2009 Functional and Structural Nanomaterials: Fabrication, Properties, and Applications: Nanoscale Oxides: Synthesis and Applications

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

Program Organizers: Gregory Thompson, University of Alabama; Amit Misra, Los Alamos National Laboratory; David Stollberg, Georgia Tech Research Institute; Jiyoung Kim, University of Texas at Dallas; Seong Jin Koh, University of Texas at Arlington; Wonbong Choi, Florida International University; Alexander Howard, Air Force Research Laboratory

Tuesday AM   Room: 3020
February 17, 2009   Location: Moscone West Convention Center

Session Chairs: Alexander Howard, Air Force Research Laboratory; Jiyoung Kim, University of Texas at Dallas

8:30 AM
Solution-Processed Nanostructured Zno Electrodes for Photovoltaics: *Rodrigo Noriega-Manez*[1]; Ludwig Goris[1]; Sujay Phadke[1]; Greg Kusinski[2]; Alberto Salleo[1]; [1]Stanford University; [2]Clemson University

Zinc oxide (ZnO) is a strong candidate for replacing indium tin oxide in solar cell electrodes and transparent electronics, provided that a high quality material with optimized properties can be obtained with a low-cost and high-throughput process. With this in mind, aluminum- and gallium-doped ZnO nanowires were synthesized using a low-temperature solution-based process, improving their electrical conductivity without affecting optical transparency. The resulting nanowires were characterized with electron microscopy (SEM, TEM), XRD, and composition-sensitive techniques (AES, XPS, EDS); the electrical properties were observed with a four-terminal probe. The dependence of sheet resistance with temperature and doping was studied. In-plane alignment of the nanowires allowed us to observe the effect of morphology in the film's resistivity, and single-wire measurements provided insight into their electronic characteristics. Early attempts to achieving uniform dopant incorporation are presented, as well as their effect on device performance.

8:45 AM
Anodic Growth of Self-Organized TiO2 Nanotubes in Organic Electrolytes: *Mojtaba Hamedi*[1]; Masood Askari[1]; [1]Sharif University of Technology

Anodization of titanium in a fluorinated ethylene glycol (EG) and dimethyl formamide (DMF) electrolytes is investigated. The prepared anodic film has a highly ordered nanotube-array surface architecture. Compared with water-based electrolytes, in EG and DMF electrolytes much longer and smoother tubes can be grown. Topologies of the anodized titanium change remarkably along with the changing of applied voltages, electrolyte concentration and oxidation time. Increasing anodization potential results in longer nanotube arrays of greater diameter and length of the nanotubes increases with time up to a certain maximum length beyond which it declines.

9:00 AM
Synthesis of Nanostructured Anatase and its Grain Size Effect on Catalytic Properties: *Francisco Robles Hernandez*[1]; Leonardo Gonzalez-Reyes[2]; Isaias Hernández-Pérez[3]; Hector Dorantes Rosales[2]; Elsa Arce Estrada[2]; [1]University of Houston; [2]Instituto Politécnico Nacional; [3]Universidad Autónoma Metropolitana

In the present paper are given the results of sono-synthesis of anatase nanoparticles that was heat treated at different times and temperatures to coarsen and investigate the effect of grain size and purity effects on catalytic properties of anatase. Pure anatase was coarsened from 6.2 nm to 28.3 nm and in a mix of anatase and rutile anatase reached a grain size of 89 nm, while pure rutile reaches a grain size of 232 nm. The coarsening kinetics of anatase and rutile show behaviors similar to those of described by the LSW theory. In this work is proposed an algorithm to predict the surface characteristics of pure anatase based on the X-Ray diffraction results. Results of electro and photo catalysis are presented and are related to the surface characteristics of nanometric anatase. Anatase and/or rutile were characterized by means of: TEM, SEM, XRD, BET, UV-vis, Raman, Infrared, photo- and electro-catalysis.

9:15 AM
ZnO Nanowires Doped with Al and Ga Synthesized by a Low-Temperature Solution-Based Process: *Greg Kusinski*[1]; Pooja Puneet[1]; Rodrigo Noriega-Manez[2]; L Goris[2]; Alberto Salleo[2]; [1]Clemson University; [2]Stanford University

Zn-based oxides, due to the great natural abundance and low toxicity of Zn, are attractive replacement materials for indium tin oxide as transparent electrode in thin-film solar cells. ZnO can be made conductive by doping with group III elements. In this study, a solution-based chemistry was used to synthesize intrinsic, Al-doped and Ga-doped ZnO nanostructures. The nanowires were grown at 300°C in trioctylamine by dissolving zinc acetate. The amount of dopant atoms was modulated by controlling the dopant salt (Aluminum acetate or Gallium nitrate) concentration in the solution to obtain the desired Al:Zn or Ga:Zn ratio. Different doping conditions gave rise to different nanoscale morphologies. The effect of a surfactant (oleic acid) was also investigated. An electron microscopy (TEM, HRTEM, EDS, EELS) study correlating the morphology, aspect ratio, surface roughness and doping of the individual ZnO wires to the electrical properties of the spin coated films is presented.

9:30 AM
Effect of Heating Temperature on Oxidation Growth of ZnO Nanopartical: *Liu Yanhui*[1]; [1]Shanghai University Of Engineering Science

With different temperature(500°, 600°, 700°) in air resistance, Granular ZnO films were grown by Zn nanoparticals, which produced by renovated hybrid induction and laser heating, dispersed by ethanol, deposited on flat fundus of Al2O3 ceramic. Results of SEM show that the morphology of ZnO nanoparticals was transformed from flake into granular by combined action of melt and oxidation of nanometer Zn.

9:45 AM
Sonochemical Synthesis of TiO2 Nanoparticles and the Effect on the Structural and Morphological Evolution under Thermal Treatment and Their Electrochemical Properties: *Leonardo Gonzalez-Reyes*[1]; Isaias Hernandez-Perez[1]; Hector Dorantes-Rosales[1]; Jose de Jesus Rivera[2]; Francisco Carlos Robles-Hernandez[3]; [1]Instituto Politecnico Nacional; [2]UASLP; [3]Transportation Technology Center Incorporated

TiO2 with an average grain size of 6 nm and BET surface area of 300 m2g-1, has been synthesized by Sonochemical method. The structure and particle size were determined by XRD and TEM. The stability of TiO2 was studied in the range of 400 to 900°C. The TEM study elucidates porous spheres. Heat treated was accompanied by a change on the BET from 300m2g-1 to 25 m2g-1. The electrochemical properties shows singularities that could be explain for the complex network of an original sample and quantum size effects. The influence of the size dependent and structure phase on store charge and current density, respectively. In fact, the highest current-charge magnitude is presented in the sample with a heat treatment of 450°C. It means that there is an optimal size, with its structural and morphological properties, to improve electro-reduction process. These analyses has been demonstrated and reported herein.

10:00 AM Break

10:15 AM
Formation of Zirconia Coatings by Laser Ablation: *Maxim Pugachevsky*[1]; Alexander Kuz'menko[2]; Victor Zavodinskiy[1]; Sergey Pyachin[1]; [1]Institute of materials of Khabarovsk scientific centre of Far Eastern Branch of the Russian academy of Sciences; [2]Pacific national university

Zirconia coatings were formed on monocrystalline silicon substrate by laser ablation. Zirconia monoclinic powder alloyed by CO2 laser was used as a source. The pulsed radiation (YAG: Nd3+) was used with pulses up to 1ms and power up to 1 kWatt. The maximal size of particles deposited on the substrate was limited by the special filter. The SEM, TEM, AFM and X-ray analyses have shown that the coating consists of particles of the cubic phase. A probable mechanism for cubic ordering of the ablated ZrO2 is thermal stabilization.

10:30 AM
Tailoring of TiO2 Nanotube Growth by Crystallographic Orientation of Ti Substrate: *Grant Crawford*[1]; Nikhilesh Chawla[1]; [1]Arizona State University

Titanium oxide coatings exhibit desirable properties as photocatalysts for hydrogen generation, dye sensitized solar cells, chemical sensors, and bioactive coatings on titanium implants. In this talk we report on the relationship between the mechanistic aspects of nanotube growth as controlled by the orientation of the Ti substrate. Crystallographic orientation of the Ti grains was determined using electron backscatter diffraction (EBSD). TiO2 nanotubes were fabricated via anodic oxidation of Ti. Characterization of the as-processed TiO2 nanotubes was conducted using scanning electron microscopy. We will show that nanotube growth can be controlled by the crystallographic orientation of the Ti substrate. This finding presents a novel and exciting new approach for tailoring the rate of nanotube formation by simple texturing of the Ti surface.

10:45 AM
Fabrication and Characterization of Single TiO2 Nanotube for Chemical and Bio Sensor Applications: *Mingun Lee*[1]; Dongkyu Cha[1]; Hyunjung Shin[2]; M.J. Kim[1]; Jiyoung Kim[1]; [1]University of Texas at Dallas; [2]Kookmin University

Focus has been placed on TiO_2 nanotubes as a material for emerging applications particularly chemical and biological sensors.It is nontoxic and provides not only a large surface to volume ratio but also an open capped hollow structure. The nanotubes also exhibited n-type semiconductor behavior and show electrical conductance modulation under different environments. This implies the feasibility of using TiO_2 nanotubes for chemical sensors. Additionally, we explore their applications as biomaterial detectors. The surface of the nanotubes was functionalized by SAM (Self Assembly Monolayer) compounds with various terminal functional groups, such as carboxylic and alkosyl groups, to detect other species selectively. In this study, we will present the viability of functionalized TiO_2 nanotubes for bio-sensors with selective detection. This research was supported by a grant (code #: M105KO010026-05K1501-02611) from 'Center for Nanostructured Materials Technology' under '21st Century Frontier R&D Programs' of the Ministry of Science and Technology, Korea.

11:00 AM
The Photocatalytic And Antimicrobial Activity of Cotton Fabrics Treated with Silver-Doped Titanium Dioxide Nanocrystals: Guoliang Li[1]; *Bing Peng*[1]; [1]School of Metallurgical Science and Engineering, Central South University

Cotton fabrics were treated with silver-doped titanium dioxide nanocrystals in self-made finishing agent and general non-iron finishing agent to provide the photocatalytic and antimicrobial properties for cotton fabrics by linking of silver-doped titanium dioxide to cellulose structure. The concentration of silver-doped TiO2 nanocrystals in the finishing agents as well as the treatment conditions significantly affected the properties of treated cotton fabrics. The treated cotton fabrics showed fine photocatalytic activity to enhance the decolorization, degradation of methyl orange under UV light irradiation, good antimicrobial activity against Escherichia coli, and strong time effectiveness of photocatalytic and antimicrobial properties which was characterized by the standard test to washing. The optimal property was obtained when the cotton fabrics were first treated with general non-iron finishing agent, then self-made finishing agent including 3% silver-doped TiO2 nanocrystals, predried at 80° for 5 min, and cured at 120° for 3min.

11:15 AM
The Photocatalytic Activity of N-Doped TiO2 under Sunlight: Liqiang Liu[1]; Bing Peng[1]; *Liyuan Chai*[1]; [1]School of Metallurgical Science and Engineering,Central South University

In order to utilize sunlight in a photocatalytic reaction, yellow N-doped titania was prepared by calcination of a mixture of the hydrolysis product(H2TiO3) of TiOSO4 and urea. The catalysts were characterized by XRD, SEM and UV-Vis absorption spectra. The results showed that all catalysts were anatase, and the doping of nitrogen could extend the absorption spectra from UV light to the visible light region. The photocatalytic activity of N-doped TiO2 was also characterized by degradation of methyl orange. The results further showed that the photocatalytic activity was affected by calcination temperature, time, Ti/N ratio, pH and concentration of titania. The photocatalytic reaction rate of N-doped TiO2 was more rapid than the pure titania. There existed an optimal Ti/N ratio for the catalysts calcined at 400°, which resulted in the highest photoctalytic activity.

11:30 AM
Erosion-Corrosion Resistance of Plasma Sprayed Nanostructured Titanium Dioxide Coating: Abdul B Jabbar[1]; *Ahmad Zaki*[1]; [1]KFUPM

Nanostructured Titanium Dioxide (n-TiO2) thermal sprayed coatings have demonstrated a superior strength and durability compared to conventional TiO2 coatings. Whereas some studies on the corrosion resistance of plasma air sprayed n-TiO2 coatings have been conducted in the past, data on the erosion-corrosion behavior of these coatings is seriously lacking. Nanostructured powder (99% min purity) from METCO; AE9340, AE9342, and AE9303 were used to make agglomerates. Powder AE9342 was spray dried and densified, whereas powder AE9303 was chemically precipitated and spray dried. A high density polyvinyl chloride (HDPVC) was constructed for erosion-corrosion studies. AE9342 showed a dense oxide layer and elongated oxide covered lamellae with a spherical morphology and a very narrow inter-lamellar zone. AE9303 showed an uneven surface morphology and high pore density. The specimens were subjected to a maximum velocity of 4 ms–1 in a NaCl-polystyrene slurry. Specimen AE9342 dried and densified showed a lower sensitivity to erosion-corrosion compared to AE9303 (dried and sintered n-TiO2 coating). No appreciable difference between the corrosion resistance of conventional TiO2 coated (standard) and n-TiO2 coated (AE9342 was observed). The erosion-corrosion resistance of plasma sprayed microstructure TiO2 coating depends mainly on the characteristic of the feed powder, dispersion of slurry, reconstituted nano-powder and the control of key spraying parameters. The resistance of the nanostructured coatings to erosion-corrosion may further be improved by using impervious sealants and bond coatings with greater adhesion with the substrate.

11:45 AM
Corrosion Behavior of Nanostructured Titanium Dioxide Coating in Neutral Sodium Chloride Solutions: *Ahmad Zaki*[1]; Abdul B Jabbar[1]; [1]KFUPM

Studies were undertaken to determine erosion corrosion resistance of plasma air sprayed nanostructured and conventional titanium dioxide coatings (n, TiO2) in a sodium chloride polystyrene slurry. Nanostructured powder (99% min purity) from METCO; AE9342, and AE9303 were used to make agglomerates. Powder AE9342 was spray dried and densified, whereas powder AE9303 was chemically precipitated and spray dried. Erosion corrosion studies were conducted in a customized loop. Specimen AE9342 showed a spherical surface morphology and very narrow inter-lamellar zone whereas AE9303 showed an uneven surface morphology and high pore density. Specimen AE9342 showed lower sensitivity to erosion-corrosion compared to AE9303. No appreciable difference between erosion corrosion resistance of conventional TiO2 coated specimens and n-TiO2 coated was found.It was observed that the erosion-corrosion resistance of plasma sprayed microstructure TiO2 coating was dependent on the characteristic of feed powder, dispersion of slurry, reconstitution of nanopowder and control of key spraying parameters.

Alumina and Bauxite: Bauxite Ore Handling and Benefication

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Everett Phillips, Nalco Co; Sringeri Chandrashekar, Dubai Aluminum Co

Tuesday AM   Room: 2002
February 17, 2009   Location: Moscone West Convention Center

Session Chair: Songqing Gu, Zhengzhou Light Metal Research Institute

8:30 AM Introductory Comments

8:35 AM
Mining Scheduling at Paragominas Bauxite Mine: *Octavio Guimaraes*[1]; Henrique Santos[1]; Flavio Zelante[1]; Leonardo Alves[2]; [1]Vale; [2]Runge do Brasil

The chain of the bauxite and alumina business in terms of quality, costs and longevity is critical for the mine and refinery installations. This paper describes the impact of the size of the mine according to the bauxite quality required by the Refinery, the influence of level of knowledge of the deposit on the life of the mine, and finally, some scenarios changing the some economic factors, such as price on the bauxite. So, there are several scenarios, which could help the decision maker to get the best solution joining the financial, quality and other strategic issues. Beside this, each scenario results gives a better understanding of the business risks and opportunities to improve the results.

9:00 AM
Transformation of Sodalite to Cancrinite under High Temperature Bayer Digestion Conditions: Peter Smith[1]; *Bingan Xu*[1]; Christine Wingate[1]; [1]CSIRO

Cancrinite forms in high silica bauxite digestion at high temperature and is important to the Bayer process owing to its capacity to sequester impurities.

Transformation conversion of sodalite (SOD) to CAN in synthetic pregnant liquor at 250°C was measured by using a quantitative XRD method on residues. The effect of sodium carbonate in liquor, lime charge, lime type and reaction time was investigated on the transformation. It was found that the proportion of cancrinite increased with the concentration of Na_2CO_3 in the absence of lime. Lime dramatically promoted the transformation which was largely complete in a few minutes. Lime charge and type had little effect on the amount of total CAN (both sodium and calcium type) but was related to the fraction of total CAN that was calcium substituted. Formation of tri-calcium aluminate and calcite consumed a large portion of the added lime, reducing the efficiency of CAN formation. Silica concentration in Bayer green liquor decreased with increasing CAN, suggesting CAN formation is beneficial to alumina quality and scaling control. Soda reduction in DSP is directly related to calcium content in CAN, indicating that the mechanism of soda reduction is the substitution of calcium for sodium in cancrinite cage.

9:25 AM

Desilication of Bauxite Ores Bearing Multi-Aluminosilicates by Thermochemical Activation Process: *Guanghui Li*[1]; Tao Jiang[1]; Na Sun[1]; Xiaohui Fan[1]; Guanzhou Qiu[1]; [1]Central South University

The bauxite ores are relatively abundant in China, but most of them are disapore type, which is characterized as high aluminum, high silicon content, and low A/S ratio in comparison with gibbsite ores. Silica minerals mainly occur as aluminosilicates such as kaolinite, pyrophyllite, illite etc. It is difficult to remove SiO_2 from the ores and to increase A/S ratio by physical processing methods. As the three aluminosilicate minerals can be activated and form active amorphous SiO_2 by thermal treatment, thermochemical activation (TCA) process followed alkali-leaching was developed to remove SiO_2 from the ores. By the process, a concentrate with 11 A/S ratio and 50% desilication has been achieved for a raw bauxite ore of 5.88 A/S ratio, with 12.5% illite, 6.0% pyrophyllite, 3.2% kaolinite under the optimum conditions of thermal activation and alkali leaching. Chemical principle of thermochemical activation of aluminosilicates has also been investigated in this paper.

9:50 AM

Reductive Roasting and Magnetic Separation of Greek Bauxite Residue for Its Utilization in Iron Ore Industry: *Anthimos Xenidis*[1]; Charalabos Zografidis[1]; Ioannis Kotsis[1]; Dimitrios Boufounos[2]; [1]NTUA; [2]Aluminium of Greece SA

The treatment of Greek bauxite residue through reduction roasting and magnetic separation is investigated. A sample of Greek bauxite residue, hematite being the predominant iron carrier mineral, is subjected to reduction roasting to produce a calcine with intense magnetic properties, where magnetite or metallic iron are the main iron mineral phases. The calcine is then subjected to wet magnetic separation so that the magnetic product can be utilized as a feed for sponge or cast iron production. The effect of parameters such as temperature, intensity of reducing conditions, intensity of magnetic field or dispersing agent addition rate on the result of both processes is investigated. It was indicated that reductive roasting at 500°C followed by wet magnetic separation of the calcine produced at low current intensity (0.04 A) and simultaneous addition of a dispersant resulted in the production of a magnetic product with iron content as high as 36%. The obtained results regarding iron recovery were very promising providing input for further research on the optimization of the proposed method.

10:15 AM Break

10:35 AM

Reaction Behavior of Sulphur Existed in Diasporic Bauxite in Bayer Digestion Process: *Zhonglin Yin*[1]; Wu Guobao[1]; Li Xinhua[1]; Lu Peiqian[1]; [1]Zhengzhou Research Institute,CHALCO

The reaction behavior of sulphur in Diasporic bauxite in Bayer digestion process is studied in this essay. The reaction extent of sulphur in Diasporic bauxite is enhanced along with the increase of the lime dosage in Bayer digestion process. The majority of the sulphur contained in the bauxite was leached into digestion liquor with in the form of S^{2-} and the percentage of the sulphur which would be in the red mud is about 20-30%. Only a little sulphur can be removed when the bauxite is calcined at some conditions. The main form of sulphur in the digestion liquor is SO_4^{2-} and about 50-60% sulphur in the bauxite will come into the red mud, the Na2S concentration in the digested liquor is less than 0.05g/L after the bauxite is calcined.

11:00 AM

Roasting Pretreatment of High Sulfur Bauxite of China: *Guozhi Lv*[1]; Ting-an Zhang[1]; Li Bao[1]; Yan Liu[1]; Zhihe Dou[1]; Yan Li[1]; Xiaochang Cao[1]; Jicheng He[1]; [1]Northeastern University

Effects of roasting temperature and roasting time on sulfur content of bauxite, digestion performance and settling performance of high sulfur bauxite by roasting pretreatment using Rotary tube furnace were studied. Changes of microscopic appearance and crystal structure were analyzed by SEM and XRD.The results indicate that the sulfur content are successfully discharged as gas form through roasting, sulfur content of high sulfur bauxite meets the industrial standard at the conditions roasting temperature of 750° and holding time of 60min. The digestion rate of alumina is above 97% at the condition of digestion temperature of 220° and holding time of 60min. Roasting pretreatment make the apparent of red mud loose and porous, and transformed goethite into hematite, the settling performance were improved, particles size of red mud became smaller greatly. The digestion slurry of roasting ore in Rotary tube furnace has better settling performance than head ore.

11:25 AM

Techno-Economic Review on Processing Technologies on Low Grade Diasporic Bauxite: *Jibo Liu*[1]; Wangxing Li[1]; [1]Aluminum Corporation of China Limited

As the alumina refineries in China being greatly developed, the grade of bauxite have rapidly dropped, many technologies including Flotation-Bayer process, Lime-Bayer process, Bayer- Sintering (serial, parallel and mixed combined) and Bayer-Hydrometallurgy process etc. have developed or innovated to process on the low grade disporic bauxite in China, which one is the relatively economical process at the present situation is a difficult problem to clarify. In this article, process models of different processes were developed, the economic indices including investments, costs and consumptions were estimated by calculating the equipment scale and material consumption in each phases of the process. The techno-economic evaluation was done to analysis the advantages and disadvantages of each processes, and the suggestions of the process choice on dealing with the low grade diasporic bauxite in China was proposed.

