are thoroughly analyzed for specific pattern recognition with emphasis on potential formation of singularities and ``static shocks''. The emergence of these patterns is discussed in light of predicted size effects on plastic flow under nominally uniform compressive loading. The analyses provide new insight into the scaling of flow stress with specimen size and also highlight the connection between individual dislocationmechanisms, collective phenomena and overall behavior.

5:40 PM

Computer Simulation of the Peierls Stress, Dislocation Dynamics and Dislocation-Loop Interaction in Alpha-Zirconium: Hassan Khater[1]; *Anna Serra*[1]; David Bacon[2]; [1]Technical University of Catalonia; [2]The University of Liverpool

Dislocations with Burgers vector b = 1/3<11-20> glide preferentially on the first-order {1-100} prism planes in h.c.p. zirconium, yet most of the interatomic potentials developed for this metal result in a preference for slip on the (0001) basal plane. This arises from dissociation of the dislocation core on the (0001) plane due to the relatively low value of the intrinsic basal stacking fault energy. A more recent potential based on ab initio data reverses the order of the {1-100} and (0001) fault energies and is used here to investigate the gamma surface, core structure and Peierls stress for glide of edge and screw dislocations of the two slip systems. Their glide velocity has also been computed as a function of applied shear stress and temperature. The interaction of interstitial dislocation loops with 1/3<11-20>{1-100} edge and screw dislocations has been simulated in order to assess their obstacle strength and reaction mechanisms.

5:55 PM

Multiscale Modeling of Thin Films: Linking Dislocation Dynamics with Macroscopic Mechanical Behavior: *Ray Fertig*[1]; Shefford Baker[2]; [1]Firehole Technologies; [2]Cornell University

The difficulty in linking macroscopic mechanical behavior in thin films with dislocation-level behavior has hampered multiscale modeling efforts in thin films for many years. Previous research has suggested that knowledge of particular dislocation interactions cannot be readily translated into knowledge of film strength. But in this work, we present a method to unite dislocation dynamics with macroscopic mechanical behavior of thin films. We use dislocation dynamics simulations to statistically characterize the relationship between stress evolution and the behavior of dislocations in films, including specific interactions and interaction strengths. Our novel method applies the knowledge obtained from the simulations to predict not only macroscopic mechanical behavior, but also the types of dislocation interactions that occur, as well as the distribution of stresses.

6:10 PM

Understanding Some of the Microstructural Reasons for the Small-Scale Mechanical Behavior of Directionally Solidified Mo Micropillars: *E. P. George*[1]; R. I. Barabash[1]; H. Bei[2]; G. E. Ice[2]; [1]Oak Ridge National Laboratory and University of Tennessee, Knoxville; [2]Oak Ridge National Laboratory

When specimen dimensions approach characteristic microstructural length scales (e.g., mean dislocation spacing), interesting mechanical behavior (e.g., "smaller is stronger") is expected and, indeed, found to occur at sizes

of a few tenths to tens of micrometers. Here we discuss how 3D spatially-resolved x-ray microdiffraction can be used to understand the stress-strain response of monocrystalline, directionally-solidified, Mo micropillars. Elastic strain gradients and dislocation densities were measured both in the NiAl-Mo composite containing embedded fibers as well as after the matrix was etched to expose Mo micropillars. The results are discussed in terms of the previously observed mechanical behavior of pillars given different amounts of pre-strain before microcompression. Research sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy. XRD was performed on beamline ID-34-E at the Advanced Photon Source supported by the Office of Basic Energy Sciences, U.S. Department of Energy.

Three-Dimensional Materials Science VI: 3D Representative Volume Elements and Simulated Microstructures

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, ASM-MSCTS: Texture and Anisotropy Committee, TMS/ASM: Phase Transformations Committee

Program Organizers: Alexis Lewis, Naval Research Laboratory; Anthony Rollett, Carnegie Mellon University; David Rowenhorst, Naval Research Lab; Jeff Simmons, AFRL; Stuart Wright, EDAX Inc-TSL

Tuesday PM Room: 401
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Alexis Lewis, U S Naval Research Laboratory; Somanth Ghosh, Ohio State University

2:00 PM Invited
The Role of Representative Volume Elements on Homogenized Material Properties for Heterogeneous Solids: *Somnath Ghosh*[1]; [1]The Ohio State University

The representative volume element or RVE of material microstructure plays an important role in the analysis of heterogeneous materials like metals and composites. Effective material properties like stiffness and strength depend not only on constituent properties but also on the local microstructural morphology. This is compounded with the evolutionary state of the microstructure for problems involving plasticity and damage. The effect of local morphology is pronounced on failure properties such as fracture toughness and ductility. In this work, a combination of statistical and computational tools will be discussed for identifying the statistically equivalent RVE or SERVE for materials with non-uniform dispersions. Both undamaged microstructures with continuous interfaces and damaging solids will be considered. The final part of this presentation will deal with models for ductility in metallic materials e.g. cast aluminum alloys. The development of a homogenized non-local model of plasticity-damage for ductility from an identified SERVE will be discussed.

2:30 PM
Dual Grid Approach for Meshing 3D Images: Stephen Sintay[1]; *Anthony Rollett*[1]; [1]Carnegie Mellon University

Utilizing 3D microstructure models as a method of studying microstructure-properties-performance relationships requires representative models of the polycrystalline microstructure. This work focuses on processes for generating statistically representative implicit microstructure models of polycrystalline materials, and extracting explicit geometries from implicit microstructure data. A novel method is presented that uses the centers of mass of linear portions of the dual grid and the partial entity structure boundary representation to explicitly define the interface geometry of the non-manifold, multiple-region microstructure data. The dual grid center of mass method provides a well-defined set of rules that enables a unique smoothed interface to be obtained. An example of statistical microstructure generation for aluminum alloy AA7075-T651 is given, where the distribution of the synthetic microstructure features are well matched EBSD observations. The synthetic aluminum alloy can then be used for physics-based modeling of microstructurally small fatigue cracks, for example.

2:50 PM
High-Fidelity Hexahedral Mesh Generation for Large 3D Material Microstructures: *Andrew Geltmacher*[1]; Jin Qian[2]; Wenyan Wang[2]; Yongjie Zhang[2]; Alexis Lewis[1]; Siddiq Qidwai[3]; [1]Naval Research Laboratory; [2]Carnegie Mellon University; [3]SAIC

Improved finite element mesh generation algorithms and tools required for high-fidelity representations (correct topology and accurate geometry) of complex, space-filling specimen geometries in 3D are needed for accurate computational simulations and predictions. Novel octree-based isocontouring methods with pillowing are used to generate non-manifold hexahedral meshes of material microstructure. These meshing tools are used to produce conforming 3D meshes with good mesh quality while controlling local mesh density. Thus, computational efficiency can be increased by producing more accurate 3D geometric interfaces while reducing the total number of elements by varying the mesh density in regions of high gradients. Several different meshes will be evaluated for computational speed and accuracy.

3:10 PM Break

3:30 PM Invited
Evaluation and Generation of Representative Volume Elements - A Characterization and Modeling Based Approach: *Stephen Niezgoda*[1]; David Turner[1]; David Fullwood[2]; Surya Kalidindi[1]; [1]Drexel University; [2]Brigham Young University

With the advancement of three-dimensional characterization, researchers are building a vast library of digital microstructure maps. A rigorous yet functional representative volume element (RVE) or statistical volume element (SVE) ensemble concept is central to building knowledge (i.e. structure-property-processing relationships) from the vast amount of raw data. Traditional stochastic mechanics RVE approaches are of limited use to the characterization community in determining the appropriate size and number of regions to examine and often lead to RVEs or SVE ensembles that are too large for computationally expensive numerical models such as crystal plasticity FEM. To this end we describe a framework, based on microstructural n¬-point correlations, to describe the RVE as an optimized minimum set of selected realizations that accurately capture a wide range of effective properties and microstructure metrics of interest. Case studies including 3D polycrystalline microstructures and natural materials such as wood and bone will be presented.

4:00 PM
Simulations of Realistic Three-Dimensional Multi-Phase Microstructures: *Arun Gokhale*[1]; Harpreet Singh[1]; Yuxiong Mao[1]; [1]Georgia Institute of Technology

Material microstructures are three-dimensional, stochastic, and they often contain three or more phases (multi-phase). Microstructural features have complex shapes/morphologies, their spatial arrangements are not necessarily uniform-random, and their morphological orientations are frequently partially anisotropic. Current methodologies for microstructure simulations are mostly restricted to single phase or two-phase microstructures, they involve idealized simple particle/feature shapes; uniform-random spatial distribution of microstructural features; and isotropic feature orientations. In this contribution, we present a methodology that enables simulations of realistic three-dimensional multi-phase microstructures where feature shapes/morphologies, spatial arrangement, and feature orientations are statistically similar to those in the corresponding real microstructures. The simulation parameters used for generation of these microstructures can be correlated to the process parameters. The correlations permit generation of a set of "virtual" microstructures that cover a wide range of process conditions. The virtual microstructures can be then implemented in computational models of materials behavior.

4:20 PM
Synthetic Microstructure Builders for Rare Events: *Michael Groeber*[1]; Jeff Simmons[1]; Mary Comer[2]; [1]AFRL; [2]Purdue

As the tools for generating synthetic instantiations of microstructure have matured, little emphasis has been paid towards the study of rare events. Microstructure builders to date have been focused on incorporating more and more statistical descriptors that are formed by estimating mean values from the data. This provides valuable inputs for homogeneous property simulations, but is inadequate for flaw-sensitive properties like fracture and fatigue, which are dominated by local behavior. This work will examine the modifications of crystal builders needed to account for large deviations from the mean. Some

recent results where limited experimental data are extrapolated to give extreme or other large microstructural deviations suitable for developing a rare event crystal builder will be given.