11:50 AM

Study on Bayer Process and Soda-Lime Sintering Process of Special Diasporic Bauxite with High Silica: *Wenzhong Cao*[1]; Weiwei Tian[1]; Bo Qiao[2]; [1]Environmental College of Nanchang University; [2]Chemistry Engineering College of Jiujiang University

The bauxite in eastern region of China is diasporic type with high silica containing about 8-16% SiO_2. Main silica minerals in the bauxite are kaolinite, chamosite and illite. Technological investigations work carried out based on Bayer process and Soda-lime sintering process, Sintering and leaching properties of clinker in sintering process, the settling characteristic of red mud were determined. The presence of high silica causes high bound soda losses in the red mud in Bayer process, though a part of the silica content was in the form of chamosite. However, the bound soda losses can be greatly reduced using the soda-lime sintering process. Therefore, it is necessary to study the Bayer process. Soda-lime sintering and leaching properties of the special diasporic bauxite with high silica.

12:15 PM Concluding Comments

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

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

Tuesday AM Room: 2001
February 17, 2009 Location: Moscone West Convention Center

Session Chair: Gyan Jha, ARCO Aluminum Inc

8:30 AM
Multiple Extrusion and Consolidation of Al-4Mg-1Zr: *Daniel Aguilar Garcia*[1]; Richard Dashwood[2]; Martin Jackson[3]; David Dye[1]; [1]Imperial College; [2]University of Warwick; [3]University of Sheffield

In the continued quest for metallic alloys with better properties, metallurgists have employed a variety of thermo-mechanical processing routes and non-conventional methods. While steady progress has been made in this area, recent work promises to produce alloys with a step change in properties via severe plastic deformation (SPD) techniques. Several SPD techniques are now being studied such as Equal-Channel Angular Pressing (ECAP), High Pressure Torsion (HPT) and Accumulative Roll Bonding (ARB). In this paper a new SPD technique is applied to a novel experimental alloy, Al-4Mg-1Zr. The alloy has been sub jected to six passes of conventional extrusion. The mechanical properties and the microstructure was studied after each pass. Analysis showed that after the each pass the microstructure has been refined, the primary Al3 Zr particles were broken down and the hardness increased slightly. However, the yield stress and the ultimate strength increased significantly after the first pass and decreased for the following passes.

8:50 AM
Al-Zn-Mg for Extrusion - Hot Workability: *Hugh McQueen*[1]; Paola Leo; Emanuela Cerri[2]; [1]Concordia University; [2]University of Salento

Al-Zn-Mg alloys (without Cu: lower strength, less quench sensitivity) are useful for terrestrial applications due to good extrudability. As-cast Al-5.5Zn-1.2Mg was torsion tested over 250 - 500°C and 10-2 to 5 s-1. The peak strength fell markedly from 300 to 400°C and then slowly up to 500°C; it was lower for lower strain rates. In the aged starting material, this is indicative of more rapid precipitate coalescence and solution at higher temperature. The ductility rose rapidly from 300 to 400°C and with decreasing strain rate, but a 500°C, it was high but inconsistent. The constitutive analysis by the sinh equation gave Q, 165 kJ/mol; n = 1. Elongated grains exhibited larger subgrains at higher temperature. In hot tensile tests, flow curve shapes are similar, peak stresses are fairly consistent (218 kJ/mol; n, 1.75) and ductility (fivefold less) varies similarly. Comparison is made to 7004, 7020, 7075 and 7012 alloys.

9:10 AM
Microstructural Control through Heat Treatment Process in an Aerospace Aluminum Alloy: *Zainul Huda*[1]; [1]University of Malaya

The 2017 aerospace aluminum alloy was characterized through metallographic investigations. A series of precipitation strengthening and age-hardening heat treatment processes involving solution treatment at 550°C followed by quenching (and tempering for various time-durations) were conducted for the 2017 alloy. Microstructural characterization of the heat-treated samples showed effective distribution of fine γ' particles in the α-matrix of the aluminum alloy; these microstructural features enable us to develop proper precipitation strengthening and age-hardening heat-treatment process parameters for the 2017 aluminum alloy for aerospace application.

9:30 AM
Influence of Ge-Si Additions on Aging Response and Property in 2xxx Alloys: *Linzhong Zhuang*[1]; Shangping Chen[1]; Andrew Norman[1]; Achim Bürger[2]; Sabine Spangel[2]; [1]Corus RD&T; [2]Aleris Aluminum Koblenz GmbH

2xxx alloys added with Ge-Si display a unique combination of rapid aging response, higher peak strength, and extended thermal structure stability. Furthermore, due to a much refined distribution of precipitation, 2xxx+Ge/Si alloys show also a significant improvement in fracture toughness, or damage tolerance. High resolution transmission electron microscopy (HRTEM) examination showed that the Ge-Si additions modify the conventional precipitation sequence in Al-Cu based alloys. In Ge-Si added alloys, Ge-Si clusters quickly nucleate and grow during elevated-temperature aging. The Ge-Si particles then act as nucleation sites for γ' precipitates, resulting in a peak-aged microstructure consisting of a dense distribution of γ' attached to Ge-Si.

9:50 AM
Microstructure and Mechanical Properties of Cast Hypereutectic Al-SiAlloys with High Magnesium Content: *Animesh Mandal*[1]; M.M. Makhlouf[1]; [1]Worcester Polytechnic Institute

Magnesium in excess of the quantities typically found in commercial hypereutectic Al-Si alloys can produce alloys with enhanced microstructure and attractive mechanical properties. With addition of Mg to hypereutectic Al-Si alloys, the primary silicon phase is suppressed and is replaced with a fine dispersion of small Si particles. However, an abundance of large Mg2Si particles with Chinese script morphology also forms in the microstructure and unfavorably influence the tensile properties of the alloy. Efforts were made to overcome the negative effects of these particles by manipulating their size and morphology. Several additives were made to a hypereutectic Al-Si-Mg alloy and their effect on the cast alloy was determined. The alloy treated with Misch Metal and Strontium showed promising results. The Mg2Si particles that formed in castings made from this alloy were very small and almost spherical; and the room temperature tensile and yield strengths of cast bars were remarkably high.

10:10 AM
Electron Microscopy of Commercial Purity Al-2024 (Al-Mg-Cu) after Accumulative Roll-Bonding: *Andreas Kulovits*[1]; Bryan Webler[1]; Anirudha Deshpande[1]; Jorg Wiezorek[1]; [1]University of Pittsburgh

Al-2024 has been severely plastically deformed using accumulative roll bonding (ARB), which increased the hardness from a value equivalent to a tensile strength of about 410MPa (HV=138) prior to deformation to about 720MPa (HV=225). Changes in the microstructure have been investigated using imaging, diffraction and analytical methods of transmission electron microscopy (TEM). Two morphologically different regions, namely an ultra-fine grained (UFG) region that contains elongated grains and regions comprised of equiaxed nanocrystalline (NC) grains, have been observed in the ARB product. Electron diffraction analysis showed that the same phases were present prior to and after severe plastic deformation by ARB in the UFG regions. In the NC region, however, evidence of dynamic solid-state reactions has been observed experimentally. The application of high strains in conjunction with dynamic microstructural transformations facilitates grain refinement beyond the UFG regime into the NC size regime for this commercial purity Al-alloy.

10:30 AM Break

10:45 AM
Annealing Behavior of an Heavily Deformed Aluminum Alloy in 20 Tesla Magnetic Field: *Samuel Adedokun*[1]; [1]FAMU-FSU College of Engineering

A plate of aluminum alloy 6061 was given 85% deformation by cold rolling. Samples from the rolled specimen were heat treated for different times and at different temperatures in a 20 tesla resistive magnetic field. The effect of time and temperature on the heavily deformed specimens under 20 Tesla magnetic field was examined with the use of an Environmenetal Scanning Electron Microscopy equipped with an Orientation Imaging Microscopy (OIM) to study the changes in the grain size distribution and the grain boundary misorientation of the samples tested. The results indicate that the magnetic field of 20 Tesla increased the average grain size of the Aluminum alloy 6061 when compared with the aluminum alloy heat treated when the magnetic field was turned off. No effect on the grain boundary misorientation was noticed.

11:05 AM
Distribution of Trace Elements in Sr-Modified and Grain Refined Hypoeutectic Al-Si Alloy: *M. Faraji*[1]; L. Katgerman[2]; [1]M2i/ Delft University; [2]Delft University of Technology

The nucleation process of a commercial hypoeutectic Al-Si foundry alloy (Al-7Si-0.4Mg) unmodified, Sr-modified, grain-refined and Sr-modified + grain-refined has been investigated using optical microscopy and scanning electron microscopy (SEM). The results showed that adding strontium increased the number of eutectic silicon particles sensibly; however, the size of particles did not change considerably. Additionally, the results showed that grain refiner (AlTiB) reduced the incidence and size of porosities in both grain-refined and combined modified and grain-refined specimens compared to untreated condition. To study solidification and responsible mechanism of nucleation and modification in this alloy, electron probe microanalysis technique (EPMA) was used and the distribution of trace elements such as titanium, boron, phosphorus and strontium in the microstructure was analysed. Using EPMA, the negative interaction of strontium and AlTiB was closely examined and the optimised level of addition is suggested as 20 ppm boron (present in Al-3Ti-B) and 200 ppm strontium.

11:25 AM
Effects of Ultrasonic Treatment on Microstructures and Properties of Hypereutectic Al-Si Alloys: *Haikuo Feng*[1]; [1]Jilin University

Microstructures and properties of Al-23%Si alloys were gained with and without ultrasonic treatment in a novel horn crucible designed specially for this experiment. Evolution, morphology and distribution of microstructure of alloys were investigated. The results show that the size of primary Si particulates decreased from 500μm to 180μm and the morphology of a-Al phase had been changed from dendritic crystal to equiaxial crystal under the ultrasonic treatment. However eutectic phase was coarser than that without ultrasonic treatment. In addition the ultimate tensile strength and the wear resistance of Al-23%Si alloy with ultrasonic treatment were better than that without ultrasonic treatment. The key on better properties of alloys treated by ultrasonic were discussed.

11:45 AM
Effects of Zn on the Microstructures and Mechanical Properties of Al-Mg-Mn-RE Alloys: *Hua Shen*[1]; Guangchun Yao[1]; Weidong Yang[1]; [1]School of Materials and Metallurgy Northeastern University

Al-2,3,4wt%Mg-0.6wt%Mn-0.3wt%RE alloys were prepared and effects of different Zn contents on the microstructures and mechanical properties of Al-Mg-Mn-RE alloys were studied in this work.. Microstructures mechanism thinks that intensifying phase MgZn2 could be formed while adding Zn and Mg simultaneity to aluminum, so intensifying action produced obviously to alloys. Mechanical tests were carried out at room temperature. The results showed that Al-Mg-Mn-Zn-RE alloys were of resisting crazing stress and enough intension when Zn/Mg ratio was 2.7. Rigidity tests results showed that these series of alloys were harder than pure aluminum.

12:05 PM
Solidification and Processing of Aluminum Based Immiscible Alloys: *Hiren Kotadia*[1]; Jayesh Patel[1]; Zhongyun Fan[1]; Evelyn Doernberg[2]; R Schmid-Fetzer[2]; [1]Brunel University, West London; [2]Clausthal University of Technology

The Al-Sn based immiscible alloys have significant potential for bearing and superconducting applications. However, the mixing and understanding of solidification process for immiscible alloys have been long standing challenges for their development. This paper presents solidification, microstuctural evolution of the Al-Sn-Cu alloys and describes the mechanism of effective mixing by the intensive shearing. The solidification path of Al-Sn-Cu alloys was systematically investigated with differential scanning calorimeter and compared with the calculated phase diagrams. The experimental work was also focused on analyzing the effects of shear rate, temperature and time on Sn-rich droplet size and their distribution. Mechanical properties of solidified Al-Sn-Cu alloys have been investigated. Experimental results suggest that the intensive shearing process produces homogeneous, finely dispersed Sn-rich droplets and improves mechanical properties.

Aluminum Hot Rolling: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee
Program Organizer: Kai Karhausen, Hydro Aluminium

Tuesday AM — Room: 2010
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Kai Karhausen, Hydro Aluminium

8:30 AM Introductory Comments

8:35 AM Keynote
Through-Process Texture and Microstructure Modeling of Aluminum Alloys from Hot Rolling through Final Annealing: *Guenter Gottstein*[1]; Carmen Schaefer[1]; Volker Mohles[1]; Olga Sukhopar[1]; [1]RWTH Aachen

An improved through-process modeling scheme is proposed for the prediction of recrystallization textures and microstructures during sheet processing. The deformation behavior is modeled by the advanced deformation texture model GIA-3IVM+, which is based on a multi-grain approach and incorporates the work hardening behavior in terms of the dislocation density evolution. The recrystallization model utilizes a spatially resolved adaptive cellular automaton code CORe, which also considers orientation dependent recovery on a grain level. Since the treatment of recovery in CORe is consistent with the formulation in GIA-3VM+ this enables the consequent tracking of changes in dislocation densities throughout the whole process. Nucleation is accounted for by separate nucleation models which allow for different nucleation mechanisms to become active. The final microstructure after recrystallization depends sensitively on the nucleus frequencies and on the amount of prior recovery. The presented modeling setup allows an improved prediction of final recrystallization textures and grain sizes.

9:15 AM
Modeling Hot Rolling of Al-705X Alloys: *Margaret Koker*[1]; Jonathan Dantzig[1]; Armand Beaudoin[1]; [1]University of Illinois At Urbana-Champaign

We present a model for texture, damage and recrystallization evolution during hot rolling of Al-705X alloys. A constitutive model, based on experimental hot compression tests, is implemented as a non-Newtonian fluid. The rolling process is then simulated through multiple passes at user-specified draft and speed, and the results are used as input to a viscoplastic self-consistent model for texture evolution to predict the final texture as a function of position in the laboratory. The model is calibrated using measured temperatures, textures, roll torque, and other process features. We also predict damage in the rolled slab using a model that combines shear rate and temperature. The model is conveniently packaged into a graphical user interface. We present several case studies to show how the model can be used to improve rolling practices. This work represents the results of an ongoing development program supported by ALCOA, DOE and Kaiser.

9:35 AM
Property Control during Aluminum Sheet Fabrication using the Through Process Modelling (TPM) Approach: *Nitin Singh*[1]; Nicolas Kamp[2]; Richard Hamerton[1]; [1]Novelis Global Technology Centre; [2]Novelis Deutschland GmbH

The importance of linking fabrication process steps with microstructural evolution within the Alumiuim Rolling industry is widely recognized and work has progressed in alliance with universities in developing process informed microstructural models. The aim is to gain an indepth understanding of the link between certain process steps e.g. casting, homogenization, rolling etc. and the material microstructure. This knowledge becomes particularly important when new alloys and new processes are being introduced on an industrial scale. Novelis uses the physically based models developed by IMM, RWTH University, Aachen in their TPM efforts. In this presentation, our experience with successes and difficulties in calibrating these models to specific alloys and their implementation in predicting microstructures would be discussed. A comparison would be made with certain in-house models developed over many years to suggest directions for further development of these models.

9:55 AM
Profile Optimization on a Duo Hot Mill: *Kai Karhausen*[1]; Ioannis Neitzel[1]; Luca Francescutti[1]; [1]Hydro Aluminium

Although state of the art hot rolling mills are usually in quarto-design, equipped with computer controlled profile and shape control actuators, older duo mill stands are still in operation in some locations. Such mills are either limited in their product range or have only limited possibilities to control the strip profile for a larger variety of product qualities. Essentially only the ground work roll crown combined with a suitable pass schedule can be changed to affect the strip profile.In the present paper, a duo mill has been analyzed with the aim of finding an operating window for a given target profile covering a large spectrum of alloys and strip widths. By a series of profile/shape simulations on the whole production spectrum, the thermo-mechanical behavior of the stand was characterized and the sensitivity of the main factors for the profile generation was determined. Finally an optimum set of differently ground work roll crowns was determined by computer simulation to achieve a defined strip profile for the whole production spectrum.

10:15 AM Question and Answer Period

10:25 AM Break

10:40 AM
Evaluation of Recovery Kinetics of the Aluminum Alloy AA3103 Using Stress Relaxation and Double Tension Tests: *Sheila Bhaumik*[1]; Günter Gottstein[1]; Volker Mohles[1]; [1]IMM

The softening processes, recovery and recrystallization, are of significant scientific and technological relevance especially for materials with high stacking fault energy. Since recovery is always connected to recrystallization, there is an urgent need to advance our understanding of recovery in particular with regard to through process modeling. Within the scope of the project stress relaxation and double tension tests at elevated temperatures were conducted on a commercial aluminum alloy 3103. The recorded stress-time evolution based on the stress relaxation and double tension tests were compared to gain a better understanding of the mechanisms governing the recovery processes and to obtain a reliable evaluation of recovery kinetics as well. This comparison enables to verify whether the low cost stress relaxation measurements can, in principle, replace the more laborious double compression tests. Furthermore, the obtained parameters for recovery were analyzed in terms of temperature dependency etc.

11:00 AM
Patterns of Deformation and Associated Recrystallization in Warm/Hot Deformed AA6022: *S. Raveendra*[1]; S. Mishra[1]; H. Weiland[2]; I. Samajdar[1]; [1]IIT Bombay; [2]Alcoa

Microstructural developments during hot-rolling of aluminum alloys significantly impact the forming process. In a deformation simulator, AA6022 samples were plane strain compressed (equivalent to hot-rolling) at different temperatures and strain rates to strains of 1 and 2. The deformed samples contain finite percentage of static recrystallization. The deformed/recrystallized regions were partitioned based on in-grain misorientation developments and grain size. Zener Holloman parameter (Z), which ties deformation temperature and strain rate had clear effects on microstructure and textural changes. At lower Z, i.e. at higher deformation temperatures, there is increase in deformed Cube {001} <100> (with increase in strain), which is due to the thickening of deformed Cube bands. Such a pattern cannot be explained from Taylor type deformation texture simulations, incorporating both octahedral and non-octahedral slip systems. These, in turn, strongly affected the associated recrystallization behavior – including relative contributions from particle stimulated nucleation and contributions from deformedgrains/bands of different ideal orientations.

11:20 AM
Pore Evolution during the Homogenization and Rolling of Direct Chill Cast Al–6Mg Alloy: *Richard Dashwood*[1]; Anirut Chaijaruwanich[2]; Hiromi Nagaumi[3]; Peter Lee[4]; [1]University of Warwick; [2]King Mongkut's Institute of Technology; [3]Nippon Light Metal Company Ltd; [4]Imperial College London

The evolution of porosity during homogenisation and rolling (with a low reduction per pass) was quantified using two-dimensional metallography and three-dimensional X-ray microtomography (XMT) techniques. This paper will demonstrate the misinterpretations that can take place when only considering metallographic evidence. For example the metallographic evidence implied that the mean pore size increased during homogenisation and also in the centre of the plate during the initial rolling passes. However XMT demonstrated that intra-pore Ostwald ripening of the tortuous pore networks formed during DC casting was the key mechanism driving the evolution of pore morphology. Finite element modelling showed that during the initial low reduction ratio rolling passes the central region of the plate experienced a tensile, rather than a compressive, hydrostatic stress, explaining the initial pore ripening. A relationship between the roll geometry and the hydrostatic stress has been derived and the critical geometry identified.

11:40 AM
Effect of Homogenization Treatment on Microstructural Evolution of 1050 and 1200 TRC Aluminium Alloys: *Aziz Dursun*[1]; Beril Corlu[1]; Canan Inel[1]; Murat Dundar[1]; S. Levent Aktuð[1]; [1]Assan Aluminium

Homogenization in twin roll casting(TRC) is an essential annealing treatment in order to obtain desired mechanical and microstructural properties like deep drawability in heat shield(1050) and matte surface appearance in packaging foil (1200) applications. Gradients in solification rates developed during TRC results in concentration gradients throughout the thickness with a supersaturated region of alloying elements near the surface. In order to minimize the influence of concentration gradients on material performance and ensure homogeneously distributed particles, a series of homogenization treatments between 520 and 580°C at different holding time were performed on 1050 and 1200 aluminium alloys. Size, distribution and composition of secondary particles were determined using an optical and scanning electron microscope. Microstrucal evolution was monitored by hardness and tensile tests results. The results showed that a critical homogenization temperature and holding time lead to significant coarsening and more uniform particle size distribution thereby superior properties for both alloys.

12:00 PM
Characterization of Edge Cracking Using a Crystal Plasticity Model with Damage Evolution: *Soondo Kweon*[1]; Armand Beaudoin[1]; Russell J. McDonald[1]; [1]University of Illinois at Urbana Champaign

A challenge to the analysis of edge cracking in aluminum rolling industry lies in the nature of the stress state at the side of the slab: traditional models for damage evolution are dependent on the state of tensile hydrostatic stress, whereas edge cracking is developed at free surfaces and in the presence of shear. Recent research efforts have augmented traditional models for damage to include the effect of shear, through the Lode parameter. These models are generally applied through use of J2 plasticity. In this study, we focus on thermo-mechanical conditions leading to edge cracks, giving attention to the mesoscale process of grain-to-grain interaction. A polycrystal, modeled using finite elements, is subjected to different loading conditions. Hydrostatic stresses develop through grain interaction, even with "average" deformations of pure shear. The development of damage in the polycrystal, both with and without use of the Lode parameter, is contrasted with experimental results.

12:20 PM Question and Answer Period

Aluminum Reduction Technology: Potline Performances and Vision

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Gilles Dufour, Alcoa Canada, Primary Metals; Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Alcan; Jayson Tessier, Alcoa Deschambault

Tuesday AM Room: 2004
February 17, 2009 Location: Moscone West Convention Center

Session Chair: Jules Côté, Aluminerie Alouette Inc

8:30 AM Introductory Comments

8:35 AM Panel Discussion
Representatives from eight smelters around the world are invited to present performances achieved at their smelters.

11:15 AM Question and Answer Period
The smelter representatives will answer questions from the audience.

Biological Materials Science: Drug Delivery and Imaging

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Ryan Roeder, University of Notre Dame; John Nychka, University of Alberta; Paul Calvert, University of Massachusetts Dartmouth; Marc Meyers, University of California

Tuesday AM — Room: 3014
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

8:30 AM Keynote
Lipid-Modified Polymers as Biomimetic Nucleic Acid Carriers: *Hasan Uludag*[1]; Vanessa Incani[1]; Artphop Neamnark[1]; Orawan Suwantong[1]; [1]University of Alberta

Cationic polymers are utilized to deliver anionic plasmid DNA into cells. The polymer condense string-like DNA molecules into compact structures for passage through the plasma membrane. The cationic polymers, however, are effective only at doses where significant toxicities on cells are observed. Our research program aims to utilize naturally–occurring lipids to enhance the ability of polymers for DNA delivery into primary cells. Using lipid conjugates, we obtained polymers that were capable of condensing plasmid DNA effectively into 100-200 nm particles. The particle sizes were effectively controlled by the hydrophobic substitution on the polymer, achieving lower particle sizes with more hydrophobic polymers. The hydrophobic polymer were capable of enhancing the delivery of plasmid DNA into the cells by ~10-fold. Using a model gene (GFP), the designed polymers enabled improved GFP expression in primary cells. Structure-function relationships on the effectiveness of designed cationic polymers will be presented.

9:10 AM Invited
Cell Membrane Penetrating Nanoparticles: *Francesco Stellacci*[1]; [1]MIT

Non biological materials when in contact with cells are either endocytoced or created transient pores that allow cell entrance but are cytotoxic. Cell penetrating peptides on the other hand have the property of permeating cell membranes without generating transient pores. We will show that gold nanoparticles coated with a mixture of hydrophobic and hydrophilic ligand molecules are simply endocytoced when these molecules have no special arrangement but behave as cell penetrating peptides when the molecules are arranged in an ordered fashion.

9:40 AM
Exploring Transferrin-Receptor Mediated Nanoparticle Cell Interactions at Single-Molecule Level during Cellular Uptake: *Abhilash Vincent*[1]; Suresh Krishna Moorthy[1]; Eric Heckert[1]; William Self[1]; Christopher Reilly[2]; Sudipta Seal[1]; [1]University of Central Florida; [2]Virginia Tech

Recent studies indicate that cerium oxide nanoparticles (Ceria NPs) can function as biological antioxidants due to their ability to switch between different oxidation states. Hence Ceria NPs show tremendous potential in cancer therapeutics and it is important to develop Ceria NPs supported chemotherapeutics drugs that can target cancerous cells and destroy them without affecting neighboring healthy cells. This is achieved by bioconjugating Ceria NPs with targeting agents that can bind to the over expressed receptors on the target cancer cell. In this work we are focusing on understand the cellular uptake mechanism of Transferrin protein coated Ceria NPs by A549 lung cancer cell. Single Molecule Force Spectroscopy (SMFS) was used to study the interaction force between Transferrin protein coated Ceria NPs and cancer cells. Inductively Couples Mass Spectrometer (ICPMS) results indicates A549 cells treated with Transferrin coated Ceria NPs showed better uptake compared to the cells treated with uncoated NPs.

10:00 AM
Controlled Release of Bovine Serum Albumin Protein Using Calcium Phosphate Nanocarriers: Sudip Dasgupta[1]; Amit Bandyopadhyay[1]; *Susmita Bose*[1]; [1]Washington State University

Calcium phosphate (CaP) based ceramics are of significant interest due to their bioactivity and chemical similarity with inorganic component of bone. Bovine serum albumin (BSA) protein release behavior from β- tricalcium phosphate (β-TCP) and calcium deficient hydroxyapatite (CDHA) nanoparticles (NP) were studied. Both surface adsorption and chemical synthesis route were used to make protein loaded CaP nanocarriers. Pure and Zn^{2+} /Mg^{2+} doped hydroxyapatite (HA)-BSA NPs were synthesized by in situ co-precipitation technique. BSA release rate from ex situ synthesized β-TCP-BSA NPs found to be faster compared to that from CDHA-BSA NPs. Pure HA-BSA NPs showed slower BSA release compared to doped HA-BSA NPs. The presentation will focus on the effect of doping, crystallinity and synthesis process on BSA release behavior from CaP based nanocarriers.

10:20 AM Break

10:30 AM Invited
Characterization of Implantable Drug Delivery Bioceramics Using Magnetic Resonance Imaging (MRI): Joshua Bray[1]; Mark Filiaggi[1]; *Steven Beyea*[2]; [1]Dalhousie University; [2]National Research Council of Canada

While novel and innovative interventions in regenerative medicine hold great promise, such methods are only as effective as the ability to develop an empirical and mechanistic understanding of how/why they work. One such example is the use of resorbable bioceramics, which have potential to provide large, sustained concentrations of therapeutic agents to a specific tissue, while not exceeding the minimum toxic concentration in other tissues. Optimization of resorbable bioceramic design requires methods that will permit the non-invasive and non-destructive study of the spatially and temporally varying physicochemical changes that occur due to material degradation. The study of drug delivery biomaterials using Magnetic Resonance Imaging (MRI) therefore has the potential to significantly improve our understanding of the performance of such devices. A host of complimentary MRI techniques (e.g. microscopy, diffusometry and relaxometry) were used to study changes in CPP bioceramics due to initial material processing and subsequent degradation.

11:00 AM
Modeling Bio-Scaffolds: Structural and Fluid Transport Characterization Based on 3D Imaging Data: Liang Hao[1]; David Raymont[1]; Bruno Notarberardino[1]; *Philippe Young*[1]; [1]University of Exeter

Bio-scaffolds - which are commonly open celled porous structures - are increasingly used for tissue engineering and regenerative medicine. Numerical studies exploring the influence of architecture on structural and flow characteristics of porous media have been carried out assuming an idealized repeating unit cell approach. However, a number of studies have shown that the bulk properties of such irregular structures are poorly modeled using idealized unit cell approaches. High resolution three dimensional imaging techniques such as Micro-CT allow realistic porous structures to be straightforwardly and accurately scanned with sub-micron image resolutions. Combined with novel meshing techniques, these imaging techniques allow for robust conversion of the 3D data into models suitable for physics-based simulations. A number of studies will be shown which demonstrate the ease with which fidelic models can be generated and parametric studies will be presented which explore both fluid flow and structural properties of a range of bio-scaffolds.

11:20 AM
Imaging Microdamage in Bone Using a Barium Sulfate Contrast Agent: Matthew Landrigan[1]; Huijie Leng[1]; Ryan Ross[1]; Carl Berasi[1]; Xiang Wang[1]; Glen Niebur[1]; *Ryan Roeder*[1]; [1]University of Notre Dame

Accumulation of microdamage in bone tissue can lead to increased fracture susceptibility, including stress fractures in active individuals and fragility fractures in the elderly. However, clinically relevant damage and failure mechanisms remain poorly understood, in part, due to limitations imposed by current methods for imaging microdamage, which are inherently destructive, tedious and two-dimensional. Therefore, micro-computed tomography (micro-CT) has been investigated using a precipitated barium sulfate contrast agent to label damaged tissue. For proof-of-concept, the presence, spatial variation and accumulation of microdamage in cortical and trabecular bone specimens was nondestructively detected using micro-CT after staining with barium sulfate. Damage quantification using micro-CT with a barium sulfate stain was further validated against conventional histological methods. Opportunities and limitations for the new imaging technique will be discussed.

11:40 AM
Molecular Surface Modification of Gold Nanoparticles to Impart Specificity to Damaged Bone Tissue: *Ryan Ross*[1]; Ryan Roeder[1]; [1]University of Notre Dame

The accumulation of microdamage in bone tissue can lead to increased fracture susceptibility. However, there are currently no non-invasive methods to detect damage in bone tissue. Therefore, gold nanoparticles (Au NPs) are being investigated as a potential damage-specific X-ray contrast agent due to their biocompatibility, ease of surface functionalization and high X-ray attenuation. Au NPs were synthesized by citrate reduction to a mean particle size of 20 nm and surface functionalized with either glutamic acid, 2-aminoethyl-phosphonic acid, or 2-aminethyl dihydrogenphosphate, to impart either carboxylic acid, phosphonate or phosphate functionality to the particle surface. The particle size distribution and stability of as-synthesized and functionalized Au NPs was confirmed using DLS, TEM and UV-vis spectroscopy. Functionalized Au NPs exhibited specificity for artificially damaged regions on the surface of cortical bone tissue as shown by SEM and EDS. The binding affinity of functionalized Au NPs on hydroxyapatite crystals and was quantified using ICP-OES.

Bulk Metallic Glasses VI: Structures and Mechanical Properties 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, The University of Tennessee

Tuesday AM Room: 3007
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Takeshi Egami, University of Tennessee; Daniel Miracle, US Air Force

8:30 AM Keynote
Statistical Mechanics of Glasses and Liquids: *Takeshi Egami*[1]; Valentin Levashov[1]; Rachel Aga[2]; James Morris[2]; [1]University of Tennessee; [2]Oak Ridge National Laboratory

In crystals the atomic vibrations are described by phonons, and the statistical mechanics can be formulated with phonons as the bases. In liquids and glasses, however, phonons are strongly scattered and have a very short lifetime, and cannot form the basis for statistical mechanics. We found recently that the atomic dynamics are so localized in high temperature liquids that the dynamics of an atom and its nearest neighbor shell can be an excellent basis for the analysis of the atomic dynamics. They were found to obey the equipartition law for the potential energy, $U = (3/2)kT$. We show how this leads to calculating the glass transition temperature, structural relaxation and other thermodynamic quantities. This research has been sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract DE-AC05-00OR-22725 with UT-Battelle.

8:55 AM Invited
Size Effect in the Deformation and Failure of Metallic Glasses: *Ju Li*[1]; [1]University of Pennsylvania

Recent experimental and theoretical works have suggested possible size effect in the plasticity and failure resistance of metallic glasses. While bulk metallic glasses manifest negligible tensile ductility, nanoscale metallic glasses show significant ductility under tension (PNAS 104, 11155). Nanopillar compression experiments also show intriguing behavioral differences sensitive to pillar size. Multiple reasons could contribute to size dependence: (a) proliferation of amorphous-crystal interfaces or surfaces that directly mediate plasticity, as well as indirectly alter the glass structure, (b) Weibull-like statistics for the nucleation of a runaway flow defect from the condensation of shear transformation zones in a finite volume, and (c) intrinsic lengthscales such as the glue zone width in the aged-rejuvenation-glue-liquid model of a runaway shear band (Acta Mater. 54, 4293). A “Hall-Petch” like relation may exist with respect to the metallic glass sizescale, but for the useful tensile or compressive ductility instead of strength (Phys. Rev. B 77, 155419).

9:10 AM Invited
Atomic Structure of Solute-Rich Metallic Glasses: *Daniel Miracle*[1]; Dmitri Louzguine[2]; Larissa Louzguina[3]; [1]US Air Force; [2]Advanced Institute of Materials Research; [3]Institute of Materials Research

The efficient cluster-packing (ECP) model describes the atomic structure of metallic glasses using solute-centered atomic clusters with predominantly solvent atoms only in the first coordination shell as representative structural units. However, bulk metallic glasses are typically solute-rich, and the ECP model suggests that the first coordination shell surrounding a solute atom in such glasses must therefore contain a significant number of solute atoms. In fact, the number of solute atoms in the first coordination shell may approach the number of solvent atoms in the first shell, confusing conventional definitions of solute and solvent. These solute-rich structures have not been described in detail, but may hold important insights into the unusual stability of bulk metallic glasses. Drawing on experimental data from a recent assessment of binary and ternary metallic glasses, a description of solute-rich structures will be developed and discussed. Structural origins of bulk metallic glass stability will be examined.

9:25 AM
Characterization of Local Deformation during Low and High Strain Rate Joining of Bulk Metallic Glasses: *Nicholas Hutchinson*[1]; Yuan Zhang[1]; Glenn Daehn[1]; Katharine Flores[1]; [1]The Ohio State University

Previous work has demonstrated the viability of solid state joining techniques for bulk metallic glasses. However, optimization of these techniques requires detailed study of mechanical and diffusive mechanisms operative near the joint interface and their dependence on surface roughness and stress state. Recently, we characterized two solid state joining techniques which rely on mechanical work and plastic deformation at mating interfaces:. a high strain rate electromagnetic impact process and a quasi-static electro-thermo-mechanical process. Interface strengths of ½ the bulk strength have been obtained with the electro-thermo-mechanical process, while the electromagnetic process has produced glass to crystalline joints exhibiting apparent interface strengths that exceed the strength of the crystalline material. In the present work, interface geometry is varied to evaluate the effect of stress state and roughness on deformation at the interface during joining. Interface failure is characterized, and cross sections and failure surfaces are examined using SEM and TEM.

9:35 AM Invited
Anelasticity in a Metallic Glass and Local Flow at Shear Bands: *Michael Atzmon*[1]; Adam Ganuza[1]; Dongchan Jang[1]; Koteswararao Rajulapati[1]; [1]University of Michigan

Our recent indentation experiments have suggested that rolled amorphous Al86.8Ni3.7Y9.5 undergoes time-dependent deformation at room temperature. This result has motivated a detailed study of time-dependent deformation in this alloy. We used a combination of bend stress relaxation measurements and cantilever bending with a nanoindenter. Anelastic, i.e., time dependent and reversible, deformation is dominant at room temperature. We observe at least four distinct anelastic sites, in contrast with the common assumption that only two such site types exist. In order to observe local deformation at shear bands, we have created samples with a small number of shear bands by forming kinks in a melt-spun ribbon. These samples were then subjected to low, prolonged, stress. Using ex situ atomic-force microscopy, we have observed the change in offset at the intersection of individual shear bands with the surface. The results will be interpreted by considering the residual stress profile in the sample.

9:50 AM Invited
Relaxation Behavior of Ca-Based Bulk Metallic Glasses: *Oleg Senkov*[1]; Daniel Miracle[2]; [1]UES Inc; [2]US Air Force Research Laboratory

The temperature dependence of the Maxwell relaxation time of three Ca-based bulk metallic glasses (BMGs), $Ca_{65}Mg_{15}Zn_{20}$, $Ca_{50}Mg_{20}Cu_{30}$, and $Ca_{55}Mg_{18}Zn_{11}Cu_{16}$ was studied in the super-cooled liquid range, near the glass transition temperature, using a differential scanning calorimetry (DSC) method. The relaxation behavior of $Ca_{65}Mg_{15}Zn_{20}$ was found to be similar to that of SiO_2. The behavior of two other Ca-based BMGs was more fragile, but they were stronger than the Zr- and Mg-based BMGs. The strong liquid behavior of the Ca-based BMGs was concluded to be one of the reasons of their excellent glass forming ability.

10:05 AM Break

10:15 AM Invited

Atom Probe Analysis of Phases in a Zr-Based Bulk Metallic Glass with the Expectation Maximization Algorithm: *Michael Miller*[1]; Xun-Li Wang[1]; Daniel Haley[2]; Michael Moody[2]; Simon Ringer[2]; [1]Oak Ridge National Laboratory; [2]Australian Key Centre for Microscopy and Microanalysis

Atom probe tomography has been used to characterize the local structure and the phases present in a $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ bulk metallic glass. The atom probe data from materials in the as-quenched condition and after a heat treatment of 3.5 h at 658 K were analyzed with the Expectation Maximization algorithm and other standard techniques to decompose underlying phase information. The Expectation Maximum method provides the statistically most likely number of phases present and the fraction of each phase by calculating the nearest neighbor histograms in the atom probe data and then applying a maximum likelihood method. These results will be compared to the results from other traditional atom probe tomography phase analysis techniques. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by Basic Energy Sciences, U.S. Department of Energy.

10:30 AM

Contribution to a New Understanding of Deformation-Induced Ductility in Bulk Metallic Glasses (BMG): *Denise Beitelschmidt*[1]; Simon Pauly[1]; Min Lee[2]; Uta Kuehn[1]; Jürgen Eckert[1]; [1]IFW Dresden; [2]Korea Institute of Industrial Technology

Most BMGs show very limited plastic strain. In order to achieve a reliable process to enhance the plastic properties it is necessary to improve the understanding of atomic order in deformed samples. The change of mechanical behaviour of BMGs under different deformation conditions as pre-load, compressive or tensile stress is investigated. Different alloys are tested with regard to their mechanical properties: as-cast, after preload or deformation. Many (rolling-)deformed samples show series of shearbands with characteristic enhancement of the plastic properties. In order to get a better understanding a detailed investigation program with special interest in the shearbands' cross-over spots was performed. Furthermore the effect of structural changes under compressive pre-load below yield strength is investigated to obtain structural knowledge about materials with pre-load enhanced plasticity and the predicted non-lowered fatigue strength. Different investigations are presented leading to new concepts concerning the role of free volume in the mechanically treated samples.

10:40 AM Invited

Fragile to Strong Transitions in Zr- Based Alloys: *Ralf Busch*[1]; [1]Saarland University

The viscosities of six bulk metallic glass forming Zr-based melts of different complexity have been measured in the equilibrium liquid state. The viscosity vs. shear rate behaviour of the three quintary (Vitreloy105, Vitreloy106, Vitreloy106a), two quarternary (Vitreloy101, Zr65Cu17.5Ni10Al7.5) and one ternary (Zr60Cu25Al15) alloys have been studied above their respective liquidus temperatures in a custom built Couette concentric cylinder viscosimeter. Earlier, in the Be bearing Vitreloy 1, shear thinning behavior as well as a strong to fragile transition in the undercooled liquid had already been observed.[1] The actual study shows that for all tested alloys a fragile state was present above the liquidus temperature, like in Vitreloy 1 with fragility parameters of about D*=10 and a weak shear thinning behavior. Results from parallel low temperature studies by three point beam bending show that they are much stronger liquids in the vicinity of the glass transition with D* higher than 20, this being a strong indication for a strong to fragile transition between the glass transition and the liquidus temperature also in these alloys. Comparable to the behavior of the fragile state of Vitreloy1 shear thinning exponents varying between 0.8 and 1 have been found for the tested alloys. To directly confirm the strong to fragile transition, studies are done in the supercooled liquid regions of the respective alloys. However, like in Vitreloy 1 the fragile to strong transition seems to also promote crystallisation and only a small temperature and time window is available to observe the transition in the supercooled liquid directly. [1]C. Way, P. Wadhwa und R. Busch, Acta Mater. 55, 2977 (2007).

10:55 AM Invited

Structural Changes in Metallic Glasses Induced by Mechanical Deformation.: *Wojciech Dmowski*[1]; Takeshi Egami[1]; Andrew Chuang[1]; Yoshihiko Yokoyama[2]; Akihisa Inoue[2]; Yang Ren[3]; Bogdan Palosz[4]; [1]University Tennessee/MSE; [2]Institute for Materials Research/Tohoku University; [3]Advanced Photon Source/ANL; [4]Institute of High Pressure Physics/PAS

Glass deformation is accompanied by local rearrangement of atoms to accommodate shear strain. Disordered nature of the glass and small deformation volumes make it difficult to observe experimentally. However, the use of area detectors and high flux/energy X-rays makes such studies practical. We have examined structural changes induced by high temperature creep, axial tension, high temperature isostatic compression and high pressure torsion. Axial deformation leads to small but observable structural anisotropy, which can be analyzed in terms of the anisotropic components of the pair distribution function. We found that mechanical deformation involved rearrangement in clusters of atoms by local bond exchange that supported structural anisotropy in the deformed state. Structural changes induced by high pressure annealing were isotropic and affected chemical ordering. High pressure torsion resulted in isotropic but inhomogeneous changes in the local atomic structure. Work supported by the U.S. DOE under DE-AC05-00OR-22725.

11:10 AM

Structural Influences on Metallic Glass Relaxation: *Garth Wilks*[1]; Daniel Miracle[1]; [1]Air Force Research Laboratory

Variations in constitution and processing route (cooling rate and subsequent annealing) in ribbons made from the Al-La-Ni system are used to probe the deformation activation spectra for several conditions via bend stress relaxation. Deviations in spectra between conditions are rationalized in terms of "defect" states predicted by the Efficient Cluster Packing model that contribute to local free volume fluctuation and act as sites for unit shear process nucleation.

11:20 AM

The Effects of Partial Crystallinity on the Hydrogen Permeation Properties of Bulk Amorphous Metallic Systems: *Kyle Brinkman*[1]; Elise Fox[1]; Paul Korinko[1]; Thad Adams[1]; [1]Savannah River National Laboratory (SRNL)

It is recognized that hydrogen separation membranes are a key component of the emerging hydrogen economy. Potentially exciting materials for membrane separations are bulk metallic glass materials due to their low cost, high elastic toughness and resistance to hydrogen "embrittlement" compared to crystalline Pd-based membrane systems. However, at elevated temperatures and extended operation times structural changes including partial crystallization (devitrification) may appear in these amorphous metallic systems. A systematic evaluation of the impact of partial crystallization/devitrification on the diffusivity and solubility (i.e., permeability) behavior in multi-component Metallic Glass materials would provide great insight into the potential of these materials for hydrogen applications. This study will report on the impact of phase transformations occurring under different gas environments. This thermodynamic data as well the calorimetric determined crystallization rate will be used to intentionally crystallize a given material to varying degrees. Measurements of the hydrogen permeation flux as a function of phase composition have been performed and structure/property effects will be presented for commonly available Zr and Fe/Co based Metallic Glass materials.

11:30 AM Invited

Formation, Structure and Crystallization Behavior of Cu-Based Bulk Glass-Forming Alloys: *Dmitri Louzguine*[1]; Guoqiang Xie[2]; Song Li[2]; Qingsheng Zhang[2]; Wei Zhang[2]; C. Suryanarayana[3]; Akihisa Inoue[1]; [1]Tohoku University, WPI Advanced Institute for Materials Research; [2]Institute for Materials Research, Tohoku University; [3]University of Central Florida

We studied the structure and properties of Cu-Zr-based alloys having exceptionally high glass-forming ability and investigate the influence of Ag addition on their structure and crystallization behavior. Some bulk glassy alloy samples studied by high-resolution TEM were found to contain well developed medium-range order zones and nanoparticles. Some glassy alloys are also found to be highly sensitive to electron-beam irradiation. The crystallization kinetics of Cu55Zr45, Cu50Zr50, Cu55-xZr45Agx (x = 0, 10, 20), Cu45Zr45Al5Ag5 and Cu36Zr48Al8Ag8 glassy alloys was also analyzed and will be discussed based on classical nucleation theory. Cu35Zr45Ag20 alloy was found to exhibit possible phase separation upon heating within a supercooled liquid region. An influence of the cooling rate on the structure and properties of the Cu-based glassy alloys on heating was also studied. Some differences in the crystallization kinetics and phase composition of the ribbon-shape and bulk glassy samples of Cu36Zr48Al8Ag8 alloys will be presented.

11:45 AM
Fabrication and Mechanical Properties of Metal Particulates Reinforced Ni-Based Bulk Metallic Glass Composites by Spark Plasma and Microwave Sintering: *Guoqiang Xie*[1]; Dmitri V. Louzguine-Luzgin[1]; Song Li[1]; Akihisa Inoue[1]; [1]Tohoku University

The lack of ductility limits the number of applications of bulk metallic glasses. The most common method to overcome this problem is to introduce nano- or micro-scale crystalline phases into glassy matrix leading to the formation of multiple shear bands and an enhanced plasticity. In this study, using the mixed powders of gas-atomized Ni-based glassy powder blend with nano- or micro-scale metal (W, Cu, etc.) powders, we fabricated large-size glassy alloy composites (GACs) with ultra-high strength and enhanced ductility by a spark plasma sintering process. The microstructure of the sintered compacts and the interface between powder particles were characterized by SEM and HRTEM. The good bonding state among the particulates and the glassy matrix was recognized. The additional metal particulates causing deviation, branching and multiplication of shear bands should be responsible for good mechanical properties of the fabricated bulk GACs. Microwave-induced sintering behavior of the mixed powders was also investigated.