4:40 PM Invited
Distinguishing Statistical and Representative Volume Elements in Structure-Property Simulations: *David McDowell*[1]; [1]Georgia Institute of Technology

Modeling metal plasticity associated with dislocation processes occurring over a broad range of length and time scales is among the most challenging scientific problems due to the long range fields in crystalline metals and complexity of many body interactions. This talk will provide a perspective regarding an assessment of the current state-of-the-art, nuances of the role of heterogeneity in modeling microstructure evolution, the development of hierarchical and concurrent multiscale models, and modeling concepts to address the role of grain boundaries in polycrystals. Particular attention is devoted to issues related to computing at the scale of a representative volume element (RVE) size for evolving microstructure that meets requirements of statistical homogeneity and has relevance to problems of interest involving field gradients. These conflicting demands promote the use of multiple realizations of statistical volume elements (SVE) to inform statistical models for evolving microstructure.

Ultrafine Grained Materials – Sixth International Symposium: Characterization and Computational Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS: Shaping and Forming Committee
Program Organizers: Suveen Mathaudhu, U.S. Army Research Laboratory; Mathias Goeken, University Erlangen--Nürnberg; Terence Langdon, University of Southern California; Terry Lowe, Manhattan Scientifics, Inc.; S. Semiatin, Air Force Research Laboratory; Nobuhiro Tsuji, Kyoto University; Yonghao Zhao, University of California - Davis; Yuntian Zhu, North Carolina State University

Tuesday PM Room: 607
February 16, 2010 Location: Washington State Convention Center

Session Chairs: Yonghao Zhao, University of California - Davis; Scott Mao, University of Pittsburgh; Mahmoud Nili Ahmadabadi, University of Tehran; Malgorzata Lewandoska, Warsaw University of Technology

2:00 PM Invited
Atomistic Simulations of Defect and Microstructure Evolution in Irradiated Nanocrystalline Materials: *Paul Millett*[1]; Yongfeng Zhang[1]; Michael Tonks[1]; Dieter Wolf[1]; [1]Idaho National Laboratory

One potential application of ultra-fine grained materials is in nuclear energy, in which the abundance of grain boundary (GB) sinks may enhance irradiation tolerance. In this work, molecular dynamics (MD) simulations are used to characterize the emission, diffusion, and annihilation of point defects in nanocrystalline materials to achieve a mechanistic understanding of how these processes evolve under the conditions of high stress, temperature, and irradiation. In particular, we will present results focusing on how GB properties such as structure, diffusion, and migration are affected by on-going point defect nucleation and/or annihilation, and whether GBs sink strengths are infinite or eventually saturate. Finally, deformation behavior including diffusion creep and dislocation plasticity will be analyzed in the presence of irradiation-induced defect structures.

2:20 PM
EBSD Characterization of ECAE Deformed Nb Single Crystals: *Liang Zhu*[1]; Hugo R.Z. Sandim[2]; Marc Seefeldt[1]; Bert Verlinden[1]; [1]K.U.Leuven; [2]University of Sao Paulo

The evolution of microstructure and local crystallography has been examined in three cylindrical Nb single crystals deformed at room temperature by equal channel angular extrusion (ECAE) to a strain of e=1.15. Characterized by orientation imaging microscopy (OIM) and X-ray diffraction (XRD), the microstructures show a large influence of initial crystallographic orientation on grain subdivision. In crystal I (ID//[11-1], TD//[101]), only a small amount of high angle grain boundaries were formed while crystal II (ID//[-111], TD//[-2-1-1]) displays uniformly spaced coarse parallel sets of large misorientation bands inside the matrix. Crystal III (ID//[-1-1-1], TD//[0-11]) has a large amount of curly structures with curved boundary in transverse section and straight one in longitudinal section. Based on the pole figures measured by OIM, the texture evolution is mainly characterized by the lattice rotation around the transverse direction. The correlations between slip behavior and the initial crystallographic orientation are analyzed.

2:35 PM
Microstructure Evolution through Heavy Compression Aided by Thermodynamic Calculations: Farideh Hajiakbari[1]; *Mahmoud Nili-Ahmadabadi*[1]; Behrang Poorganji[2]; Tadashi Furuhara[2]; [1]Tehran University; [2]Tohoku University

Sever Plastic Deformation techniques such as simple heavy compression is one of the most promising techniques which can produce Ultra Fine Grain (UFG) materials. As a result, the UFG materials exhibited superior mechanical properties, such as high strength and toughness, compared with as-received samples. In this paper, the microstructural evolution of heavy deformed a dual phase bainitic-ferrite steel (Fe-0.22%C-2.03%Si-3%Mn) was estimated by thermodynamic computations. Compression tests were conducted at temperatures of 298K and 573K on wedge and rectangle samples at strain rate of 0.001s-1. The specimens were deformed to 40% and 70% of their original thickness. EBSD, SEM and X-Ray diffraction analysis, before and after compression process were performed to verify the thermodynamic calculations. It was found that at 70% compressed samples, at both temperatures; the austenite transformed to the martensite. Additionally, heavy compression resulted in formation of fine grain with high angle grain boundary which confirms grain refinement.