Cast Shop for Aluminum Production: Environment, Health and Safety

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Pierre Le Brun, Alcan CRV; Hussain Alali, Aluminium Bahrain

Tuesday AM — Room: 2005
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Seymour Epstein, Aluminum Association

8:30 AM Introductory Comments

8:35 AM
Sustainability and the Aluminum Industry: Future Strength: *Steve Larkin*[1]; [1]The Aluminum Association

We live today in an increasingly constrained world. Both as individual consumers, and as representatives of the manufacturing sector, we live and operate our businesses in the midst of record commodity, energy, and now food prices. This paper will discuss the opportunities these constraints present to the aluminum industry, and how sustainability has become the dominant paradigm through which customers will judge performance. Key metrics for measuring the success of the industry are used to highlight aluminum's strength versus competing materials.

8:55 AM
Meeting Environmental Challenges in the Casthouse: *Corleen Chesonis*[1]; Edward Williams[1]; David DeYoung[1]; [1]Alcoa Inc

Sustainability has become an important issue to the aluminum industry in recent years. Environmental regulations have become more stringent and societal pressures to reduce the environmental footprint of our operations have increased. In U.S. casthouses, the Secondary MACT regulations set specific limits on gaseous and particulate emissions from both furnaces and in-line metal treatment units. These concerns have led to efforts to reduce or eliminate the use of chlorine gas in metal treatment while maintaining metal quality and meeting environmental regulations. The steady progress made in Alcoa over the last twenty years can be characterized as a series of step changes in technology, both in processes and in equipment. These step changes have included mixed gas tube fluxing, bagged salt addition, bath carryover reduction, rotary gas fluxing, and rotary salt fluxing. The effectiveness of these methods and their impact on emissions levels will be summarized.

9:15 AM
Life Cycle Assessement (LCA) – A System Approach to Product Environmental Management: *Jinlong Marshall Wang*[1]; [1]The Aluminum Association

Life Cycle Assessment (LCA) is a method for assessing environmental impacts of products and services. It takes a system approach and life cycle thinking on product's environmental impacts and it is extensively used for decision making, learning/exploration, and/or communication purposes. However, this environmental management tool is not only expensive and time consuming, but also has the potential to be manipulated or completely misinterpreted. This article gives a brief introduction to LCA, and some of the key aspects of conducting and interpreting LCA. In particular, it explores the use of LCA as a tool, among other environmental assessment tools, to help promote the sustainability of aluminum among its producers, consumers, and policy makers.

9:35 AM
Beryllium in Dross Produced during Aluminum Melting: *David DeYoung*[1]; Jon Peace[1]; [1]Alcoa Inc

Beryllium has historically been used in various aluminum alloys and is still used today in certain alloys. During the melting of scrap that contains beryllium, some portion of the beryllium reports to the dross; it potentially can even concentrate in the dross. Since dross handling can produce respirable dust, it is important to understand the beryllium content of the dross. A method to analyze beryllium in dross has been developed and was used to measure its distribution between the alloy and the dross. To overcome the inherent non-homogeneous nature of dross the method involved analysis of relatively large sample sizes. This method was used to analyze dross produced from alloys with varying concentrations of beryllium, and the data was then used to model potential beryllium exposure during dross handling.

9:55 AM Break

10:15 AM
Molten Metal Explosions Are Still Occurring: *Seymour Epstein*[1]; [1]Aluminum Association

The Aluminum Association continues to collect reports on explosions occurring in the aluminum industry around the world. Since the program began in 1985 more than 2500 reports have been received and entered into an ongoing database of molten metal incidents. A summary of the incidents will be presented, the causes will be discussed and several recent incidents will be detailed.

10:35 AM
Safety Coatings to Prevent Molten Aluminum-Water Explosions: *Joe Roberts*[1]; Alex Lowery[2]; [1]Pyrotek Inc; [2]Wisechem, LLC

Safety coatings to prevent molten aluminum-water explosions: A brief history and application guides. All the coatings require clean, oil free surfaces and must be mixed and applied properly for adhesion to steel or concrete substrates. There are a variety of ways to clean and apply these coatings

10:55 AM
The Role of Automation in Explosion Prevention in Sheet Ingot Casting: *Denis Bernard*[1]; [1]Rio Tinto Alcan

Over the past decades, sheet ingot production has evolved from completely manual systems using float and diptube and steady-eddy metal level control to more sophisticated automatic systems that have primarily been introduced for improved safety and process consistency. The reliability of these systems have allowed to adopt in specific cases a complete "hands-off" cast start-up procedure which has completely eliminated the need for operators intervention during this critical phase of the cast. These systems have not only reduced the probability of explosion though less process variability during the start-up phase but have also decreased the possibility of injuries that could be caused by a DC explosion.This presentation will highlight the evolution of the use of automation for sheet ingot casting as well as the need to expand the "hands-off" approach to other casting processes such as billet and T ingot casting.

11:15 AM
Benefits to Safety Performance at ALBA from Use of the Wagstaff AutoFlo™ System for Casting of Extrusion Ingot: *Talib Al Ansari*[1]; Hussain Hassan Al Ali[1]; Michael Jacobs[1]; Jalal Mohammed[1]; Mohammed Kadhem[1]; Garry Martin[1]; [1]Aluminium Bahrain (ALBA)

The use of Wagstaff AirSlip® Air Casting Technology for casting of extrusion ingot results in enhancement of the ingot quality for improved extrusion performance. The control of the airflow does require operator attention by manual adjustment during casting. Wagstaff (USA) has developed a new automated gas control system for Airslip tooling called AutoCast™ AutoFlo™ Automated Casting Gas Control. Aluminum Bahrain (ALBA), in the Kingdom of Bahrain, purchased and installed this new control system in 2007 to provide improved consistency in the casting of extrusion ingot and further reduce the risk to operators in the casting area whilst further optimizing production performance and quality of extrusion ingot. This paper details the use of the new automated gas control system from Wagstaff at ALBA and assesses its capability in providing an improved casting environment for enhancement of both operator safety and overall production performance including ingot quality.

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

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

Program Organizers: Toru Okabe, University of Tokyo; Ann Hagni, Geoscience Consultant; Sergio Monteiro, UENF

Tuesday AM Room: 3009
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Donato Firrao, Politecnico Di Torino; Tzong Chen, CANMET-MMSL

8:30 AM
Materials Characterization Analytical Techniques of Minerals, Metals and Materials: *Ann Hagni*[1]; [1]Geoscience Consultant

This paper is an overview of current analytical techniques available for characterization of minerals, metals, and materials, emphasizing chemical techniques, phase identification, and phase quantification techniques. Applications, sample preparation, capabilities, as well as limitations of techniques will be addressed. New developments and advancements over the past few years will be discussed. Practical examples from industry, product development, process improvement, and research and development may be included. This will be an excellent basic primer for the younger scientists, as well as a good refresher with updated information for the experienced materials scientists.

8:45 AM
Development Of Atmosphere-Controlled Mass Spectrometry Equipment: *Takashi Nagai*[1]; Masao Miyake[1]; Masafumi Maeda[1]; [1]The University of Tokyo

Mass spectrometry is a new method to measure thermodynamic properties at high temperature. In this method, vapor pressures of gaseous species in equilibrium with specimen can be measured. Although thermodynamic properties of metals and alloys have been reported by this method, it has not yet been employed for oxides and oxide systems, because of the difficulty in controlling oxygen potential in Knudsen cells. This is true despite this being one of the most important factors in the thermodynamic measurement of oxide systems. Equipment for mass spectrometry which has a mechanism to introduce directly gaseous reactant, such as carbon monoxide and carbon dioxide mixture, to the Knudsen cells to control oxygen potential in the cells has been developed in this study. The change of oxygen potential in the cells following introduction of the reactant was demonstrated, and the thermodynamic properties of oxide systems containing phosphorus, such as calcium phosphate, were investigated.

9:00 AM
Quantitative Phase Analysis of a Dual Phase Steel using Electron Backscatter Diffraction: Jun-Yun Kang[1]; Do Hyun Kim[1]; Sung-Il Baek[1]; Young-Woon Kim[1]; *Kyu Hwan Oh*[1]; Hu-Chul Lee[1]; [1]Seoul National University

Phase differentiation between ferrite and martenstie in a dual phase (DP) steel was examined to automatically assess the volume fraction of each phase using electron backscatter diffraction. A cold rolled and annealed sheet was prepared to have 27% of martensite fraction. As the symmetry in the diffraction pattern could not differentiate the two phases due to the crystallographic similarity, band contrast (BC) value which represents pattern clarity was used. Using grain-averaged value of BC, a reasonable martensite fraction of 25% could be obtained. Concerning the distribution of the grain-averaged BC, there were two peaks detected in the martensite regime, which signified two types of them, lath and plate martensite.

9:15 AM
Defect Analysis using Resonant Ultrasound Spectroscopy: Kevin Flynn[1]; *Miladin Radovic*[1]; [1]Texas A&M University

This paper demonstrates the practicability of using Resonant Ultrasound Spectroscopy (RUS) in combination with Finite Element Analysis (FEA) to determine the size and location of defects in a material of known geometry and physical constants. Defects were analyzed by comparing the actual change in frequency spectrum measured by RUS to the change in frequency spectrum calculated using FEA. Based on the analysis of many FEA-generated frequency spectra, it is possible to develop the model to determine size and position of the defects from measured resonant frequency, and acceptance/rejection criteria for Non-Destructive Testing. Experiments conducted on various materials and geometries show that cracks can be detected by RUS, and their depth and location determined with reasonable accuracy. However, results also indicate that there are limits to the applicability of such a method, the primary one being a lower limit to the size of crack for which this method can be applied.

9:30 AM
Applications Of A Commercially Available BENCHTOP TXRF System: *Alexander Seyfarth*[1]; Hagen Stosnach[1]; [1]Bruker AXS Inc.

Total reflection X-ray fluorescence (TXRF) spectroscopy is a well-established and versatile method for the trace element analysis in solid and liquid samples with manifold matrices. Introduced in March 2008, a new bench top TXRF system, the S2 PICOFOX, offers similar performance than ICP-OES or AA without the lengthy sample preparation and standardization. The analytical range is from ppb to % levels for the elements from Al to U. We will be describing the principle of TXRF, comparing to other methods, including XRF and detailing the fast and convenient sample preparation. We will show studies done by the application laboratory and customers ranging from the analysis of nano materials to exploration screening for metals. This new implementation enables the chemical laboratory to finally take advantage of TXRF.

9:45 AM
Non-Destructive Analysis Of Dislocations In Bulk Samples Using ECCI: *Martin Crimp*[1]; [1]Michigan State University

Electron channeling contrast imaging (ECCI) allows near surface dislocations and other crystal defects to be imaged and characterized with high spatial resolution using a field emission gun SEM. Using this approach, dislocations may be assessed using varying electron channeling conditions, allowing the crystallographic details to be characterized in a manner similar to that carried out with diffraction contrast TEM. Because the dislocations are imaged in bulk samples, ECCI has a number of advantages over other approaches for imaging defects including allowing for analysis over large areas/volumes, being non-destructive in many applications, and being very conducive to in-situ testing. This talk will review the fundamental issues and experimental parameters involved with imaging dislocations using channeling contrast. Case studies will be presented that illustrate the flexibility of ECCI for assessing dislocation structures and morphologies in both metallic and semiconductor materials.

10:00 AM Break

10:20 AM
Identification of Corrosion Product on Corroded Rebar in Concrete: *Jian Li*[1]; Gordon Gu[1]; Valery Guertsman[1]; Pei Liu[1]; [1]CANMET-Materials Technology Laboratory

Corrosion resistance of materials is highly dependent on the microstructure of the specific material. Apart from general corrosion rate measurement using techniques like linear polarization and A.C. impedance, microstructural investigations are frequently needed to identify the root cause of corrosion. Advanced techniques, including scanning electron microscopy (SEM) and transmission electron microscopy (TEM), have been well integrated into routine characterization studies. Other microscopy techniques, including electron probe micro-analyzer (EPMA), Auger, X-ray photon spectroscopy (XPS) and secondary ion mass spectroscopy (SIMS), can provide detailed chemistry information on corrosion products. In recent years, focused-ion beam (FIB) microscopes have evolved into an important microstructural characterization instrument. In this study, corrosion of rebar encased in concrete was studied in great detail using advanced microscopy techniques. Corrosion product on the rebar surface was identified as ferrous oxide with relatively large crystal size. The morphology of this passive layer was thoroughly analyzed, and its formation mechanism is proposed.

10:35 AM
Linear Measures for 3-D Microstructures: *Martin Glicksman*[1]; Paulo Rios[2]; Daniel Lewis[3]; [1]University of Florida; [2]Universidade Federal Fluminense; [3]Rensselaer Polytechnic Institute

Linear microstructure measures, including the average caliper, C, and mean width, L, may be employed to characterize the geometric properties of polyhedral grains, including their areas, volumes, and face curvatures. Moreover, linear measures permit prediction of curvature-mediated grain growth rates, thereby providing an important connection between kinetic behavior, grain size, and shape. The authors' development of regular polyhedra to represent each topological class of grains found in well-annealed polycrystals was used to calculate exact C and L values. The behavior of these linear measures for polyhedra with different face numbers and shapes will be discussed. Limitations were found, including a surprising insensitivity to grain shape that might limit the use of linear measures for practical characterization of 3-D microstructures. Alternative predictors of kinetic behavior for annealed polycrystals will be proposed.

10:50 AM
Particle Size And Shape Analysis With CILAS Instruments: *Nicolas Marchet*[1]; [1]CILAS

The particle size determination using Laser Diffraction is becoming the most popular instrument to analyze polymers, ceramics or metallic particles in chemistry, pharmaceutical or building applications. CILAS particle size analyzers permit to measure particle size distributions by three fast ways, with laser diffraction in wet and dry mode and with the optical microscopy thanks to the shape analysis option. This last instrument calculates more than twenty shape factors with the Expert Shape Software which permit to characterize the morphology of particles in order to give important information about the quality process, physical and chemical powder properties. This presentation gives some examples of applications using CILAS instruments in particle size and shape characterizations. The ways of particle characterization are according to the needs of each user and each material and present a good accuracy and repeatability in agreement with the ISO standard.

11:05 AM
Two- and Three-Dimensional Analyses of Martensitic Steels: *George Spanos*[1]; David Rowenhorst[1]; Jerry Feng[1]; Keith Knipling[1]; Rick Everett[1]; Gregory Olson[2]; Stephanie Chan[2]; [1]Naval Research Laboratory; [2]Northwestern University

This presentation outlines progress made on both two dimensional (2D) and three dimensional (3D) studies of martensitic steels. The first part of this talk will be centered about 2D studies of martensite in low carbon steels, including HSLA-100 and a high (10%) Ni steel. In particular, findings will be presented on a 2D quantification scheme of lath martensite structures based on automated analyses of Electron Backscatter Diffraction (EBSD) scans. The second part of the talk will focus on 3D analyses of martensitic steels. This will include progress on two 3D reconstruction efforts, including a project which utilizes the dual beam Focused Ion Beam and EBSD to study martensite in low carbon steels, and an investigation of cracks and voids in a titanium modified 4330 steel. The latter study employs both serial sectioning and X-ray tomography techniques.

11:20 AM
Analysis Of Microstructure Evolution During Cold Deformation Of Air-Hardening Steel LH800: *Olexandr Grydin*[1]; [1]Leibniz University of Hanover

The evolution of grain and dislocation structures during cold deformation of the new steel LH800 of Salzgitter AG is investigated in the frame of this work. The main feature of this material is its ability to air-harden by carbon content of about 0.1%. The initial ferrite structure of the metal provides high plasticity at cold deformation and subsequent heat treatment increases tensile strength approximately two times. Described are results of in-situ tensile tests in the SEM. SEM and TEM analysis of grain and dislocation structures evolution in samples, which have been uniaxially deformed to certain strains, are carried out. After statistical evaluation of results the grain elongation against strain is determined. A microstructure of a deep drawn cup is analyzed by means of SEM. A quantitative evaluation of grain elongation on the different cup zones and comparison of these data with prediction results based on tensile tests are carried out.

11:35 AM
Comparison of Phase Identification of EBSD and TEM in TRIP-Aided Steel: *Sung Il Baik*[1]; Jun-Yeun Kang[1]; Do-Hyeon Kim[1]; Kyu-Hwan Oh[1]; Hu-Chul Lee[1]; Young Woon Kim[1]; [1]Seoul National University

Electron Back-Scattered Diffraction (EBSD) is a convenient and powerful tool to identify the phases and orientations in multiphase steel. Transmission Electron Microscopy (TEM), on the other hand, provides an accurate determination of phases even with the drawbacks of small area of observation. Direct comparisons of phase distributions were made from the Transformation Induced Plasticity (TRIP)-aided steel, which is consisted of ferrite, bainite and retained austenite. Unlike ferrite and martensite, bainite was known as a challenging phase to identify in EBSD. Phase mapping was made using EBSD and then the individual phases were identified using TEM in same region of the sample. Discrepancies in phase identification were observed in the region containing bainite, grains with low-angle grain boundaries, and small grains with less than 3µm diameter.

Computational Thermodynamics and Kinetics: Functional Materials

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, ASM Materials Science Critical Technology Sector, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Long Qing Chen, Pennsylvania State University; Yunzhi Wang, Ohio State University; Pascal Bellon, University of Illinois at Urbana-Champaign; Yongmei Jin, Texas A&M University

Tuesday AM — Room: 3002
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Long Qing Chen, Pennsylvania State University

8:30 AM Invited
Anhysteretic Response of Compositionally Heterogeneous Alloys with Displacive Phase Transformations: *Armen Khachaturyan*[1]; Yong Ni[1]; [1]Rutgers University

According to the phase rule, any diffusionless (displacive) transformation of a homogeneous solid solution is a metastable reaction developing within a two-phase field of the equilibrium phase diagram. Therefore, a sufficiently long annealing near the diffusionless transformation line should always result in a partial decomposition that eventually produces a compositionally heterogeneous state. This decomposition and displacive transformation in such a system are considered. In particular, we discuss a strain response to the applied stress/electric/magnetic fields in the martensitic/ferroelectric/ferromagnetic systems. It will be shown that in all these cases the field-induced displacive transformation and/or reorientation of structural domains of the low symmetry phase generates the macroscopic deformation. Given the energetic preference of the initial structural state, a removal of the external field can result in a recovering of the initial undeformed state, which means that this deformation is a pseudo-elastic recoverable strain. The pseudo-elastic response of the body can be an anhysteretic (or slightly hysteretic). If the applied field is stress, the response is a pseudo-elastic effect accompanied by the extrinsic softening of the shear modulus. Application of the theory and modeling results to the ferroelectric solid solution with high piezoelectric response and to the Fe-Ga alloys with a giant magnetostriction is discussed.

9:00 AM Invited
Domain Microstructures and Mechanisms in Morphotropic Phase Boundary Ferroelectrics: Phase Field Model and Simulation: Wei-Feng Rao[1]; *Yu Wang*[1]; [1]Virginia Tech

Phase field model is employed to study underlying domain microstructures and mechanisms responsible for enhanced electromechanical properties near morphotropic phase boundaries (MPBs) in ferroelectric solid solutions. This talk will present some new insights gained into the phase-coexisting domain microstructures and field-induced inter-ferroelectric phase transformations and their relations to the advanced piezoelectric properties around MPB. The modeling and simulation show that extrinsic domain mechanisms play dominant roles in the strong piezoelectricity around MPBs, and crystallographic domain engineering is effective to fully exploit the domain mechanisms for property enhancement. Together with crystallographic and diffraction analyses, the study reveals a nanodomain perspective of MPB ferroelectrics, where coherent scattering and interference effects produce an adaptive diffraction phenomenon, which is peculiar to nanodomain microstructures.

9:30 AM

Grain Boundaries-Ferroelectric Domains Interactions in Polycrystalline Ferroelectrics: Eva Anton[1]; *R. Edwin Garcia*[2]; John Blendell[2]; Keith Bowman[2]; [1]Darmstadt TU; [2]Purdue University

Ferroelectric Lead Zirconate Titanate (PZT) films display physical behavior that makes them an important candidate for random access memory applications. In such devices, ferroelectric domains are locally switched by the application of an electric field, thus fixating the state of a memory unit. Today's technological advancement, however, demands ever higher memory densities. Therefore, as the device size shrinks, the microstructural features become increasingly important and the spatial variation of the hysteretic behavior increases, making the memory unit potentially unreliable. The local crystallographic orientation and the local grain-grain interactions play an important role in determining the switching of domains. In particular, large spatial variations of the fields arise as a combined result of the stresses that develop due to the thermal expansion and lattice mismatch of the film-substrate system, the anisotropy of the properties of the involved materials, and the processing conditions.

9:50 AM

Phase-Field Simulation of Domain Stabilities and Structures in Strained SrTiO3 Thin Films: *Guang Sheng*[1]; Yulan Li[1]; Jingxian Zhang[1]; Samrat Choudhury[1]; Darrell Scholom[1]; Quanxi Jia[2]; Zi-Kui Liu[1]; Long-Qing Chen[1]; [1]Pennsylvania State University [2]Los Alamos National Laboratory

Strontium titanate (SrTiO3) is known as a classical example of a system with coupled structural and incipient ferroelectric instabilities. In this study, the antiferrodistortive transition and ferroelectric transition in a strained (100) SrTiO3 thin film are analyzed using phase-field approach. Based on the simulation results, the misfit strain-temperature domain stability diagrams, graphical representation of stable ferroelectric and structural domain structures as a function of strains and temperature, are constructed. The misfit strain-misfit strain domain stability diagrams at several representative temperatures were also generated, and the corresponding domain structures were analyzed and compared with experimental studies. By taking into account the different domain structures obtained from the variations of Landau coefficients used in the simulation, it is expected that such diagrams will provide guidance for interpreting experimental measurements and observations as well as to the design of SrTiO3 films with specified domain structures.

10:10 AM Invited

Defects and Domain Walls in LiNbO3: Insights from Microscopic Simulation: Haixuan Xu[1]; Donghwa Lee[1]; Jun He[1]; Venkatraman Gopalan[2]; Volkmar Dierolf[3]; Susan Sinnott[1]; *Simon Phillpot*[1]; [1]University of Florida; [2]Pennsylvania State University; [3]Lehigh University

We use electronic-structure, density functional theory calculations integrated with thermodynamic calculations to determine the structure and stability of point defects and point defect clusters in LiNbO3. In particular, we identify the dominant defects at different temperatures, oxygen partial pressure, and compositions. In addition, we use classical molecular-dynamics simulation approaches to characterize the structure and energetic of domain walls in LiNbO3. A discussion of the interaction of domain walls and point defects is presented. This work is supported by the National Science Foundation under awards DMR-0602986 and DMR-0303279.

10:40 AM Invited

Phase-Field Modeling of Defect Interactions in Active Materials: *Chad Landis*[1]; [1]The University of Texas at Austin

A continuum thermodynamics framework is presented to model the evolution of domain structures in active/smart materials. In a departure from previous derivations of the phase-field equations, a set of micro-forces and governing balance laws are postulated and applied within the second law of thermodynamics to identify the appropriate material constitutive relationships. To investigate the consequences of the theories, fundamental defect interactions are studied. A principle of virtual work is specified for the theory and is implemented to devise a finite element formulation. For ferroelectrics, the theory and numerical methods are used to investigate the interactions of 180º and 90º domain walls with arrays of charged defects and dislocations to determine how strongly domain walls are electromechanically pinned by the arrays of defects. For ferromagnetic shape memory alloys the interaction between a martensite twin boundary and magnetic domain wall is modeled to explain the finite blocking stress in these materials.

11:10 AM Invited

Microstructure Evolution of Ferromagnetic Shape Memory Alloys: *Jiangyu Li*[1]; [1]University of Washington

Magnetoelastic domains in ferromagnetic shape memory alloys (FSMA) evolve through either variant rearrangement or magnetization rotation, resulting in large or small magnetic field-induced strain depending on the magnitude of applied compressive stress. A mesoscopic theory is developed to study the magnetoelastic behavior of FSMA to account for both variants rearrangement and magnetization rotation. A multi-rank laminated domain configuration is constructed first under the constrained theory, which is then relaxed by allowing the magnetization to rotate away from its easy axis, resulting in incompatibility in both magnetization and magnetostrictive strain. It is observed that microstructure evolution of FSMA is dominated by rearrangement of variants when the applied stress is small, but such rearrangement is blocked when the applied stress is relatively large, under which magnetization rotation takes over as the dominant mechanism. A novel phase-field simulation is also carried out to verify the theoretical analysis.

11:40 AM

Phase Field Simulation of Coupled Twin Boundary and Domain Wall Motions in Magnetic Shape Memory Alloys: *Yongmei Jin*[1]; [1]Texas A & M University

Magnetic field-induced deformation in magnetic shape memory alloys (MSMAs) results from coupled ferromagnetic and ferroelastic domain evolutions. The coupling occurs through elastostatic and magnetostatic interactions as well as magnetocrystalline anisotropy, and is investigated by computer modeling and simulation. It reveals that the motions of twin boundaries and domain walls depend not only on external magnetic fields but also on internal domain configurations, leading to complex domain processes. It is demonstrated that twin boundary can continue its motion under decreasing magnetic field, or even reverse motion direction without changing magnetic field, producing peculiar magnetomechanical behaviors. Based on the simulations, domain microstructure-dependent driving forces for the coupled motions of martensite twin boundaries and magnetic domain walls in magnetic shape memory alloys are analyzed.

12:00 PM

Phase Field Modeling of the Martensitic Transition: i) Comparison between Geometrically Linear and Non-Linear Elasticity, and ii) Microstructures in Ni-Ti-Pd Alloys with Special Lattice Parameters: *Alphonse Finel*[1]; Umut Salman[2]; [1]ONERA; [2]CNRS

Martensitic transformations are characterized by large strain misfits between the martensite and the austenite, and also between the different orientational variants of the martensitic phase. The transitions are often athermal and microstructures dictated by strain accommodation and thermoelastic equilibrium. We analyze these microstructures using a Phase Field method that incorporates kinetic energy and a Ginzburg-Landau modeling of the elastic energy. We first discuss the differences between a geometrically non-linear (i.e. invariant by rotation, and thus exact) and the often-used linear (i.e. approximate) form of this elastic energy and show that the metastable states differ considerably between the two models. We also compare our numerical results to experimental observations. Finally, we present briefly an investigation of the martensitic microstructures in Ni-Ti-Pd alloys with special lattice parameters.

CO_2 Reduction Metallurgy 2009: Mechanisms and Electrolysis

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

Tuesday AM   Room: 2012
February 17, 2009   Location: Moscone West Convention Center

Session Chairs: Ramana Reddy, The University of Alabama; Jiann-Yang Hwang, Michigan Technological University

8:30 AM Introductory Comments

8:35 AM Invited
Metal Cations in CO2 Assimilation and Conversion by Plants: *Sergey Shabala*[1]; [1]University of Tasmania

Green leaf tissues convert solar energy into the energy of chemical bonds of sugar molecules during the process of photosynthesis. The efficiency of this conversion is at least twice higher then efficiency of any of currently known silicon-based solar panels. Importantly, vast amounts of CO2 are assimilated during this process. The efficiency of photosynthesis is critically dependent on the availability of a large number of nutrients, among which metal cations such as K, Ca, Mg, Cu, Zn, Fe, Mn, and Ni play a key role. In this talk I will summarize basic requirements and major functions for each of these essential nutrients in plant photosynthesis, both at the whole-plant and molecular level. I will talk about how these requirements may be affected by the global climate trends and discuss the prospects of creating artificial photosynthetic "bioreactors" for efficient energy conversion and CO2 assimilation.

9:05 AM
CO2 Mitigation in Metallurgical Processes Using Concentrated Solar Energy: *Aldo Steinfeld*[1]; [1]ETH Zurich

Solar thermochemical processes make use of concentrated solar radiation as the energy source of high-temperature process heat. Considered are the thermal and carbothermal reduction of metal oxides for the production of metals, metal nitrides, and metal carbides. The research work encompasses fundamental studies on chemical reactor engineering, with emphasis on the analysis of radiative heat transfer in reacting particle flows. Solar reactor prototypes are designed, fabricated, modeled, and tested in a high-flux solar furnace, further optimized for maximum solar-to-chemical energy conversion efficiency, and finally scaled-up for (MW) industrial application using concentrating solar towers.

9:25 AM
Aluminum Industry and Climate Change — Assessment and Responses: *Subodh Das*[1]; John Green[2]; [1]Phinix LLC; [2]Secat Inc

The aluminum industry is a latecomer to the suite of industrial metals.This paper assesses the impact of the global aluminum industry on climate changes. Subsequently, this paper also suggests several proactive strategies in the broad areas of production, application, recycling and carbon trading to minimize the impact.

9:45 AM
Effect of Electrode Physical Properties on Dendritic Deposition of Al Using EMIC-$AlCl_3$ Ionic Liquid Electrolytes: *Debabrata Pradhan*[1]; Ramana Reddy[1]; [1]The University of Alabama

Electrorefining of aluminum scrap was investigated from 1-Ethyl-3-methyl-imidazolium chloride (EMIC)-$AlCl_3$ (60 wt%) electrolyte using copper/aluminum cathodes at 90 ± 3°C. The deposits were characterized using scanning electron microscope (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD). The study was focused to determine the effect of electrode surface modifications, anode and cathode materials, surface roughness of electrodes and deposition time on dendritic deposition of aluminum. Also, their effect on current density was investigated. It was shown that the surface modification of electrodes reduced the dendritic deposition of aluminum at higher overpotentials. Pure aluminum (>99%) was deposited for all experiments with current efficiency of 95-99%.

10:05 AM
Room-Temperature Production of Ethylene from Carbon Dioxide: *Kotaro Ogura*[1]; [1]Yamaguchi University

Ethylene has been produced in aqueous solution from CO_2 by the electrochemical reduction driven by a natural energy. This process is useful for storing a large amount of the natural energy. In the closed system, the conversion efficiency of CO_2 is almost 100%, and the maximum selectivity for the formation of ethylene is more than 70%. On the other hand, the current efficiency for the competitive reduction of water is less than 10%. The electrolysis is practicable under such special coditions as three-phase interface consisting of gas, solution and metal, concentrated solution of potassium halide, low pH and copper or copper halide-confined metal electrode. These conditions are thoroughly examined, and the grounds to reply upon are revealed. A series of chemical apparatuses including an electrolytic cell in a large scale are designed for the ethylene production, which allow us continuously to supply raw CO_2 and to extract the product.

10:25 AM Break

10:40 AM
Electrochemical Reduction of Carbon Dioxide in Ionic Liquids: *Huimin Lu*[1]; Xiaoxiang Zhang[1]; Pengkai Wang[1]; [1]Beijing University of Aeronautics and Astronautics

In this paper, the authors studied an electrochemical reduction process of carbon dioxide in ionic liquids such as 1-n-butyl-3-methylimidazolium hexafluorophosphate (BmimPF6) as the electrolytes. The electrolysis experiments were carried out under current and potential controls. The cathode products contained carbon nanotubes, carbon nanofibers, nanographites, and amorphous carbon. To establish the actual current and potential ranges, the electroreduction of carbon dioxide dissolved in the ionic liquid was studied by cyclic voltammetry on glass-carbon (GC) electrode at a temperature range from 100 to 145°C. The electrochemical mechanism of carbon dioxide electroreduction was studied for explanation of all obtained results. As the last ting, Carbon dioxide in ionic liquids was electroreduced as metals oxides were electroreduced in molten salts.

11:00 AM
Formation of Tetra Ethyl Ammonium Formate by Electro Reduction of Carbon Dioxide in a Membrane Cell: Kandasamy Subramanian[1]; K.G. Gomathi[1]; *Krishnasamy Asokan*[1]; [1]Central Electrochemical Research Institute

Composite perfluoro sulphonic acid/carboxyalte cation exchange membrane fitted electrochemical flow cell was designed and operated. The carbon dioxide absorbed in phosphate buffer solution was flowed as catholyte. Tetra ethyl ammonium bromide (TEAB) solution was fed to the anode compartment. Tetra ethyl Ammonium formate (TEAF) is formed in cathode compartment. Tetra ethyl ammonium tribromide (TEATB) was formed at the anode. Optimum values of flow rate and current density were evaluated for the formation of Tetra ethyl ammonium formate KEY WORDS: carbon dioxide, Tetra ethyl ammonium formate, Posphate buffer, flow cell, Nafion 961, Iridium oxide, Lead-Tin cathode.

11:20 AM
Silicon Dioxide As A Solid Store For CO_2 Gas: *Victor Zavodinsky*[1]; Sergey Rogov[1]; [1]Institute for Materials Science

Pseudopotential fully relaxed total energy calculations are used to predict a hypothetical $Si_{1-x}C_xO_2$ (x<0.5) compound formed from SiO_2 β-cristobalite by substitution of some SiO_2 complexes by CO_2 molecules. The simulation shows that the $Si_{1-x}C_xO_2$ compound can be quasi stable if the CO_2 content is less than fifty per cent. It is assumed that six-molecule $Si_{6-n}C_nO_{12}$ (n=3) rings can play a role of nucleuses for formation of the $Si_{1-x}C_xO_2$ compound from SiO_2 and CO_2 molecules. Thus, silicon dioxide can be considered as a possible solid store for gaseous CO_2.

11:40 AM
Recent Developments in Carbon Dioxide Capture Materials and Processes for Energy Industry: *Malti Goel*[1]; [1]Former Advisor and Senior Scientist, Ministry of Science and Technology

The cost-effective capture of CO2 from the point sources for its reduction in the atmosphere offers many challenges in materials science. Novel CO2 capturing approaches using chemical, physical and biological methods are in the research stage and are aimed to minimize the cost. Appropriate materials development which can withstand required temperature or pressure as the case may be for CO2 emanating from coal gas or industrial waste gases form the minimum condition.

Other requirements are reclyclability of material and cost of separation. Nano-material composites can be more effective in selective capture of CO2 and can offer solutions for large-scale separation process. Nano-porous material catalysis can enhance the reaction rate of CO2 with other chemicals and thus help in faster removal of CO2. This paper reviews recent industrial scale developments. In the Indian context, R&D priority areas in CO2 capture process development with a focus on energy industry are presented.

Diffusion in Materials for Energy Technologies: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Nuclear Materials Committee, TMS: Solidification Committee, ASM-MSCTS: Atomic Transport Committee
Program Organizers: Jeffrey LaCombe, University of Nevada, Reno; Yongho Sohn, University of Central Florida; Carelyn Campbell, National Institute of Standards and Technology Afina Lupulescu, GE; Ji-Cheng Zhao, Ohio State Universityersity

Tuesday AM — Room: 3006
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Jeffrey LaCombe, University of Nevada, Reno; Dennis Keiser, Idaho National Laboratory; Afina Lupulescu, GE

8:30 AM Invited
Computation of Diffusion Coefficients: Current Capabilities and Perspectives: *Erich Wimmer*[1]; Clive Freeman[1]; Hannes Schweiger; Walter Wolf[1]; Paul Saxe[1]; [1]Materials Design, Inc.

Fueled by the remarkable progress in hardware and software, computational materials science based on first-principles quantum mechanics is now becoming an integral part of industrial engineering. The present contribution focuses on diffusion processes, which are of particular importance for the performance and aging of materials for energy technologies. As illustrative example the diffusion of hydrogen isotopes in transition metals demonstrates that temperature-dependent diffusion coefficients can be computed from first-principles reaching an accuracy which is comparable with experiment. In the near future we can anticipate a dramatic growth of compute power in terms of number of processors. This will provide a fascinating opportunity to explore diffusion mechanisms in complex systems such as nano-structured and composite materials by carrying out thousands of simultaneous calculations. To harness this power, we will need highly automated and extremely robust computational schemes as well as sophisticated methods to cope with the wealth of data.

9:05 AM Invited
Composition Dependent Diffusion Coefficients From First Principles: *Anton Van der Ven*[1]; [1]University of Michigan

Diffusion in both interstitial and substitutional alloys is a complex kinetic process that depends on the nature of intrinsic defects, the energetically most favorable hop mechanisms and the degree of short and long-range order among the constituents of the alloy. In this talk, I will describe how these factors can be rigorously accounted for in the first-principles prediction of diffusion coefficients in non-dilute alloys. The approach relies on the evaluation of Kubo-Green expressions, which provide the link between macroscopic diffusion coefficients and atomic trajectories sampled in kinetic Monte Carlo simulations. A first-principles description of the thermodynamics of short and long-range order in multi-component solids is achieved with the cluster expansion formalism. As examples, I will describe recent work on the prediction of diffusion coefficients in the B2-NiAl compound used as bond coat in turbine blades and in Li ion battery electrode materials.

9:40 AM Invited
New Paradigm in Developing Atomic Mobility Databases: *Zi-Kui Liu*[1]; [1]Pennsylvania State University

Atomic diffusion is a common and important non-equilibrium process in solids that takes place at finite temperatures. To computationally simulate atomic diffusion processes, the thermodynamic and atomic mobility databases of the materials of interest are needed. The modeling technique of atomic mobility databases and related software has been becoming more and more matured in the last decades. However, the input data for the modeling is exclusively taken from experimentally measured tracer and chemical diffusion coefficients. In this presentation, a new modeling paradigm is presented which integrates quantum mechanics calculations, statistic analysis, and phenomenological modeling. Firstly, our recent progress in predicting self and dilute diffusion coefficients by quantum mechanics calculations will be discussed. Secondly, our approach to the unstable vibrational mode of transition states during diffusion will be outlined. Finally, the contribution to phenomenological modeling of atomic mobility will be presented.

10:15 AM Break

10:25 AM Invited
Mechano-Chemistry, Foundations And Modeling: *Marek Danielewski*[1]; Bartek Wierzba[1]; Jolanta Janczak[2]; Magdalena Pawelkiewicz[1]; [1]AGH University of Science and Technology; [2]EMPA

The volume continuity equation is used to define the material frame of reference in the multicomponent alloys. It allows to omit Darken postulate of constant molar volume, extends his method, defines frame of reference for diffusion and allows using Navier-Lamé equation of mechanics. Proposed form of conservation equations is self-consistent with the literature from classical Kirkendall experiments and their interpretation by Darken, Shimozaki and Onishi. The method allows for phenomenological description of multiscale phenomena and opens vast number of entirely new possibilities. We will show four series of Ni-Cu-Ag-Sn quaternary diffusion multiples of various geometries and distributions of elements in 3D fragments. Comparison of experimental and modeling results are reviewed. The new software and interpretation of experimental results will be presented.

11:00 AM Invited
Modelling of Oxidation and Creep Resistance in Fe-Cr High-Temperature Steels: *John Agren*[1]; Samuel Hallström[1]; Johan Jeppsson[1]; Lars Höglund[1]; [1]Royal Institute of Technology

Modelling, based on oxidation controlled by bulk and grain boundary diffusion through the alloyed oxide on the steel surface, is presented. The model for volume diffusion is based on a vacancy mechanism and mobilities and thermodynamic factors assessed by means of the Calphad type of analysis. The model is implemented in the DICTRA software and allows the calculation of oxide growth controlled by diffusion of metal ions as well as oxygen ions. The effect of porosity caused by a Kirkendall effect in oxides is discussed. The creep resistance is modeled by considering dislocation mechanisms that involve particle strengthening and solution hardening. The evolution of precipitate structure is predicted by kinetic calculations involving diffusion controlled phenomena such as growth and coarsening and also dissolution of less stable phases. Comparisons with experimental data are presented.

11:35 AM
First-Principles Calculation on Impurity Diffusivities in Ferritic Iron: *Shenyan Huang*[1]; Daniel Worthington[2]; Mark Asta[3]; Peter Liaw[1]; [1]University of Tennessee, Knoxville; [2]University of Texas, Austin; [3]University of California, Davis

To assist the alloy design for a creep-resistant ferritic Fe-based superalloy useful up to 1,033K, first-principles calculations have been applied to compute impurity diffusivities in the ferritic iron. To augment existing kinetic databases, which lack experimental measurements for a number of 4d and 5d solutes, diffusivities have been derived by incorporating first-principles calculated jump rate probabilities into a generalized five frequency model of vacancy mediated diffusion in the dilute limit. Using a transition-matrix approach with input parameters derived from the first-principles calculations, the correlation factors for solutes in ferritic iron have been determined. First-principles calculations were also conducted to calculate the induced magnetization of the impurity in the first and second neighbors, and thus, to investigate the relation between activation energies in ferromagnetic and paramagnetic states, employing an established empirical relation. This project is acknowledged by the Department of Energy Office of Fossil Energy Program, with Dr. Patricia Rawls.

11:55 AM
Isotopic Diffusion Studies in Mg-rich Light Metal Alloy Systems: *Nagraj Kulkarni*[1]; Peter Todd[2]; Yongho Sohn[3]; [1]University of Tennessee; [2]Oak Ridge National Laboratory; [3]University of Central Florida

The development of an Integrated Computational Materials Engineering (ICME) framework for Mg-based light-weight alloys required for next-generation automotive materials will require a reliable diffusion database that can be integrated with other modeling activities. In this study, we focus on SIMS-based tracer diffusion studies in the Mg-Al-Mn system that are carried out using stable isotopes. The procedures and challenges involved in such studies will be discussed and preliminary results will be presented. Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Vehicle Technologies, as part of the Automotive Lightweighting Materials Program, under contract DE-AC05-00OR22725 UT-Battelle, LLC.

12:15 PM
Variational Approach to the Boltzmann Matano Methods for Determination of the Diffusivity Coefficient.: *Alonso Jaques*[1]; Jeffrey LaCombe[1]; [1]University of Nevada

The Boltzmann-Matano method is used extensively in the determination of diffusivities in alloys. In the course of analyzing experimental data to determine D(C), numerical integrations and differentiations of the concentration profile are performed. With experimental data containing point-to-point noise, there are challenges related to calculating the slope (for example). Therefore, smoothing of the experimental data is often performed prior to analysis. This step can introduce numerical "artifacts" into the data and affects the confidence in the estimated parameters.We present here, an approach to the Boltzmann-Matano method that is based on a variational formulation for the numerical operations performed on the concentration data, avoiding the necessity of smoothing the data beforehand. This approach therefore, has the potential to be less subjective, and in numerical simulations, shows an increased accuracy in the estimated diffusion coefficients. The analysis method and accompanying publically-available analysis software used for this analysis will be discussed.

Dislocations: 75 Years of Deformation Mechanisms: Effects of Obstacles, Surfaces, and Scale on Dislocation Generation and Motion

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: David Bahr, Washington State University Erica Lilleodden, GKSS Research Center; Judy Schneider, Mississippi State University Neville Moody, Sandia National Laboratories

Tuesday AM — Room: 3022
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

8:30 AM Invited
Method for Determining Dislocation Viscous Drag Coefficients: *John Gilman*[1]; [1]University of California

In imperfect crystals, dislocation motion is of the stick/slip type so fundamental viscosity coefficients are difficult to measure. However, the maximum velocities are determined by a balance between the driving stress, and the drag. The driving stress is limited by cohesion so the drag can be determined at the terminal velocity where the motion is of the slip type alone. Fortunately, there is a reliable equation for extrapolating from intermediate velocities to the terminal velocity. Given the terminal velocity, an expression without disposable parameters yields the viscosity coefficient. The latter can be compared with values measured directly at low stresses, or derived from internal friction measurements. This method will allow values for a wide range of materials to be made and a library to be constructed that will be useful for fundamental studies.

9:00 AM
The Role of Solute Segregation on the Evolution and Strength of Dislocation Junctions: *Bulent Biner*[1]; Q. Chen[1]; X. Y. Liu[2]; [1]Ames Laboratory (USDOE); [2]Los Alamos National Laboratory

In this study, the role of solute segregation on the strength and the evolution behavior of dislocation junctions is studied by utilizing kinetic Monte Carlo and 3D dislocation dynamics simulations. The different solute concentrations and the character of the junctions are all included in the simulations in an effort to make a parametric investigation. The results indicate that the solutes have a profound effect on the strength of the junctions. Solute segregation can lead to both strengthening and weakening behavior depending upon the evolution of the dislocation junctions. The local solute concentration seems to be the more relevant parameter to characterizing the solute and dislocation interactions, due to the short-range stress field of solutes; and its bounds are set by the unconstrained volume dilatation. *This work at the Ames Laboratory was supported by the Department of Energy-Basic Energy Sciences under Contract No. DE-AC0207CH11358.