2:50 PM
Prediction of the Stress-Strain Response of the Ultrafine-Grained Nickel Using Multi-Scale Analysis: *Mihaela Banu*[1]; Mitica Afteni[1]; Alexandru Epureanu[1]; Clement Keller[2]; Eric Hug[3]; Anne-Marie Habraken[2]; Laurent Duchene[2]; [1]University of Galati; [2]Universite de Liege; [3]Universite de Caen

The present paper proposes a multi-scale analysis of the tensile test applied to ultrafine-grained nickel to predict the stress-strain response. The work is divided into (i) nanostructuring of the 3 mm diameter coarse-grained nickel rod to ultrafine-grained nickel using a laboratory-scale SPD process named controlled multidirectional shearing process, (ii) crystallographic analysis of the structure and identification of the crystal plasticity model coefficients, (iii) tensile testing of the ultrafine-grained nickel specimens for obtaining the true stress-strain behavior and (iv) modeling of the tensile test using finite element method coupled with strain gradient and crystal plasticity model. This model uses internal variables the densities of statistically stored dislocations and geometrically necessary dislocations that improves the calculation of the isotropic hardening of the material. The proposed multi-scale analysis gives an accurate prediction of the mechanical behavior of the ultrafine-grained materials that can be further applied to finite element modeling in the microforming processes.

3:05 PM Invited
Fracture Behavior Analysis in Hard-To-Deform Materials during Equal Channel Angular Pressing by the Finite Element Method: *Hyoung Seop Kim*[1]; Seung Chae Yoon[2]; Taek Soo Kim[3]; [1]POSTECH; [2]Hyundai HYSCO; [3]KITECH

Equal channel angular pressing (ECAP) uses specially designed channel dies, which enables ECAP to be repeated to produce bulk UFG materials relatively easily. Even if ECAP is a simple technique with less number of variables than any other metal forming processes, it is not easy to process ECAP without fracture of hard-to-deform metallic material due to the accumulation of damage with repeating processing. In this presentation, we investigated the deformation and fracture behavior of pure magnesium using experimental and numerical methods. The finite element method with different ductile fracture models was employed to simulate plastic deformation and fracture behavior of the workpiece. The theoretical approach used in the paper will be useful for the optimum processing design, especially for hard-to-deform materials. This research was supported by a grant from the Center for Advanced Materials Processing (CAMP) of the 21st Century Frontier R&D Program.

3:25 PM
Micro-Mechanical Modeling of Damage in IF Steel Strengthened by Severe Plastic Deformation: Nisrin Abdel Al[1]; *Amine Benzerga*[1]; [1]Texas A&M University

The mechanical behavior of interstitial-free (IF) steel strengthened by means of severe plastic deformation is investigated in order to link processing conditions, microstructure and fracture locus in stress space. Equal channel angular extrusion (ECAE) was used to obtain three types of ultra-fine grain microstructures by varying extrusion rate and processing temperature. The strain-hardening behavior was investigated for the three UFG materials as well as the as-received material using round smooth bars whereas the damage behavior was studied using tensile round notched bars with varying notch radii. A micromechanical model that accounts for the three stages of damage accumulation, namely micro-void nucleation, growth and coalescence is used to describe the fracture behavior. This model is essential towards quantification of strength versus ductility in advanced high strength steels as affected by processing conditions. Some difficulties associated with the uncertainties associated with determining model parameters are highlightedand possible remediation outlined.

3:40 PM
HRTEM and EELS Study on Aluminum Nitride in Nanostructured Al 5083/B4C Metal Matrix Composites: *Ying Li*[1]; Zhihui Zhang[1]; Rustin Vogt[1]; Wei Liu[1]; Enrique Lavernia[1]; Julie Schoenung[1]; [1]University of California Davis

The presence of aluminum nitride in nanostructured Al 5083/B4C metal matrix composites fabricated by cryomilling process was studied by high resolution transmission electron microscopy (HRTEM), electron energy loss spectroscopy (EELS) and energy dispersive X-ray spectroscopy (EDX) analysis. Three different types of aluminum nitride structure were identified; in the predominant phase the N atoms occupy the tetragonal interstitial position in the Al lattice. The cubic and hexagonal aluminum nitride particles were also identified. The present results suggest that the aluminum nitride phases were frequently accompanied by O and Mg. Moreover, the aluminum nitride structure was noted to preferentially form in close proximity to boron carbide particles, arguably related to the segregation of N, O, and Mg in the same region. The results suggest the evolution of the aluminum nitride phase from intermediate transitional structures that involved N atoms in the Al lattice.

3:55 PM Break

4:10 PM
Synergic Effects of Grain Refinement and Precipitation Strengthening: *Malgorzata Lewandowska*[1]; Krzysztof Kurzydlowski[1]; Romuald Dobosz[1]; [1]Warsaw University of Technology

Grain refinement down to nano-scale range offers a great possibility of grain boundary induced improvement of low temperature strength of a wide selection of metals. However, the strength of nano-sized metals is still well below the theoretical limit of E/30, which implies 2.4 GPa for aluminium. This is due to physical limits in strengthening of metals by grain size refinement alone, due to the inverse Hall-Petch relationship and diversity in grains size. In this situation further increase in mechanical strength of nanostructured metals can be achieved by synergic effects brought about by combination of strengthening mechanisms. In this context the paper describes combined contributions of grain size strengthening and by second phase particles. The results obtained for 7475 aluminium alloy are presented, which show the non-additive character of the two strengthening mechanisms in question. These results are rationalized by Finite Element Model of the plastic deformation of two-phase nano-metals.