9:20 AM
On The Origin Of Plastic Instability Of Al-Mg Alloy 5052 During Stress Rate Change Test: *Chen-ming Kuo*[1]; Chi-Ho Tso[1]; [1]I-Shou University

Plastic instability or Portevin-Le Chetelier effect is observed during stress rate change test of Al-Mg alloy 5052 at room temperature. In the stress rate change experiments, strain retardation and plastic instability are observed, that is, although the applied stress rate changes, plastic strain is insignificant until the plastic instability occurs. By slightly increasing the final stress level, plastic instability is observed and modeled by the typical plastic deformation mechanism, that is, thermally activated kinetic flow theory coupled with structural evolution law. By changing the values of suitable parameters to simulate the microstructure change of instability, the origin of plastic instability could be understood.

9:40 AM
Serrated Flow and the Portevin-LeChatelier Effect in Austentic Steel with Twinning Induced Plasticity: *Louis Hector*[1]; Pablo Zavattieri[1]; Vesna Savic[2]; James Fekete[2]; [1]General Motors R&D Center; [2]General Motors Corporation

The twinning induced plasticity (TWIP) effect in high manganese austenitc steels leads to extreme strain hardening and elongation. Twinning helps retain the austenitic structure and twin boundaries act as barriers to dislocation motion. True stress-true strain curves exhibit step-like serrations beyond a critical strain suggesting the Portevin-LeChatelier (PLC) effect and negative strain rate sensitivity. Here, PLC band nucleation and propagation in TWIP steel were investigated with a digital image correlation (DIC) technique. Images of one surface of a tensile specimen were recorded with a variable framing rate high speed digital camera and custom image acquisition software. Post-processing of the data resulted in color strain and strain rate contour maps. Band nucleation, the direction of band propagation, and strain accumulation in the wakes of the bands were explored in the vicinity of individual serrations in flow curves. The present results are qualitatively compared with the PLC effect in Al-Mg alloys.

10:00 AM
Twinning Dislocations and Twin/Matrix Interfacial Structure in HCP Metals: *Bin Li*[1]; Evan Ma[1]; [1]Johns Hopkins University

The double-layered structure of the twinning planes of HCP metals makes the twinning processes in HCP metals distinctly different from those in high-symmetry metals where the twinning plane is also the slip plane for dislocations. While a previous study suggests that a combination of <c+a> and <a> dislocations that spreads over a number of twinning planes (a zonal dislocation) can be the source of a twin embryo, we show that the actual configuration of the twinning dislocations is controlled by the energetics at the twinning plane. Instead of bonding two single crystals in the twinning orientation and then relaxing the twins, we investigate twin/matrix interface structure during deformation twinning in magnesium, using molecular dynamics and a simulation scheme different from previous studies. Valuable information regarding the configuration of the twinning dislocations and the twin/matrix interfacial structure is obtained.

10:20 AM Break

10:40 AM Invited
Dislocation Micromechanisms and Scale-Free Flow in Microcrystals: *Dennis Dimiduk*[1]; Christopher Woodward[1]; Paul Shade[2]; Michael Uchic[1]; Satish Rao[3]; Ed Nadgorny[4]; [1]US Air Force Research Laboratory; [2]Ohio State University [3]UES, Inc.; [4]Michigan Technological University

Recent evidence shows that dislocation plasticity of crystals exhibits scale-free flow avalanches. However, there are relatively few experimental studies that reveal the dislocation mechanisms governing such behavior. Simulation studies suggest that long-range interactions between the ensemble of dislocations leads to their intermittent collective motion and the observed power law avalanche statistics. Those observations appear to be supported by slip step-height statistics and, indirect acoustic-emission experiments have been interpreted in a similar way. The present study examined the flow behavior of a variety of micrometer-sized single crystals deformed at room temperature via compression testing. The avalanche statistics have been examined as a function of material type and assessments of expected dislocation micromechanisms. The results of the study are discussed with attention to the nature of avalanches, their maximum size cut-off and the size-affected flow stress observed for such crystals.

11:10 AM
In-Situ Mechanical Testing At The Micro-Scale: *Paul Shade*[1]; Robert Wheeler[2]; Michael Uchic[3]; Dennis Dimiduk[3]; Yoon-Suk Choi[2]; Hamish Fraser[1]; [1]The Ohio State University; [2]UES, Inc.; [3]Air Force Research Laboratory

Mechanical testing of micron-size samples provides distinct advantages over macroscopic testing for quantifying selected fundamental processes governing plastic flow, such as intrinsic size effects and direct, quantitative measures of strain heterogeneity and intermittency. We have developed a custom device for performing uniaxial mechanical tests on micron-scale samples that can operate inside a scanning electron microscope (SEM). When this device is employed within an SEM one can access both tensile and compressive test modes, and also directly observe the spatial distribution of deformation events through continuous recording of SEM images. The present study will highlight the effect that the device construction—in particular, the lateral stiffness of the compression platen or tensile grip—has on the resultant mechanical response of microcrystals that are oriented for single slip deformation. We also compare the observed deformation response to results obtained with crystal-plasticity finite element modeling of similar test structures.

11:30 AM
Size Scaling Aspects of Plastic Flow in Single Crystal Ni-Based Superalloys: *Jaafar El-Awady*[1]; [1]University of California, Los Angeles

We utilize large scale 3-dimensional Parametric Dislocation Dynamics (PDD) simulations coupled with the Boundary Element Method (BEM) to study the deformation behavior of cylindrical micron sized single crystal Ni-based superalloys. The BEM is used to calculate the image fields due to the interaction of dislocations with precipitates and the finite dimensions of the crystal. The solution is then superimposed on the elastic field resulting from dislocation ensembles in an infinite medium as calculated from the PDD. Statistical analysis are performed on cylindrical samples have diameters in the range 0.25 to 5 micrometers and the range of initial dislocation densities in the simulations are varied from 10^11 /m^2 to 10^13 /m^2. The fundamental aspects of size scaling aspects of plastic flow in a precipitation-strengthened material are identified through the use of these simulations. The simulation results are seen to agree well with experimental findings.

11:50 AM
Analysis of the Hertzian Estimate of Dislocation Nucleation Stresses in Nanoindentation Experiments: *Li Ma*[1]; Dylan Morris[1]; Stefhanni Jennerjohn[2]; David Bahr[2]; Lyle Levine[1]; [1]NIST; [2]Washington State University

The dislocation nucleation stress of crystalline materials is frequently estimated from the maximum shear stress assuming Hertzian contact up to the first "pop-in" event, which is a sudden displacement burst during load-controlled nanoindentation. However, the irregular indenter tip shape will significantly change the stress distribution, and therefore the maximum shear stress from Hertzian estimation. In this work, the near-apex shape of two real Berkovich indenters, one lightly and another heavily used, were measured by SPM and directly input into FEA models for "virtual" nanoindentation experiments on <100>-oriented single-crystal tungsten. Simultaneously, experiments were carried out using the same indenters. The load-displacement curves from FEA simulation show good agreement with those from the experiments. Hertzian-estimated radii for both indenters were significantly larger than those directly measured from the scanning-probe experiments and those obtained from FEA spherical indentation. The underestimation of the dislocation nucleation shear stress from both indenters is studied.

12:10 PM
Atomic-Scale Deformation Kinematics for Simulations of Dislocation Nucleation and Bicrystal Grain Boundary Evolution: *Jonathan Zimmerman*[1]; Garritt Tucker[2]; David McDowell[2]; [1]Sandia National Laboratories; [2]Georgia Institute of Technology

We present a method for calculating an atomic-scale deformation gradient within atomistic simulation, and use this method to analyze a biaxially stretched thin film containing a surface ledge, an FCC metal loaded by a nanometer-scale indenter, and bicrystal grain boundaries subjected to shear loading. Our analyses compare this metric's consistency with its continuum counterpart, which is known to have a zero curl for compatible deformations. Discontinuities in the deformation gradient indicate the presence of defects associated with plastic deformation, including dislocations and stacking faults. Our grain boundary simulations reveal pronounced deformation for small regions surrounding the grain boundary, and demonstrate the influence of interfacial structure on mechanical behavior. Our research provides a useful tool for linking atomistic simulation results with continuum mechanics. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Electrode Technology for Aluminum Production: Special Session: Coke Quality Changes and Countermeasures

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Barry Sadler, Net Carbon Consulting Pty Ltd; John Johnson, RUSAL Engineering and Technological Center LLC

Tuesday AM Room: 2003
February 17, 2009 Location: Moscone West Convention Center

Session Chair: Alan Tomsett, Rio Tinto Alcan

8:30 AM Introductory Comments

8:35 AM Invited
Coal Tar Pitch: Past, Present, and Future: John Baron[1]; *Robert Wombles*[1]; Stacey McKinney[1]; [1]Koppers Industries Inc

The first coal chemical recovery ovens were installed in the United States in 1893. By 1915, by-product ovens accounted for 97% of metallurgical coke produced. These by-product ovens produced coal tar as one of the major by-products. An industry developed around distillation of coal tar to produce various products. One of the major products produced is coal tar pitch. Since that time, coal tar pitch has become the binder of choice for the aluminum, commercial carbon, and graphite industries. A science has developed around defining the quality of a binder pitch based on its physical properties. Successful and unsuccessful efforts in this endeavor will be discussed. In addition some of the major changes in coal tar pitch properties as well as some of the successful and unsuccessful attempts to modify pitch properties will be discussed. Lastly, coal tar pitch supply and quality issues for the future will be addressed.

9:00 AM Invited
Refining Challenges and Opportunities for Anode Coke Quality: *Frank Cannova*[1]; Yen Hoang[1]; Bernie Vitchus[1]; Ghislain Hamel[2]; [1]BP; [2]Rio Tinto Alcan

Crude oil supply and quality has significant impact on petroleum refining as well as calcined coke availability and quality. Calcined coke quality is directly affected by the crude oil quality and refining conditions. The choice of crude to refine is mostly affected by crude price, availability and product yields. The current and future challenges for the aluminum smelting industry will be discussed from the perspective of an integrated coke producer. World crude oil impacts, challenges to anode grade coke supplies and projected calcined coke quality will be presented together with a discussion for aluminum smelter carbon plant anode options.

9:25 AM Invited
US Refining Economics – A Model Based Approach: *Todd Dixon*[1]; [1]ConocoPhillips

The US refining industry has seen a shift over the last decade towards processing heavy, sour crude. These crudes generally translate into coke that is inferior for the production of anodes versus the lighter crudes. This trend can be understood by reviewing the economics of refining a light, sweet crude versus a heavy, sour crude. To do so, a simple model refinery was developed and the economics of processing different crude types will be presented using market indices for pricing. From this model, basic coke properties that are of importance to the manufacture of anodes will also be derived.

9:50 AM Invited
Changes in Anode Raw Material Quality in China - The Impact of Imported Crudes and Refinery Modifications: *Paul Adkins*[1]; [1]AZ China Limited

China has long been a source for low-cost reasonable-quality carbon anodes for aluminium smelters. More recently however, China's green coke output has seen increasing sulphur levels. As China imports increasing amounts of crude oil, the crude slate is changing inexorably. What is the future for Chinese anodes in terms of quality and price? How will China's thirst for oil impact exports of green coke, calcined coke and anodes?

10:15 AM Break

10:25 AM Invited
Enhancing Coke Bulk Density through the Use of Alternative Calcining Technologies: *Kenneth Ries*[1]; [1]Kenneth E Ries Consulting

The quality of petroleum coke used to make anodes for Aluminum production has declined in recent years and this trend is expected to continue. For example, high volatile green coke can result in low bulk density calcined coke leading to a corresponding low apparent density baked anode. This can have a serious negative impact on the smelting process. While most of the coke used in the industry is calcined using either rotary kilns or rotary hearth furnaces, there is a notable exception - the widespread use of shaft furnaces for coke calcining in China. These furnaces have some unique attributes that result in higher than expected calcined coke bulk densities. This paper will examine the potential for wider use of these shaft furnaces, and other alternative calcining technologies, to improve calcined coke density from available green cokes.

10:50 AM Invited
Anode Coating to Prevent Air Burn Oxidation in Aluminium Smelters: *Mahnaz Jahedi*[1]; Anselm Oh[1]; Enzo Gulizia[1]; Stefan Gulizia[1]; Ali Jassim Malallah[2]; Maryam Al Jallaf[2]; Najeeba Al Jabri[2]; Ali Al Zarouni[2]; [1]CSIRO/Light Metals Flagship; [2]Dubal

Carbon anodes in Aluminum smelters are subjected to Air burn oxidation which shortens anode life and increases CO2 emission. CSIRO Light Metals Flagship has developed a novel coating to protect carbon anodes from air burn oxidation without adversely affecting current production. This coating is a barrier coating with robust properties which satisfies demanding requirement of Aluminum smelters. The coating is easy to apply and resistant to damage during transport, installation and operation. The coating maintains integrity throughout the life of the anode without cracking or melting. The industry trials showed no safety issues. The laboratory tests and prototype anode trials results are presented. The performance of this coating is compared with molten Aluminum coating and The results showed this coating has 20-30 times better performance than molten Aluminum coating while it is cost effective.The industry trials so far have shown promising results.

11:15 AM
Minimizing Impact of Low Sulfur Coke on Anode Quality: *Angelique Adams*[1]; Roy Cahill[1]; Yves Belzile[1]; Katie Cantin[1]; Michel Gendron[1]; [1]Alcoa Inc

Approximately 95% of the SO_2 emissions generated by a smelter can be attributed to sulfur found in the incoming petroleum coke used in anode production. Efforts to reduce smelter SO_2 emissions have resulted in a number of plants shifting to lower sulfur coke. Shifting to lower sulfur concentrations in the anode has been demonstrated by others to negatively impact anode quality and potentially potroom performance by increasing the anodes susceptibility to reaction with carbon dioxide. In the following paper, we explore various methods to minimize this impact. These include, types of low sulfur cokes to be used in the coke blend, alterations in aggregate granulometry, and improved baking practices. The results from these studies are presented.

11:40 AM Invited
Mild Coal Extraction for the Production of Anode Coke: *Rodney Andrews*[1]; David Jacques[1]; Terry Rantell[1]; [1]University of Kentucky

The quality and availability of petroleum coke used in the manufacture of carbon anodes for aluminum production is becoming of increasing concern to the industry. Coke quality and yields have progressively declined as changes in refinery practice and the move towards processing an increasing proportion of heavier sour crudes have affected coke properties, resulting in an increase in the metal impurities and sulfur content of the coke. An alternative supply of anode coke is required to supplement or eventually replace calcined petroleum coke. The significant domestic reserves of coal could represent a viable carbon resource for anode production, provided defined coke specifications can be met and at a cost that is economically viable. This paper will present an overview of the use of coal to substitute for pet coke, with a particular focus on recent efforts to producing anode grade coke through mild solvent extraction of coal.

Emerging Applications of Neutron Scattering in Materials Science and Engineering: Microstructure Control

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Xun-li Wang, Oak Ridge National Laboratory; Brent Fultz, California Institute of Technology; Hahn Choo, University of Tennessee

Tuesday AM — Room: 3012
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Jaime Fernandez-Baca, Oak Ridge National Laboratory; Baek Seok Seong, KAERI

8:30 AM Invited
Small Angle Neutron Scattering (SANS) Studies of Nanofeatures in Irradiated RPV Steels: *G. Robert Odette*[1]; Brian Wirth[1]; Matthew Alinger[1]; Nicholas Cuningham[1]; [1]University of California

The advantage of producing both nuclear and magnetic scattering up to high q have made SANS the dominant tool for characterizing the hardening-embrittling nano-scale features (NFs) in irradiated light water reactor pressure vessel (RPV) steels. The NFs are coherent Cu-Mn-Ni zones ranging from Cu rich to Mn-Ni rich precipitates (CRPs and MNPs). We summarize an extensive SANS database on the synergistic effects of alloy composition (Cu, Ni, Mn) and irradiation variables (flux, fluence and temperature) on the NFs, including Cu free MNPs that may result in severe but previously unanticipated embrittlement during extended RPV lifetimes. We also compare SANS results to other techniques, including atom probe tomography, which has historically suggested that there is a significant quantity of Fe in the NFs; however, this is inconsistent with that SANS data. We resolved this inconsistency by measuring the temperature dependence of magnetic scattering, demonstrating that the NFs contain little Fe.

9:00 AM
Behavior of Light Elements in Steels Studied by Small-Angle Neutron and X-ray Scattering: *Masato Ohnuma*[1]; Jun-ichi Suzuki[2]; Mayumi Ojima[3]; F.G. Wei[4]; Syuji Narita[5]; Tetsuya Shimizu[5]; Kaneaki Tsuzaki[1]; Yo Tomota[3]; [1]National Institute for Materials Science; [2]JAEA; [3]Ibaraki University; [4]Yakin Kawasaki Co. Ltd.; [5]Daido Steel Co. Ltd.

Demanding on green materials with lower emission to the environment and saving natural resource, steel is now needed to be stronger by adding smaller amount of alloying elements. To achieve such requirement, quantitative characterization of size and volume fraction of precipitates is strongly required for the efficient use of them. Since Small-Angle Scattering (SAS) is one of the optimum techniques for quantitative characterization of microstructures, applications of SAS using both X-ray and neutron are now promoting in NIMS. In this talk, we show two results as examples. First is SAS characterization of nitrogen-enriched clusters formed during tempering of high nitrogen martensitic stainless steel in conjunction with hardness. In the second part, we show the detection of hydrogen (average concentration is 0.03 at% in the sample) trapped by nano-size NbC in steel by SANS.

9:20 AM
Small Angle Neutron Scattering Study on The Cold Rolled Steel Sheet: *Eunjoo Shin*[1]; Baek Seok Seong[1]; Shi-Hoon Choi[2]; Hu-Chul Lee[3]; Kye Hong Lee[1]; [1]Korea Atomic Energy Research Institute; [2]Sunchon National University; [3]Seoul National University

For low carbon steels, the effect of a cold rolling on a SANS pattern was investigated. Several cold rolled steel samples with different reduction rates and annealed samples after a cold rolling were measured by SANS. The cold rolled samples presented anisotropic 2-dimensional(2D) SANS patterns. From the 2D SANS patterns, two kinds of 1D patterns were calculated; one was for the Q RD(rolling direction), the other for the Q//RD. The scatterer sizes calculated from the 1D patterns by using a model fitting were increased with the reduction rates, only for the Q RD section. The annealed sample presented an isotropic SANS pattern. A crystal plasticity finite element method was employed to simulate the strain distribution around fine precipitates in the steels after cold rolling deformation. Representative volume elements are used to capture the inhomogeneous deformation in ferrite matrix containing hard precipitates. The <111>//ND fiber texture components were assumed as initial orientations.

9:40 AM
Neutron And X-Ray Scattering Studies Of Scale Formation Under Idealized Bayer Processing Conditions: *Nathan Webster*[1]; Melissa Loan[1]; Ian Madsen[1]; Bill Hamilton[1]; [1]CSIRO Minerals

The efficiency of the Bayer process for extraction of alumina from bauxite ore is compromised by the formation of scale on the walls of process equipment. Scale impedes the material flow through, and heat transfer to, the process streams. Practical and economical methods for scale prevention are yet to be developed because the detailed mechanisms of its nucleation and growth are not well understood. This study aims to increase this understanding, by investigating scale formation on mild steel substrates under idealized caustic Bayer processing conditions, employing surface-sensitive neutron and X-ray scattering techniques. The results of investigations into the structure and composition of the mild steel passivation layer, the mineralogy, crystallographic orientation and growth mode of scale in the early stages of formation, and the association of cations and anions in the liquid layer about the substrate, will be discussed.

10:00 AM Break

10:20 AM
Nano-Scale Solute Partitioning in Bulk Metallic Glasses: Ling Yang[1]; Michael Miller[2]; *Xun-li Wang*[2]; Chain Liu[2]; Alexandru Stoica[2]; Dong Ma[2]; Jon Almer[3]; Donglu Shi[1]; [1]University of Cincinnati; [2]Oak Ridge National Laboratory; [3]Argonne National Laboratory

Fundamental understanding of composition variations and morphology of the nanoscale structure is essential for the development of advanced materials. A single experimental technique simply cannot provide all the answers. In this paper, we demonstrate an approach that leverages the power of several state-of-the-art characterization tools, from microscopy to x-ray and neutron scattering, to uncover the structure and phase transformation of nanocrystalline particles in devitrified bulk metallic glass. Nano-scale solute partitioning, due to strong chemical order, is revealed at an unprecedented detail by a new wide field of view atom probe. This level of details is crucial for understanding the interference peaks observed in small angle x-ray and neutron scattering experiments, a mystery that has lingered for more than a decade. The implications of our experimental results are discussed with regard to the stability of metallic glass alloys.

10:40 AM
Effect Of Nano-Sized Precipitates On The Mechanical Properties Of Low-Carbon Steels By Neutron Scattering Techniques: *Baek Seok Seong*[1]; Eunjoo Shin; Shi-Hoon Choi[1]; Kye Hong Lee[1]; [1]KAERI

SANS and powder diffraction techniques were applied to study the effect of nano-sized precipitates and a boron addition on the mechanical properties of low carbon steels quantitatively. Fine core-shell spherical precipitates with an average radius of ~ 5 nm like MnS surrounded by BN layers in boron-added steels were mainly observed. In boron added steels the number of boron-precipitates such as BN, Fe3(C, B) drastically increased at higher rolling temperature. The volume fraction of the fine precipitates of the boron added steels was higher than that of the boron free steels. The boron addition to the low carbon steels resulted in reducing the strength and improving the elongation, which is related to the reduction of the solute carbon and the nitrogen contents in the ferrite matrix caused by the precipitation of the BN as well the increase of the volume fraction of the cementites.

11:00 AM Invited
In-Situ Time-Resolved Analyses Of Microstructure In Advanced Materials Under High Magnetic Fields Using Neutron Scattering: *Jaime Fernandez-Baca*[1]; Gerard Ludtka[1]; Gail Ludtka[1]; Camden Hubbard[1]; John Wilgen[1]; Roger Kisner[1]; [1]Oak Ridge National Laboratory

We will present recent developments to conduct in-situ neutron scattering measurements of transformations that occur in materials when processed at high magnetic fields and elevated temperatures. An induction heater was designed to provide temperatures up to 1200°C inside a 5-Tesla cryomagnet. The combination of this thermal magnetic system and the Wide Angle neutron Diffractometer (WAND at ORNL's High Flux isotope Reactor allowed the time-resolved neutron diffraction study of the shift in equilibrium phase transformation temperatures that occur in an Fe-C binary alloy when a high magnetic field is applied at elevated temperatures. The use of the WAND enabled several diffraction peaks to be monitored simultaneously as the microstructure evolved under the influence of the external magnetic field. The WAND is a high-intensity, medium-resolution powder instrument operated jointly by ORNL and the Japan Atomic Energy Agency (Tokai, Japan) under the US-Japan Cooperative program on Neutron Scattering.