4:25 PM
Influence of Specimen Dimensions and Strain Measurement Methods on the Apparent Ductility of Bulk Nanostructured Materials: *Yonghao Zhao*[1]; Troy Topping[1]; Yazhou Guo[2]; Qiuming Wei[2]; Yuntian Zhu[3]; Terence G. Langdon[4]; Enrique Lavernia[1]; [1]University of California-Davis; [2]University of North Carolina-Charlotte; [3]North Carolina State University; [4]University of Southern California

Miniature tensile specimens, having various sizes and geometries, are often used to measure the mechanical properties of bulk nanostructured materials. However, these samples are generally too small for use with conventional extensometers so that the strains are usually calculated from the crosshead displacements. This study uses experimental results and finite element modeling (FEM) to critically evaluate the influence of the specimen dimensions and strain measurement methods on the tensile curves obtained from miniature specimens. Using ultrafine grained and coarse-grained Cu as model materials, the results demonstrate that the values of strain obtained from the crosshead displacement are critically influenced by the specimen dimensions such that the uniform elongation and the post-necking elongation both increase with decreasing gauge length or increasing specimen thickness. The results provide guidance on the optimum procedures for the tensile testing of miniature specimens of both coarse-grained and nanostructured materials.

4:40 PM Invited
Avoiding Cracks and Inhomogeneities in Billets Processed by ECAP: *Paulo Cetlin*[1]; Maria Teresa Aguilar[1]; Roberto Figueiredo[2]; Terence Langdon[2]; [1]Federal University of Minas Gerais; [2]University of Southern California

Equal-channel angular pressing (ECAP) is an established technique for producing bulk ultrafine-grained metallic materials. The present paper discusses the occurrence of strain heterogeneities and fracture in billets of the Pb-Sb alloy processed by ECAP and also in aluminum and magnesium alloys. The Pb-Sb alloys are interesting model materials as they are age-hardenable, display dynamic recrystallization at room temperature and soften as a result of the break-down of the as-cast structure. The present work evaluates different processing variables and includes results of experiments and computer simulations of plastic flow. It is shown that pre-deformation of the as-cast material, the decrease of the ECAP strain per pass through the adoption of dies with angles higher than 90° and the heating of the material/die are effective procedures to avoid inhomogeneities and the cracking of billets during ECAP.

5:00 PM
Multiscale Modeling of Back-Stress Evolution in Equal-Channel Angular Pressing: *Enze Chen*[1]; Laurent Duchêne[2]; Anne-Marie Habraken[2]; Bert Verlinden[1]; [1]Katholieke Universiteit Leuven, Belgium; [2]FNRS Fonds de la Recherche Scientifique, Université de Liège

Fine-grained materials produced by ECAP exhibit kinematic hardening due to the existence of a back-stress. This paper discusses two different-scale models that are able to describe the kinematic hardening behavior during tensile and compression tests. The investigated material is commercial-purity-aluminum AA1050, processed by ECAP route C. A macroscopic phenomenological Teodosiu-type model is studied first. The initial parameters are identified from a series of mechanical tests. The predicted back-stress saturates quickly and remains constant during the process, which doesn't agree with the increasing trend experimental result. For better prediction, a new dislocation-based composite model is introduced. It is based on Yuri Estrin's dislocation model and adopts a back-stress description from Maxime Sauzay's. Coupled with a Full-constraint Taylor model, it can predict the evolution of the dislocation densities, the cell size, especially the back-stress. This model takes into account the microstructure evolution and gives a better prediction compared to the previous one.

5:15 PM
Microstructure of Cu and Cu + Zr Samples after ECAP and HPT Deformation Studied by Different Methods: *Radomir Kuzel*[1]; Zdenek Matej[1]; Milos Janecek[1]; Jakub Cizek[1]; Milan Dopita[2]; [1]Charles University in Prague, Faculty of Mathematics and Physics; [2]Institute of Materials Science, TU Bergakademie Freiberg

X-ray diffraction (XRD), transmission electron microscopy (TEM), electron backscatter diffraction (EBSD) and positron annihilation spectroscopy (PAS) were used for microstructural studies of Cu and Cu + Zr samples deformed by equal-channel angular pressing (ECAP), 1-8 passes, and high-pressure torsion (HPT), 1-20 rotations. In addition to grain refinement with the number of ECAP passes, TEM and EBSD revealed also the evolution of grain boundaries from low-angle to high-angle ones. PAS detected the presence microvoids and enabled determination of their size. Because of high dislocation densities (10^{15} m^{-2}) only XRD could be used for their determination. Both individual diffraction line profile analysis and total diffraction pattern fitting by own newly-developed software revealed changes in densities and dislocation correlations. XRD and EBSD were used for detailed texture measurements also from different sides of ECAP samples. XRD 2D diffraction patterns can easily be used for fast characterization of thermal stability of the microstructure.

5:30 PM
Partial Dislocation Nucleation and Travelling in Nanocrystalline Metals: *Scott Mao*[1]; Zhiwei Shan[2]; [1]University of Pittsburgh; [2]Hysitron Inc

This talk focuses on partial dislocation dynamics from grain boundary and storage in nc materials through in-situ TEM and in-situ synchrotron tests. It is believed that the dynamics of partial dislocation processes during the deformation of nanocrystalline materials can only be visualized by computational simulations. Here we demonstrate that observations of partial dislocation processes during the deformation of nanocrystalline metals using a combination of in situ tensile straining and high-resolution transmission electron microscopy. In situ synchrotron on nc and micron Ni under hydrostatic stress up to 57Gpa show that peak broadening increases during loading up to 45 Gpa in nc-Ni, which indicates high dislocation density storage, and no clear grain growth or texturing.

5:45 PM
Ultra Fine-Grained Structures Formed in Impact Welding of 6061 Aluminum Alloy and 110 Copper Alloy: *Yuan Zhang*[1]; Glenn Daehn[1]; Suresh Babu[1]; [1]The Ohio State University

Magnetic pulse welding is an impact-welding process, similar to explosive welding, that produces a metallurgical bond by the oblique impact of two metal solids at high velocity. The violent impact removes surface layers, brings new surfaces together at high strain rates, and produces high strains at strain rates on the order of 10E6 s-1. The process is very heterogeneous even at very short length scales. Local melted regions may be present, and there is also extreme grain refinement; the original 40µm grain size can be reduced to the order of 50nm near the AA6061 welded interface and the micro-twinning and micro-band structures present on welded Cu101. Further, there is no trace of the original interface and the impact-welded region typically has higher strength than the base metal. This study will correlate the structure as determined by TEM and EBSD to the local impact process.

Ultrafine Grained Materials – Sixth International Symposium: Microstructural Evolution

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS: Shaping and Forming Committee
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Session Chairs: Matthias Goeken, University Erlangen--Nürnberg; Mohammed Haouaoui, Texas A&M University; Joel House, U.S. Air Force Research Laboratory; Indranil Roy, Schlumberger

2:00 PM Invited
Atom Probe Tomography: A New Insight into the Partition and Segregation of Solutes in Ultrafine-Grained and Nanocrystalline Al Alloys: *Gang Sha*[1]; Xiaozhou Liao[1]; Rimma Lapovok[2]; Ruslan Valiev[3]; Terence Langdon[4]; Simon Ringer[1]; [1]The University of Sydney; [2]Monash University; [3]Ufa State Aviation Technical University; [4]University of Southern California

Severe plastic deformation (SPD) is well known to be effective in modifying materials microstructures and achieving significant refinement in grain size. The large volume of grain boundaries in the ultrafine-grained (UFG) or nanocrystalline materials bear important consequences for the partition and segregation of solutes and are correlated with many important properties of the materials. The quantitative characterization of solute partition and segregation microstructures has been a challenge. The recent development of atom probe tomography (APT) has significantly improved our ability to access atomistic quantitative information about the structure and chemistry of materials. In this contribution, we highlight a methodology in the application of APT to gain structural and chemical information of grain boundaries, and we summarise our recent results from UFG and nanocrystalline Al alloys. The effect of processing temperature and grain size on the partition and segregation of solutes will be addressed.

2:20 PM
Deformation Induced Grain Growth in Nanostructured Al-Mg Alloy: *Zhihui Zhang*[1]; Xiaolin Wu[2]; Ying Li[1]; Troy Topping[1]; Yizhang Zhou[1]; Wei Xu[2]; Kenong Xia[2]; Enrique Lavernia[1]; [1]University of California, Davis; [2]University of Melbourne

Deformation induced grain growth in nanostructured materials during thermomechanical processing has been widely reported. However, the governing mechanisms are not well understood. In this study, the microstructural evolution in cryomilled Al-Mg alloy powders (grain size ~25 nm) was studied following equal channel angular pressing (ECAP), and hot extrusion, respectively. For ECAP, the nanostructured powder was directly consolidated at 325°C with a back pressure 50 MPa for 4 passes whereas for extrusion, it was annealed for 24 hours at 400°C (grain size ~100 nm) and then extruded with a ratio 10:1 (grain size ~300 nm). Despite the longer thermal exposure time, the latter sample exhibited a higher yield strength (530 MPa) than the former (470 MPa), indicating the effect of plastic strain on grain growth. The growth process was characterized using TEM and texture measurement. The results showed that grain rotation and coalescence contributed to the observed grain growth.

2:35 PM Invited
Microstructural Evolution during Processing and Deformation of Ultrafine Grain Metals: *Marc Meyers*[1]; Y.-B. Xu[2]; H. J. Yang[2]; B. K. Kad[1]; [1]UCSD; [2]Institute for Metal Research Chinese Academy of Sciences

The processes of ECAP and high-strain, high strain rate deformation were used to reduce the grain size from the polycrystalline to the ultrafine grain regime in FCC metals. The grain size and morphology resulting from the two processes are similar, the result of close values of the Zener-Hollomon parameters. High-strain, high-strain rate plastic deformation was applied to the ufg structure in forced shear bands. It leads to an increase in grain size due to thermally-induced recrystallization. The microstructural evolution is analyzed with quantitative predictions of grain-size refinement during severe plastic deformation.