11:30 AM
Overview of the High Resolution Powder Diffractometer at the High Flux Isotope Reactor: *Ovidiu Garlea*[1]; [1]ORNL

The powder diffractometer HB2a at the High Flux Isotope Reactor is undergoing a major upgrade, being optimized to offer both high flux and high resolution. The instrument is equipped with a new vertically focused Ge wafer monochromator that provides one of three principal wavelengths: 2.41 A, 1.54 A, and 1.12 A. A new detector shielding, more effective and more compact, gives access to a wide scattering angle range ($-2° < 2\theta < 164°$). This diffractometer will provide high-throughput studies of nuclear and magnetic structures as a function of intensive conditions. In addition to traditional Rietveld refinements, studies of phase transitions, thermal expansion, quantitative analysis, and ab-initio structure solution from powder data can be undertaken. This presentation will give an overview of the HB2a diffractometer and illustrate its capabilities with recent neutron scattering studies on new materials ranging from ternary rare earth-alloys to organometallic systems.

Fatigue: Mechanisms, Theory, Experiments and Industry Practice: The Role of Microstructure in Fatigue

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee
Program Organizers: Koenraad Janssens, Paul Scherrer Institute; Corbett Battaile, Sandia National Laboratories; Brad Boyce, Sandia National Laboratories; Luke Brewer, Sandia National Laboratories

Tuesday AM — Room: 3008
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Corbett Battaile, Sandia National Laboratories; Luke Brewer, Sandia National Laboratories

8:30 AM Invited
Selected Problems In The Fatigue Behavior Of Titanium Alloys: Adam Pilchak[1]; Amit Bhattacharjee[1]; *James Williams*[1]; [1]Ohio State University

Titanium alloys have fatigue strengths at 10^6 cycles that typically are ~0.6 of the tensile yield strength (σ_y). Since Ti alloys essentially contain no inclusions, the fatigue strength can be tailored according to the value of σ_y without concern for intervention of inclusions as crack initiation sites. High temperature Ti alloys such as Ti-6Al-2Sn-4Zr-2Mo(+Si) and IMI834 also can exhibit a significant reduction in fatigue life if the load is held at maximum value as compared to continuously cycled in a load controlled test. This effect is called dwell fatigue. Furthermore, local variations in the microstructure due to processing or other production related events can serve as early fatigue crack initiation sites. These variations must be either eliminated or accounted for in design data, the former being preferred. This talk will describe several aspects of the fatigue behavior of Ti alloys. The practical implications of this behavior will be discussed.

9:00 AM
Deformation Of Ti-6-4 At The Microstructural Scale: Experiments And Simulations: *Philip Littlewood*[1]; Mario Nardone[1]; Fionn Dunne[1]; Angus Wilkinson[1]; [1]University of Oxford

Experiments and simulations have been conducted on the local response at the microstructural scale of the titanium alloy Ti-6-4, to imposed monotonic and cyclic deformation. Both textured and untextured material has been examined. Regions of approximately 100 µm by 100 µm have been marked on the surface using a FIB, and EBSD used to map the grain morphology and orientations before deformation. FIB was also used to generate finer surface markers in these regions. The displacements of these allow the local in plane deformation fields to be determined.EBSD measurements on the undeformed samples were used to construct a crystal plasticity finite-element simulations. Simulations were run with different combinations of allowed slip systems including a and c+a slip and basal and prismatic slip planes. The importance of these slip systems and the boundary conditions in the model are assessed through comparison between experimental observations and modelling predictions.

9:20 AM
The Population Of Databases Relating Microstructure And Fatigue In Ti-555: *John Foltz*[1]; Brian Welk[1]; Peter Collins[1]; Rajagopalan Srinivasan[1]; James Williams[1]; Hamish Fraser[1]; [1]Ohio State University

It is well known that variations in the thermomechanical history of Ti-555 (Ti-5Al-5V-5Mo-3Cr-0.5Fe), and the corresponding changes in microstructural features of the alloy can significantly impact the resulting mechanical properties. While such variation includes the fatigue life, the exact nature of the microstructure-property relationship is not well understood. In order to explore the influence of the microstructural features present in Ti-555 on fatigue life, a database relating these to the fatigue life in four-point bend (R=0.1 at 60 hertz) tests has been populated. Precisely controlled variations in thermal histories were affected using a Gleeble(R) thermomechanical simulator. The resulting microstructures have been characterized using optical and electron microscopic techniques, and subsequently quantified. The fatigue life has been measured at a constant fraction of the experimentally measured yield strengths. Fatigue life will be discussed in reference to important microstructural features.

9:40 AM
Microstructural Effects On The Mechanical Behavior Of B Modified Ti-6Al-4V Alloys: *Indrani Sen*[1]; Upadrasta Ramamurty[1]; [1]Indian Institute Of Science

Conventionally cast Ti-6Al-4V alloy possesses coarse grain size (several mm). Extensive and expensive thermomechanical processing is needed to refine the structure. However, small additions of B (< 1 wt.%) refine the as-cast grain size by an order of magnitude, hence improve their mechanical performance. In this paper, we report the effect of microstructural refinement, achieved through systematic B additions from 0.0 up to 0.55 wt.% B on the unnotched fatigue as well as fatigue crack growth of Ti-6Al-4V alloy. Experimental results show a gradual reduction in threshold for fatigue crack initiation depending on the square root of the microstructural length. Stress-controlled high-cycle-fatigue experiments reveal an enhancement in the endurance limit of the alloy with B addition, primarily due to the microstructural refinement. However, intermetallic TiB formed due to B addition, affects the crack initiation. The strain-controlled low-cycle-fatigue shows variation of the degree of cyclic-softening and tension-compression asymmetry with B addition.

10:00 AM
Fatigue Cracking Mechanisms of F.C.C. Crystalline Materials: *Z. F. Zhang*[1]; [1]Institute of Metal Research/Chinese Academy of Sciences

In the current study, fatigue cracking mechanisms of pure Cu bicrystals, polycrystalline and ultrafine-grained pure Cu, Cu-Al and Cu-Zn alloys were systematically investigated under cyclic loading. In pure Cu bicrystals, it was found that the large-angle grain boundaries (GBs) are always the preferential sites for fatigue cracking; however, we never found fatigue cracking along those low-angle GBs. In polycrystalline pure Cu, Cu-Al and Cu-Zn alloys, the large-angle GBs are still the preferential fatigue cracking sites. With the addition of Al or Zn, the surface slip bands become more homogeneous and display less localization after cyclic deformation. It is found that the annealing twin boundaries (TBs) gradually trend to produce fatigue cracks. With the grain refinement into ultra-fine level, plastic strain localization and fatigue cracking nucleated along shear bands (SB) at low strain amplitude but was changed to deformation bands (DBs) at high strain amplitude.

10:20 AM Break

10:40 AM Invited
Microstructurally Small Crack Fatigue in Lightweight Engineering Alloys: Modeling and Experiments: *Mark Horstemeyer*[1]; Haitham El Kadiri[1]; Yibin Anna Xue[1]; [1]Mississippi State University

Fatigue crack growth micromechanisms in Al7075-T651 and in four cast magnesium alloys AM50, AM60, AZ91 and AE44 were identified using fractography and in-situ SEM techniques, and predicted through a microstructurally multistage fatigue model. Namely, for magnesium alloys, the main fatigue crack initiated on shrinkage pores and to a lesser extent on large Mn-rich particles. Small cracks propagated along the a-Mg dendrite / eutectic interface, and then through the Al-rich eutectic. In the long crack regime, the crack advanced in a mix transdendritic-interdendritic mode along persistent slip bands spreading over several tens of dendrite cells. For Al7075, The fatigue crack nucleated at iron-rich intermetallics through either a debonding or crack transition from the particle into the matrix. Small cracks showed a step-like structure that changed from grain to grain. The long cracks advanced through individual damage lines along slipped planes ahead of the crack tip.

11:10 AM
Low Cycle Fatigue Variability in Single Crystal Nickel-Base Superalloys Directionally Solidified with Liquid Metal Cooled and Conventional Bridgman Processes: *Clinique Brundidge*[1]; Tresa Pollock[1]; [1]University of Michigan

Factors influencing the fatigue variability of a single crystal nickel-base superalloy tested at 538°C (1000°F) have been examined. The role of cooling rates during solidification has been investigated with the use of a liquid metal cooling (LMC) directional solidification process in comparison to a conventional Bridgman solidification technique. Additions of Tantalum to improve the shearing resistance of precipitates have also been investigated. Increases in cooling rates during solidification significantly decrease primary and secondary dendrite arm spacings as well as decrease the size of solidification shrinkage pores. Increases in cooling rates improve fatigue life by as much as a factor of seven. Solidification variables had a stronger impact on fatigue life than minor changes in chemistry. The influence of various features of cast microstructure on fatigue variability will be discussed.

11:30 AM
Influence of Grain Boundaries on the Cyclic Slip Activity of PSBs – A Comparison of Surface and Bulk Grains: *Anja Weidner*[1]; Werner Skrotzki[1]; [1]TU Dresden

The slip activity and shear strain of persistent slip bands in polycrystalline nickel were studied after half-cycle deformation at different stages of fatigue life using the combination of atomic force microscopy and scanning electron microscopy. Recent studies on surface grains showed that the half-cycle slip activity of PSBs significantly depends on the stage of fatigue life, although the local shear strain is nearly independent on it. But up to now this behaviour is indistinct. Possible reasons could be a hardening effect due to secondary slip within PSBs, the appearance of micro structurally short cracks, the influence of grain boundaries or a surface effect solely. Therefore, the surface investigations of the half-cycle slip activity as well as the local shear strain of PSBs have been extended by detailed studies on bulk grains after removing surface layer. A comparison between the results on surface and bulk grains will be discussed.

11:50 AM
The Role Of Microstructural Heterogeneity On Fatigue Lifetime Variability In The Very High Cycle Regime: *Christopher Szczepanski*[1]; Sushant Jha[2]; James Larsen[3]; J. Wayne Jones[1]; [1]University of Michigan; [2]Universal Technology Corp; [3]US Air Force

The very high cycle fatigue behavior of Ti-6246 has been investigated using ultrasonic fatigue techniques and lifetimes ranging from 10^6 to 10^9 cycles have been observed. In this regime of fatigue (0.4-0.6s_{YS}), only certain microstructural

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regions are susceptible to fatigue damage accumulation, and lifetimes are related to the distribution of these fatigue critical microstructural neighborhoods. In the current work, three distinct classes of fatigue failures have been identified; one surface initiation mechanism and two subsurface initiation mechanisms. Fatigue cracks initiate by facet formation within α_p grains favorably oriented for basal slip in microtextured regions of the microstructure. The α_p grains inherit their orientation from the prior β phase, but they do not strictly follow the Burgers orientation relationship upon transformation from the β phase. The orientation of the α_p grains with respect to the parent β phase has been investigated to determine how these microstructural neighborhoods encourage fatigue damage accumulation.

12:10 PM
Effects Of Microstructure And In Situ Development Of Crack Closure On Fatigue In Self-Healing Composites: *Eric Brown*[1]; [1]Los Alamos National Laboratory

A growing body of work in the literature is investigating a class of materials possessing the ability to self-healing in response to damage and crack growth. The first of these materials, as reported in Nature, employed ureaformaldehyde microcapsules containing a dicyclopentadiene healing agent and dispersed Grubbs' ruthenium catalyst in an epoxy matrix. This material has been demonstrated to recover over 90% of its virgin fracture properties and to exhibit significantly improved resistance to fatigue crack growth. The composite microstructure and inclusion of fluid filled microcapsules inhibit fatigue crack growth in the glassy epoxy. Self-healing functionality can lead to negative crack growth through healing under simply cyclic loading and complex loading histories. Finally, crack closure associated with the in situ self-healing mechanism is shown to arrest fatigue crack growth in high cycle fatigue loading. These fatigue mechanisms will be discussed in the initial self-healing material and subsequent variants.

Friction Stir Welding and Processing-V: Session III

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee
Program Organizers: Rajiv Mishra, Missouri University of Science and Technology; Thomas Lienert, Los Alamos National Laboratory; Murray Mahoney, formerly with Rockwell Scientific

Tuesday AM — Room: 2014
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Thomas Lienert, Los Alamos National Laboratory

8:30 AM Invited
An Experimental Framework for Advancing the Science Base of Friction Stir Processing: *Carl Sorensen*[1]; [1]Brigham Young University

Friction Stir Processing is used as a method for changing material properties as well as for joining materials. Significant effort has been expended to determine the effects of process parameters on resulting properties. In the course of developing these relationships, a number of empirical methods for understanding friction stir processing have been developed.This paper presents a framework for research in friction stir processing. It explains the domains of interest in the process, including the independent process parameter domain (spindle speed, feed rate, and depth control parameter), the dependent process parameter domain (spindle torque, process forces, power, and heat input), the microstructure domain (grain size, grain shape, texture, microstructural components), and the processed zone property domain. Research to advance the understanding of FSP should focus on the mappings between these domains. Examples of research for each of these mappings is presented.

8:50 AM
Effects Of Tool Rotation Speed And Feed Rate On Material Flow In And Next To Thread Space During Friction Stir Welding/Processing: *Zhan Chen*[1]; Song Cui[1]; [1]AUT University

In the first part of our study, the mode of material flow in and next to thread space which determines the mode of nugget zone formation was quantified. Using an Al-Si alloy, the deformation of dendrites before entering into thread space could be traced. Using this method, rapidly increases in strain and strain rate towards thread space can be observed with strain and strain rate estimated to be up to ~ 3.5 and ~ 85 s-1, respectively. In the second part of our study, how the mode of material flow affected by tool rotation speed and feed rate were studied. This series of experiments included the use of two very different alloys (an Al-7Si based an Al-4.5Cu based). Thus the effect of the different mechanical behaviors at peak temperatures on flow mode can be evaluated. Variations of welding forces associated with the mode of change will also be correlated.

9:10 AM
Bending Limits in Friction Stir Processed 5083 Aluminum Plate: *Michael Miles*[1]; Chris Smith[2]; Murray Mahoney[3]; Rajiv Mishra[4]; [1]Brigham Young University [2]Friction Stir Link; [3]Formerly with Rockwell Scientific; [4]Missouri University of Science and Technology

Bending performance of aluminum plates at room temperature can be enhanced by friction stir processing (FSP), which can locally anneal and refine grain size at the pre-tensile side of the plate. Plates with thicknesses from 8 - 25 mm of AA 5083 have been friction stir processed and then bent into a v-die to investigate the increase in ductility that results from FSP. A finite element model was also developed to predict bending limits of the friction stir processed plate, as well as an unprocessed plate. The material property gradient in the friction stir processed plate was obtained by machining tensile specimens at various locations through the thickness of the plate and then testing the specimens to generate flow stresses for the model calculations. This approach allowed for good agreement between experiments and model prediction of plate bending limits.

9:30 AM
Microstructure and Mechanical Properties of Al-Mo in situ Nanocomposite Produced by Friction Stir Processing: *Ishan Lee*[1]; P. W. Kao[1]; N. J. Ho[1]; [1]NSYSU

In this work, friction stir processing (FSP) was applied to produce aluminum based nanocomposites from powder mixtures of Al-Mo. This technique has combined hot working nature of FSP and exothermic reaction between Al and Mo. Fully dense Al-matrix composites with large amount of nanometer sized reinforcement particles, which were formed in-situ, can be fabricated by FSP without further consolidation process. The microstructure was characterized by the use of TEM, SEM and XRD. The Al-Mo intermetallic particles were identified as Al12Mo, which were formed in situ during FSP. These particles have an average size of ~200nm. Due to the fine dispersion of Al12Mo particles, the aluminum matrix has ultrafine-grained structure (~1μm). In addition, the reaction mechanism, and microstructure evolution during FSP, as well as the mechanical properties of the Al-Mo in situ composites will be presented.

9:50 AM
The Effect of Friction Stir Process on Erosion Wear Behaviors of Al-14Si Alloy: *Tun-Wen Cheng*[1]; Li-Hui Chen[1]; Truan-Sheng Lui[1]; [1]National Cheng-Kung University

In this study, die-casting Al-14Si alloy including several kinds of second phase particles were selected as base metal.Friction Stir Process (FSP) was applied to make the second phase particles more spherical and distribute uniformly in the Al matrix. The effect of second phase particles was examined by erosion test. According to the results, the erosion resistance of stirred samples is higher than that of base metal. Therefore the erosion resistance can be improved via FSP due to the modified second phase particles.

10:10 AM
Effect Of Aging Treatments On Microstructure And Mechanical Properties Of Friction Stir Processed 7075 Aluminum Alloy: *Chung-Wei Yang*[1]; Truan-Sheng Lui[1]; Li-Hui Chen[1]; [1]National Cheng Kung University

Friction stir processing (FSP) is applied on 7075-T6 aluminum alloy. The aim of present study is to clarify the effect of natural aging with reversion process and artificial aging on the microstructural homogeneity and mechanical properties of FSP-7075-T6 at the stir zone. Results show that the tensile strength and ductility is reduced after 40ºC natural aging for 96 hours. EPMA analysis shows the distribution of precipitates is asymmetrical after FSP. This phenomenon can be reduced with significant promotion in tensile strength and ductility by solid solution treatment before natural aging. The ductility can also be recovered with 200ºC reversion process. The artificial aging for FSP-7075-T6 is performed at 220ºC and 320ºC for 1 hour. Microstructural observation represents that there is no apparent difference on gain size and texture at the stir zone. The tensile strength is decreased with increasing temperature, but the ductility is significantly increased for 320ºC artificial aging.

10:30 AM Break

10:40 AM
Variation Of Metal Flow Paths In Conventional Versus Self-Reacting Friction Stir Welding: *Haley Rubisoff*[1]; Judy Schneider[1]; Arthur Nunes[2]; [1]Mississippi State University; [2]NASA-Marshall Space Flight Center

Tracer studies were conducted to compare two friction stir weld (FSW) processes using a threaded pin. Conventional (C) FSWing is used primarily for longitudinal flat welds and uses a threaded pin and backing anvil. For circumferential welding, a self-reacting (SR) FSW is made by pinching the metal between two shoulders. Copper was deposited on the crown, root, or faying surfaces of AA2219 panels before welding, and tungsten wire was placed longitudinally along the faying surface to trace the material flow in the weld. The test matrix compared tool rotational speed, travel speed, load, and pin thread pitch to better understand how the weld parameters affect the material flow. Plan, longitudinal, and transverse section radiographs were examined to determine flow paths using the copper and tungsten as markers. The results were used to model how the metal flow varied between the two FSW processes as a function of the process parameters.

11:00 AM
3-D Microstructure Modeling of Friction Stir Processed AZ31B Mg Alloy: *Zhenzhen Yu*[1]; Hahn Choo[1]; [1]The University of Tennessee

We investigated the dependence of dynamically-recrystallized grain size (d) on friction stir processing (FSP) parameters in an AZ31B Mg alloy plate. First, a systematic microstructural examination was performed on a series of compression test specimens with different initial grain sizes at various testing temperatures and strain rates in order to obtain the empirical relationship between the final recrystallized grain size (d) and Zener-Hollomon parameter (Z). Second, the Z-d relationship was applied for the prediction of final grain size distribution throughout the FSP plate in combination with the profiles of temperature and strain rate obtained from a 3-D viscoplastic model using FLUENT software under various processing conditions. Finally, the model prediction was validated by comparing to the experimentally-measured thermal history and final grain size distribution after the FSP.

11:20 AM
Producing Ultra Fine Microstructure In AZ 31 Magnesium Alloy By Submerged Friction Stir Processing: *Ali Shahnam*[1]; Fatollah Karimzadeh[1]; Mohammad Golozar[1]; [1]Isfahan University of Technology

Ultra fine grain size (UFG) microstructures with an average grain size of 300-900 nm are achieved in solution hardened AZ31 magnesium alloy prepared by submerged friction stir processing. The mean hardness of the region reaches about 95 Hv, wich is more than twice as high as that of the AZ31 matrix. The relationship between the resulting grain size and the applied working strain rate and temperature for friction stir processing in AZ31 systemically was examined. The grain refinement kinetics are analyzed and the results are self-consistent.

Frontiers in Solidification Science III: Coupled Multiphase Growth Morphologies

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Phase Transformations Committee, TMS: Solidification Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Ralph Napolitano, Iowa State University James Morris, Oak Ridge National Laboratory

Tuesday AM — Room: 2018
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

8:30 AM
Solidification Dynamics Of Regular, Irregular And Locked Eutectics: *Silvere Akamatsu*[1]; Sabine Bottin-Rousseau[2]; Gabriel Faivre[1]; [1]CNRS; [2]UPMC

A classification of eutectic microstructures into regular (periodic) and irregular (disordered) eutectics has been proposed a long time ago. According to it, regular eutectics correspond to fully nonfaceted alloys, whereas irregular eutectics arise when the interface between at least one of the solid phases and the liquid is faceted. We propose to amend this classification, on the basis of real-time observations in thin-sample directional solidification of nonfaceted, transparent and metallic alloys. We find that, in a given sample, regular eutectic grains coexist with a kind of irregular eutectic grains, within which the interphase boundaries are more or less locked onto a given direction. This direction can be strongly tilted with respect to the solidification axis, and does not vary when solidification conditions are changed. This locked dynamics signals a faceting of the interphase boundaries, independently of the properties of the solid-liquid interfaces.

8:50 AM
Spacing Selection By Curved Isotherms In Rod-Like Eutectic Solidification: *Sabine Bottin-Rousseau*[1]; Mikaël Perrut[1]; Silvère Akamatsu[1]; Gabriel Faivre[1]; [1]INSP

We report on a real-time experimental study of rod-like eutectic solidification fronts in bulk samples of a succinonitrile-camphor eutectic alloy. After a long directional-solidification time, rod-like eutectic patterns exhibit a local hexagonal order, but are neither steady nor uniform. However, the average spacing is more or less maintained constant at a value close to the minimum undercooling spacing. Simultaneously, we observe a continual drift of the rod tips towards the walls of the crucible. We assign this dynamics to a slight bulging of the front envelope, thus of the isotherms, and to the fact that rods grow perpendicular to the front envelope. After a transient, during which the pattern is stretched without rod branching, the dynamics is controlled by a balance between stretching and branching, and the system operates near the point of marginal stability for the branching instability.

9:10 AM
Early Stage Dynamics in Eutectic Solidification: *Melis Serefoglu*[1]; Ralph Napolitano[1]; [1]Iowa State University

Early-stage selection dynamics in succinonitrile-(D)camphor organic transparent rod eutectic system is investigated experimentally using directional solidification with different specimen thicknesses. The effect of the initial single-phase boundary thickness, the single-phase layer on the seeding mechanism and the overall competition between onset mechanisms are examined. The shape of the solid/liquid interface, the specimen thickness, and the grain boundaries are all observed to influence the formation of eutectic morphology in a geometrically constrained system. Additionally, the formation of single phase layer is investigated with respect to its role in the establishment of initial conditions for directional eutectic growth.