2:55 PM
Grain Refinement in Magnesium Alloys Processed by ECAP: Roberto Figueiredo[1]; *Terence Langdon*[1]; [1]University of Southern California

Equal-channel angular pressing (ECAP) is an effective tool for producing exceptional grain refinement in bulk polycrystalline f.c.c. metals. However, the situation is more complex in h.c.p. metals, as in magnesium alloys, where successful processing generally requires a preliminary grain refinement through an extrusion step prior to processing by ECAP. This paper examines the characteristics of grain refinement in magnesium alloys and the significance of the production of a bi-modal grain distribution.

3:10 PM Invited
Microstructure Evolutions in Ti and Zr by Pressure-Induced Phase Transformation under HPT-Straining: *Yoshikazu Todaka*[1]; Hiroaki Azuma[1]; Kensyu Irie[1]; Nozomu Adachi[1]; Yuuki Ohnshi[1]; Minoru Umemoto[1]; [1]Toyohashi University of Technology

HPT (high-pressure torsion) process, which is one of severe plastic deformation (SPD) techniques, has an additional unique characteristic: deformation under high pressure. It is well-known that Ti and Zr display three phases as a function of pressure and temperature. At ambient condition these materials stabilize in the hexagonal close-packed α-phase, and under high pressure at 2 GPa (Ti) and 3 GPa (Zr) at room temperature the α-phase transforms into the simple hexagonal ω-phase. The stabilization of ω-phase in Ti and Zr at ambient condition occurred by applying shear deformation in the state of ω-phase under HPT process. In this study, HPT-straining was applied to investigate the effects of pressure and shear deformation on the stabilization of ω-phase in Ti and Zr. The microstructure, thermal stability and mechanical properties were examined.

3:30 PM Invited

New Strategies to Overcome the Limits in Refinement by Severe Plastic Deformation: *Reinhard Pippan*[1]; Anton Hohenwarter[1]; Andrea Bachmaier[1]; [1]Erich Schmid Institute of Materials Science, Austrian Academy of Sciences

The evolution of the microstructure of single-phase materials deformed by SPD shows a relatively uniform behaviour: With increasing strain the size of fragmented crystalline elements decreases until at strains larger than a certain value no further refinement is observed. The minimum grain-size can be affected by deformation temperature, impurities, alloying, the mode of deformation, and strain rate. Even at very low deformation temperatures a grain-size below 30nm cannot be obtained. The aim of the paper is to present different strategies to overcome this limit. The evolution of the microstructure during high pressure torsion (HPT) deformation of different microcomposites and precipitate forming alloys has been investigated. Furthermore, examples for the generation of nanocomposites by means of powder consolidation with HPT will be presented. The possibilities, advantages and disadvantages of the different strategies to overcome the single-phase limit in refinement by SPD by means of a composite like structure will be discussed.

3:50 PM Break

4:05 PM Invited

Strain Path and Microstructure Evolution during Severe Plastic Deformation (SPD) Processing: *Terry McNelley*[1]; Juan Garcia-Infanta[2]; Srinivasan Swaminathan[3]; Alexandre Zhilyaev[4]; Fernando Carreno[2]; Oscar Ruano[2]; [1]Naval Postgraduate School; [2]Centro Nacional de Investigaciones Metalurgicas; [3]GE Global Research; [4]Centro Nacional de Investigaciones Metalurgicas and Institute for Metals Superplasticity Problems

A variety of SPD methods have been developed in order to impart exceptionally large strains and produce highly refined deformation-induced microstructures. SPD methods involving monotonic straining will be reviewed and contrasted with techniques that may result in redundant straining. The former comprise accumulative roll bonding (ARB), large strain extrusion machining (LSEM), equi-channel angular pressing (ECAP) by routes A and BA, uni-directional high-pressure torsion (HPT) and friction stir processing (FSP). The latter include ECAP by routes BC and C, and cyclic straining during HPT. In pure metals it is often difficult to distinguish monotonic and redundant strain paths from examination of the refined grains. However, monotonic and redundant strain paths may be readily distinguished in alloys containing non-deforming constituent particles. Results from several studies will be described and the implications in production of homogeneous and refined microstructures will be summarized.

4:25 PM

Nanoscale Structural Refinement and Deformation Mechanisms in Beta-Type Titanium Alloys: *Mariana Calin*[1]; Wei Xu[2]; Jürgen Thomas[1]; Norbert Mattern[1]; Michael Zehetbauer[3]; Jürgen Eckert[1]; [1]IFW Dresden; [2]University of Melbourne; [3]Universität Wien

In recent years, extensive investigations have been carried out to develop ß-type Ti-based alloys with a low Young's modulus and high strength for orthopedics applications. In this work, the ß phase stability and its effect on the deformation behavior have been investigated for a series of ß Ti-Nb and Ti-Nb-Ta-X (X= Zr, In, Ag, Cr) alloys produced by casting. Microstructural evolution was identified upon compressive loading and high pressure torsion for ß alloys with different phase stability against ß-->a" martensitic transformation. With increasing the degree of deformation, martensitic transformation and deformation twinning are initially operative followed by slip of dislocations for the less stabilized ß Ti alloys, whereas slip is initially operative followed by nanoscale deformation twinning for the relatively stabilized alloys. A pronounced grain refinement of ß grains to the nanoscale (50~100 nm) easily occurred in the In- and Ag-containing alloys with low stacking fault energy.

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Microstructural Evolution during Cryomilling of B4C Reinforced Al Nanocomposite: *Byungmin Ahn*[1]; Yuzheng Zhang[1]; Rustin Vogt[2]; Zhihui Zhang[2]; Julie Schoenung[2]; Enrique Lavernia[2]; Steven Nutt[1]; [1]University of Southern California; [2]University of California, Davis

Al matrix nanocomposite powders reinforced by B4C particulate was fabricated by cryomilling. The powders were cryomilled for different times in order to investigate the microstructural evolution during milling. The microstructure was examined using transmission electron microscopy (TEM), scanning electron microscopy (SEM) and density measurements. Grain size distribution and microvoid content were correlated with powder particle size and shape. Energy dispersive X-ray spectroscopy (EDS) was used to identify intermetallic phases derived from dispersoids and constituent particles. The evolution of oxides, intermetallic phases, microvoids, and grain structure was studied as a function of cryomilling time.

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Recrystallization of Tantalum Processed by Equal Channel Angular Pressing: *Joel House*[1]; John Bingert[2]; Philip Flater[1]; James O'Brien[3]; William Hosford[4]; Robert DeAngelis[5]; Richard Harris[1]; [1]Air Force Research Laboratory; [2]Los Alamos National Laboratories; [3]O'Brien and Associates; [4]University of Michigan; [5]University of Florida

The current study examines the effects of severe plastic deformation of commercially pure tantalum by equal channel angular pressing (ECAP) on recrystallization. Round bars 63 mm diameter of tantalum supplied by H.C. Starck and Cabot Supermetals were processed by eight passes through a 135-degree ECAP die with a 180\176 rotation between each pass and then forged into 6.3 mm thick plates. Coupons, sectioned from the plates, were vacuum annealed for one hour at temperatures ranging from 700\176C to 1250\176C. Hardness measurements, optical micrographs, and diffraction data were gathered from the annealing coupons. The hardness data indicated a 150\176C difference in the softening temperature between the two mill products. The diffraction data indicated that the severe plastic deformation reduced the texture banding in the annealed microstructures, but the final annealed microstructures differed significantly between the two mill products.

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Ultrafine Grain Refinement of Co-29Cr-6Mo Alloys with Considerably Low Stacking Fault Energy during Conventional Hot-Compression Deformation: *Akihiko Chiba*[1]; Kenta Yamanaka[1]; Manami Mori[1]; Shingo Kurosu[1]; Hiroaki Matsumoto[1]; Yunping Li[1]; [1]Tohoku University

To examine the microstructural evolution during hot compression deformation of Co-29Cr-6Mo alloy, hot compression tests have been conducted at deformation temperatures ranging from 1323 to 1473 K at various strain rates of 10^{-3} -10 s^{-1}. The DRX grain size of deformed specimens considerably decreased with increasing Zener-Hollomon parameter at strain rates ranging from 10^{-3} to 0.1 s^{-1} . Ultrafine grained microstructure with the grain size of approximately 0.6 μ m was obtained under deformation at 1323K at 0.1 s^{-1} from initial grain size of 40μ m. The grain refinement to submicrometer scale of Co-Cr-Mo alloys has been achieved with hot deformation by ~60% due to the DRX in which the bulging mechanism is not operative. The ultrafine grains obtained due to the DRX without bulging is closely related to considerably low SFE of the Co-Cr-Mo alloy at deformation temperatures.

5:25 PM Invited

Effect of Interfaces on Microstructural Evolution and Deformation Behavior of Ultrafine Ag-Cu Laminar Nanocomposites: *Nathan Mara*[1]; Dhriti Bhattacharyya[1]; Irene Beyerlein[1]; David Alexander[1]; Carl Necker[1]; [1]Los Alamos National Laboratory

We have observed unusual mechanical behavior during warm rolling of Ag-Cu nanoscale eutectic. Starting with an Ag-Cu alloy with an eutectic lamellar bilayer thickness of 200 nm, the as-cast rods are rolled to 75% to 95% nominal reduction in thickness. Cu (111) X-ray pole figures from the rolled nanocomposite indicate unexpected texture evolution during rolling of pure bulk Cu from that involving dislocation slip alone. A Visco-Plastic Self-Consistent (VPSC) polycrystal model indicates that both silver and copper deformed by slip and twinning, with the twin fraction reaching over 50%, depending on rolling reduction. We hypothesize that the unusual observation of room temperature deformation twinning in Cu is induced by twinning in Ag, aided by high Ag-Cu interfacial content. Microstructural evolution and mechanical behavior of the nanocomposite were evaluated at different strain levels in an effort to determine the effects of the interface on deformation processes at diminishing length scales.