9:30 AM
Phase Field Simulation Of Eutectic Microstructure Evolution With Faceting: Abhik Choudhury[1]; *Gandham Phanikumar*[1]; [1]Indian Institute of Technology Madras

Several phase field models to simulate eutectic microstructure are now available. In this study, we have chosen the model by Wheeler et al. (1996) with modification to include faceting in one of the two solid phases. Numerical and algorithmic details will be discussed. Results obtained for isotropic case will be compared to those available in the literature. Interesting features arising out of anisotropy for one of the phases will be highlighted.

9:50 AM
Microstructure Variation And Growth Mechanism Of Hypoeutectic Al-Si Alloy Solidification Under High Pressure: *Guozhi Zhang*[1]; [1]Northeastern University

The microstructure of hypoeutectic Al-9.21wt.%Si alloy solidified under pressure were studied. The results show that the solidification microstructure refines.The primary a phase is an extended solid solution. The solid solubility of Si in a phase is up to 8.26 wt.%. the growth mode for thea phase is cellular. The microhardness of the hypoeutectic Al-Si alloy solidified under high pressure is higher than that of the hypoeutectic Al-Si alloy solidified under normal pressure. The cellular growth mechanism of thea phase is interpreted in term of the decrease of the diffusivity and the extended solid solution under high pressure.

10:10 AM Break

10:30 AM
Solidification Morphologies In The Cu-Sn Peritectic System At Low Growth Rate: *Michel Rappaz*[1]; Frédéric Kohler[1]; [1]Ecole Polytechnique Fédérale de Lausanne

Solidification at very low speed of peritectic alloys such as Fe-Ni has shown a great diversity of microstructures: alternated bands or islands of γ and δ, coupled growth of γ' and δ' lamellae as in eutectics. Similar phenomena have been observed recently in Cu-Sn alloys, which exhibit a much larger solidification interval. Although influenced by solutal convection, bands and lamellae structures of α and β were shown to alternate in the specimen while being part of a continuous structure. Unlike the nucleation-growth mechanism proposed for Fe-Ni, the formation of bands in Cu-Sn seems to proceed by a 3D overgrowth mechanism similar to that occurring in the initial stage of eutectic coupled growth. During the overgrowth of one phase by the other, lateral instabilities can develop and lead to the cooperative growth of lamellar structures. These results and remaining open questions on peritectic solidification at low speed will be discussed.

10:50 AM
Two-Phase Microstructure Formation in Peritectic Systems: *Rohit Trivedi*[1]; [1]Iowa State University

An overview of two-phase microstructure formation in Peritectic systems will be presented by examining experimental data in selected systems. Specific emphasis will be placed on the development of coupled growth in peritectic systems and on the formation of banded microstructures. The banded microstructure is shown to consist of alternate bands of primary and peritectic phases or as alternate bands of primary phase and the two-phase coupled growth. The role of nucleation site, nucleation undercooling and competitive growth of the two phases will be quantitatively examined, and shown to be critical in the formation of these two types of banded microstructures. The mechanism of coupled growth evolution and the conditions for the formation of the coupled growth under diffusive growth conditions will be presented. Experimental results on the effect of convection on the stabilization or destabilization of the coupled growth and the banded microstructures will be discussed.

11:10 AM
Microsegregation Modelling Of Multiple Phase Transformations: *Charles Gandin*[1]; Damien Tourret[1]; [1]Ecole Des Mines

A multiple phase transformation microsegregation model for the solidification of alloys is developed based on an extension of a volume averaging method. It accounts for diffusion in all phases and for the undercooling and the growth kinetics of the solidifying microstructures. It considers the occurrence of several phase transformations taking place in the presence of liquid, including peritectic and eutectic reactions. Volume averaged conservation equations for the mass of species in each phase and at each interface are coupled with an isothermal heat balance of the domain. The composition at interfaces between phases follows thermodynamic equilibrium. The diffusion fluxes at the interfaces between phases are calculated through characteristic microstructural diffusion lengths for which analytical expressions are derived.The model predicts cooling curves, volume fractions and average compositions of phases. It predicts the occurrence of recalescences during the growth of microstructures, and the progress of peritectic transformations consuming previously formed phases.

Magnesium Technology 2009: Alloys I: Calcium

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

Tuesday AM — Room: 2006
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Norbert Hort, GKSS Research Center

8:30 AM Introductory Comments

8:35 AM
Creep Resistant Mg-Al-Zn-Ca-Sr Alloy: *Kinji Hirai*[1]; Tokuteru Uesugi[2]; Yorinobu Takigawa[2]; Kenji Higashi[2]; [1]Advanced Technologies, Inc.; [2]Osaka Prefecture University

Optimum composition for creep resistant Ca and Sr added AZ91 based magnesium alloys has been investigated. Selecting Ca and Sr for improvement of heat resistance, optimum amount of these elements added to AZ91 alloy was decided by invesigating effects of these elements to structure of the alloys including formation of thermally stable inter-metallic compounds and physical properties. For analysis of tensile properties at elevated temperature, an improved constitutive equation was adopted adding stacking fault energy and constant A'. A' is a material constant that represents effects of a second phase skeleton structure, therefore, A' is an indicator of creep resistance at elevated temperature. By these investigations optimum composition of AZ91Ca1.0Sr0.5 was determined. This alloy retains the room temperature tensile properties of AZ91 while the creep resistance at elevated temperature corresponds to alloy AE41.

8:55 AM
Effect of Ca on the Fluidity of AS41B Magnesium Alloy: *Yaqin Zhang*[1]; Tetsuichi Motegi[1]; Takanori Kojima[1]; [1]Chiba Institute of Technology

In this study the effect of Ca on the fluidity of AS41B magnesium alloy was studied using a spiral mold. The microstructures and the phases of the alloys with Ca added were characterized by SEM, EPMA and XRD. The change of liquidus temperature and solidus one were measured by Differential Thermal Analysis. The results indicate that adding a small amount of Ca degrades fluidity due to the increase of the coexistence temperature of solid and liquid. As more Ca is added, however, the fluidity improves because MgSiCa refines the crystal and the quantity of coarse Mg2Si reduces. When Ca addition exceeds 1.5%, the fluidity declines again. This can be explained by the formation of coarse MgSiCa phase.

9:15 AM
Evolution of Grain Boundary Precipitate Depleted Zones in Mg-Ca-Zn Alloy Stabilized by Zr Additions: Dmitry Shepelev[1]; Evgeniya Edelshtein[1]; Alexander Katsman[1]; *Menachem Bamberger*[1]; [1]Technion - Israel Institute of Technology

The nanoscale mechanical properties of grain boundary precipitate depleted zones (PDZ's) in Mg-Ca-Zn alloy with 1 wt% Zr were analyzed using combined nanoindentation and atomic force microscopy (AFM). These mechanical properties were then correlated to the composition and precipitate distribution in PDZ's analyzed by TEM and SEM equipped with EDS. The width of PDZ's was examined after different solution treatments (ST) at 410°C and different exposures to 175°C. An increase in ST duration from 10 h to 96 h at 410°C resulted in expansion of PDZ's from ~0.75µm to ~3µm, while the following aging at 175°C for up to 24 h did not lead to detectable change in PDZ's. The lowest hardness was found in the region where Zn_2Zr precipitates density was low, regardless of solute concentration. The nanohardness of Zn_2Zr precipitate conglomerate is two times higher then the average nanohardness of the matrix.

9:35 AM
The Flame Resistance Behavior of CaO Added Mg Alloys: *Jin-Kyu Lee*[1]; Shae Kim[1]; [1]Korea Institute of Industrial Technology

Mg chips and burrs produced during cutting process and diecasting are rapidly burned and exploded when they are exposed to ambient atmosphere. In order to solve these problems, flame retardation solution has being studied in development of new Mg alloys. It is well known that Ca is used. However, Ca is high cost and difficult to handle due to their high reactivity. More than that, Ca is prone to hot cracking and reduced fluidity. Recently, CaO added Mg have been developed to obtain the same properties, as Ca addition in Mg alloy. The flame resistance behavior of Al containing Mg alloys (3, 5 and 9wt.%) with CaO addition was investigated by DTA test under ambient atmosphere, dry air and nitrogen atmosphere. This study discussed the results of flame temperature of CaO added Mg alloys compared with other high temperature Mg alloys such as AE44, AS21 and MRI153 etc..

9:55 AM
Microsegregation and Creep in Mg-Al-Ca-Based Alloys: *Jessica TerBush*[1]; Raghavendra Adharapurapu[1]; J. Wayne Jones[1]; Tresa Pollock[1]; [1]University of Michigan

Die-cast MRI230D and AXJ530 have superior creep resistance to MRI153M, despite their similar microstructure, primary eutectic phase and dislocation substructure. In order to better understand this difference in creep resistance, solute in the primary α-Mg in as-cast Mg-Al-Ca-based alloys has been examined using the electron microprobe along with a Scheil analysis. The amount of Al in solution in the primary α-Mg is of particular interest since it is likely to significantly affect the creep behavior, due to precipitation and/or solid-solution strengthening. Despite having a higher bulk Al content, MRI230D has a similar Al concentration in the primary α-Mg as AXJ530, which may explain their similarities in creep resistance. In order to systematically vary the amount of Al in solution, quaternary additions of up to 3wt% Sn have been made to Mg-5Al-3Ca.

Microsegregation and creep behavior of these quaternary alloys will be compared to AXJ530 and MRI230D.

10:15 AM Break

10:30 AM
Thermodynamic Assessment Using CALPHAD Method and Its Application for Ternary Mg-Zn-Ca System: *JoonSeok Kyeong*[1]; Hyun Kyu Lim[1]; Hoo Dam Lee[1]; Won Tae Kim[2]; Do Hyang Kim[1]; Byeong Joo Lee[3]; [1]Yonsei University, Department of Metallurgy/NSM Laboratory; [2]Cheongju University; [3]POSTECH

Recently, it has been shown that Mg-Zn-Ca ternary system exhibits good creep resistance due to high temperature stability of Mg2Ca compound and solution hardening effect of Zn. However, detailed thermodynamic assessment for ternary Mg-Zn-Ca system has not been reported yet. In the present study, phase equilibrium and thermodynamic assessment on Mg-Zn-Ca system including two ternary compounds (τ1: Ca2Mg6Zn3, τ2: Ca2Mg5Zn13) have been investigated. In order to assess thermodynamic parameters of ternary compound τ2, isothermal section and isopleths surrounding the composition range of τ2 have been suggested using DSC, DTA, XRD, SEM, and TEM. Calculated phase diagram of Mg-Zn-Ca ternary system have been studied with ternary compound parameters. The result of solidification simulation via Scheil equation has been discussed with the specimens in as-cast state and after heat treatment. In particular, the composition range for invariant reactions has been compared with that for the bulk glass formation.

10:50 AM
The Influence of Calcium and Cerium Mischmetal on the Ignition Behavior of Magnesium: *Hongjie Luo*[1]; Yihan Liu[1]; [1]Northeastern University

Magnesium and its alloys are very active and readily igniting during heating and melting, therefore their application is limited. In this study, some anti-ignition magnesium alloys were prepared by adding Ca or Ce mischmetal into molten magnesium and their ignition points were also measured. Meanwhile, the microstructure of oxidation film was observed and the anti-ignition mechanism was analyzed. The results showed that the ignition point of pure magnesium increases gradually with increasing Ca content. The ignition point reaches 824° when Ca content is 4.5%. It is higher than that of pure magnesium about 189°. Ca and Mg composite oxidation film is key to prevent burning in the molten magnesium surface. The ignition point of pure magnesium increases first and then decreases with increasing Ce mischmetal content. The ignition point teaches 690.5° when Ce mischmetal content is 0.9%. The flame-retarded effect of Ca is better than that of Ce mischmetal obviously.

Magnesium Technology 2009: Applications, Testing and Forming

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

Tuesday AM — Room: 2007
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Wilhelmus Sillekens, TNO Science and Industry

8:30 AM Introductory Comments

8:35 AM
A Life Cycle Assessment of a Magnesium Front-End Auto Part: *Sujit Das*[1]; Alain Dubreuil[2]; Lindita Bushi[3]; Ambalavanan Thaurmarajah[4]; [1]Oak Ridge National Laboratory; [2]Natural Resources Canada; [3]GHG Measurement; [4]CSIRO/CAST-CRC

This paper focuses on the comparative life cycle assessment of a magnesium front end automotive part for a 2007 GM-Cadillac CST. The analysis framework is based on the consequential life cycle assessment approach, using an extensive life cycle inventory data collected for various magnesium manufacturing technologies including primary production, cast and wrought, and end-of-life. The recent magnesium market trend has been captured with the consideration of the latest technology developments made for the Chinese primary magnesium production. Life cycle comparative assessment includes energy, GHG emissions and selected other air emissions for the baseline steel front end and the magnesium alternative. It is estimated that both energy and GHG reductions with the magnesium front end compared to the base line scenario can be achieved only after several years of operation into vehicle life. Sustainable material management and technology improvements including recycling would further enhance the environmental benefit of magnesium use in automobiles.

8:55 AM
Initial Evaluation of Advanced Powder Metallurgy Magnesium Alloys: *Tyrone Jones*[1]; Katsuyoshi Kondoh[2]; [1]US Army Research Laboratory; [2]Joining and Welding Research Institute

The U.S. Army Research Laboratory (ARL) is interested in assessing the performance of different magnesium alloys. The ARL and the Joining and Welding Research Institute (JWRI) conducted a joint effort to develop and evaluate advanced powder metallurgy magnesium alloys AZ31B and AMX602 (Mg-6Al-0.5Mn-2Ca/mass%) sheets. JWRI performed the mechanical and metallurgical analysis, while ARL performed the ballistic analysis. The thin gauge magnesium alloy sheets were ballistically evaluated against the 0.22-cal fragment simulating projectile (FSP). The powder magnesium alloys' mechanical properties and ballistic performance are compared to the conventionally processed AZ31B-H24.

9:15 AM
Microstructural and Mechanical Aspects of Reinforcement Welds for Lightweight Components Produced by Friction Hydro Pillar Processing: *Gustavo Pinheiro*[1]; Jorge dos Santos[1]; Karl Kainer[1]; Norbert Hort[1]; [1]GKSS-Forschungszentrum

The development of new local engineering methods is one possibility to overcome the disadvantages of poor high temperature creep properties of present magnesium alloys. Friction Hydro Pillar Processing, which is a relative new friction-based welding process, can be used to improve locally the high temperature properties of magnesium alloys. Mechanical and Metallurgical aspects of magnesium AZ91D-T6 reinforced with a Mg-Al-Ca-Sr alloy were investigated. Welded samples presented a complex joint formation mechanism with plasticized material flowing towards the rotational centre in a first stage of the process and outwards after the saturation of the central region. Dynamic recrystallization has occurred in the recrystallized zone resulting in fine, equiaxed and homogeneous grains. Temperature measurement indicates solidus temperatures in regions around the bonding line were transgressed during the welding. Mechanical tests have also demonstrated the feasibility of the process in producing high efficiency joints with strength values comparable with those of base material.

9:35 AM
Die Forging of the Alloys AZ80 and ZK60: *Gerrit Kurz*[1]; Bob Clauw[2]; Wim Sillekens[3]; Dietmar Letzig[1]; [1]GKSS Research Centre Geesthacht GmbH; [2]N.V. INOFER S.A; [3]TNO Science and Industry

Overall goal of the MagForge project is to provide tailored and cost-effective technologies for the industrial manufacturing of magnesium forged components. Scientific and technological aspects are new alloys/feedstock materials with improved performance, forging process modeling and design tools with a satisfying level of predictability, machining technology for safe and rational finishing of the forgings, and typical demonstrator components to validate results. The targeted advancements are to enable high-volume applications of magnesium forgings. This paper gives an overview on the MagForge project and the first results. The commercial magnesium alloys AZ80 and ZK60 were investigated. With respect to the different microstructure of the alloys the mechanical properties of the feedstock, the parameters of the forging process and the mechanical properties of the forging parts will be presented and discussed. The results show that the magnesium alloys offer the possibility to produce parts with complex geometries similar to aluminum forging parts.

9:55 AM
Ignition-Proof of Magnesium Alloy and Preparation of Magnesium Foam: *Haibin Ji*[1]; Guangchun Yao[1]; Letian Liu[1]; Yihan Liu[1]; Guoyin Zu[1]; [1]Northeasten University

The magnesium is highly active during melting.So the Protection measures must be taken when we want to produce foam magnesium.In this experiment we used alloying method in order to prevent magnesium from burning.Mg-Al-Ca alloys were prepared. The contents of calcium were from 0.5%(wt) to 5.0%(wt) increasing by 0.5%(wt). The aluminum is 9.0% and the rest is magnesium.The result showed the magnesium alloy didn't burn when the content of Ga was 3.5%(wt). CO2 decomposited by MgCO3 can stay in the magnesium melt. So we used MgCO3 and Mg-9%Al-3.5%Ca(wt) alloy to produce magnesium foam. The foaming experiment was operated at 620°C. Different size of MgCO3 were added into melt to prepare mangnesium foam. The result showed the size of bubble was influnced obviously by the size of MgCO3. We can changE the size and content of MgCO3 to produce magnesium foam with different density.

10:15 AM
Applicability of Existing Magnesium Alloys as Biomedical Implant Materials: *Muge Erinc*[1]; Wilhelmus Sillekens[1]; Raymond Mannens[1]; [1]TNO Industrie en Techniek

Being biocompatible and biodegradable, magnesium alloys are considered as the new generation biomedical implant materials, such as for stents, bone fixtures, plates and screws. A major drawback is the poor chemical stability of metallic magnesium; it corrodes at a pace that is too high for most prospective implant applications. Requirements for biodegradable implants are biocompatibility, controlled biodegradability and sustainable mechanical properties. Various magnesium alloys containing Al, Zn, Y and rare-earth elements are analyzed in this respect. The alloys are compared on the basis of microstructure, tensile tests and potentio-dynamic polarization tests in simulated body fluid. The effects of semi-solid processing, hot extrusion, heat treatments and sterilization on corrosion resistance and tensile properties are investigated. AZ80 magnesium alloy with certain post-processing treatments fulfills the requirements best as a prospect implant material which has the potential for further improvement by trace alloying additions and surface modifications.

10:35 AM Break

Magnesium Technology 2009: Extrusion

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

Tuesday AM — Room: 2007
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Kwang Seon Shin, Seoul National University

10:50 AM Introductory Comments

10:55 AM
Influence of Alloying Additions on the Microstructure Development of Extruded Mg-Mn Alloys: *Jan Bohlen*[1]; Jacek Swiostek[1]; Dietmar Letzig[1]; Karl Ulrich Kainer[1]; [1]GKSS Forschungszentrum

In this study the effect of different alloying elements on the microstructure development of magnesium-manganese alloys during extrusion will be examined. Using alloy M1, which contains up to 1 wt.% Mn in solid solution as a basis, further elements such as rare earth elements or zirconium are added to the melt and cast into billets for extrusion. The effect of the alloying addition is analysed during indirect extrusion trials by varying the extrusion speed. Characterisation of the microstructure before and after extrusion gives information on the microstructure evolution during extrusion. Uniaxial tension and compression tests at ambient temperature relate the microstructure to the mechanical properties. The results are discussed with respect to the influence of these alloying elements on the microstructure development as well as the deformation and recrystallisation behaviour during extrusion.

11:15 AM
Texture Evolution in an AZ31 Mg Alloy during Direct and Indirect Extrusion Processes: *Shi-Hoon Choi*[1]; Hyeong-Wook Lee[1]; Dae-Ha Kim[1]; Duk-Jae Yoon[2]; Sung-Soo Park[3]; Bong-Sun You[3]; [1]Sunchon National University; [2]Korea Institute of Industrial Technology; [3]Korea Institute of Materials Science

Direct and indirect extrusion processes for an AZ31 Mg alloy were performed at various ram speeds. A finite element (FE) analysis with DEFORMTM -2D V9.1 was conducted to evaluate the deformation gradient tensor during direct and indirect extrusions. The evolution of extrusion texture in an AZ31 Mg alloy has been simulated numerically using a visco-plastic self-consistent (VPSC) polycrystal model. In order to capture crystallographic rotation during extrusion deformation, four slip and a tensile twin systems were considered in the polycrystal model. From direct and indirect extruded specimens, macrotexture was measured using X-ray diffractometer. The experimental results were compared with the results predicted by the theoretical approach. The FE analysis combined with the VPSC polycrystal model successfully predicted the inhomogeneous texture distribution through thickness direction in the extrusion specimens.

11:35 AM
Influences of Load Direction and Grain Size on Tension-Compression Yield Asymmetry of Extruded AZ31 Magnesium Alloy: *De Liang Yin*[1]; Jing Tao Wang[1]; [1]Nanjing University of Science and Technology

Mechanical tests were conducted to investigate the influences of load direction and grain size on tension-compression yield asymmetry of an extruded Mg-3Al-1Zn alloy. A criterion for the activation of deformation modes was proposed to analyze the effect of load direction on twinning activity. When the load angle was 45o, the twin area fractions after tension and compression resembled each other, and the corresponding ratio between compression yield stress and tension yield stress equaled 1.02. As the load direction was parallel or perpendicular to the extrusion axis, the yield stress and twinning activity in tension differed obviously from those in compression, showing marked tension-compression yield asymmetry. Although grain-coarsening promotes twinning in tension along extrusion axis, it cannot reduce the yield asymmetry. Further, the contributions of twinning to strain during yield deformation were evaluated based on the quantitative statistic of twin area fraction.

Materials for High Temperature Applications: Next Generation Superalloys and Beyond: Refractory Alloys I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS: Refractory Metals Committee
Program Organizers: Joseph Rigney, GE Aviation; Omer Dogan, National Energy Technology Laboratory; Donna Ballard, Air Force Research Laboratory; Shiela Woodard, Pratt & Whitney

Tuesday AM — Room: 3010
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Omer Dogan, National Energy Technology Laboratory; John Perepezko, University of Wisconsin

8:30 AM Invited
Very High-Temperature Nb-Silicide Based Alloys: Bernard Bewlay[1]; Laurent Cretegny[1]; *P.R. Subramanian*[1]; [1]GE

The present paper will describe progress in the development of Nb-silicide-based alloys with particular emphasis on the investment casting. These alloys are being developed for structural applications with service temperatures of up to 1350°C. These composites contain high-strength Nb silicides and they are toughened by a Nb solid solution. From a commercial perspective, investment casting of Nb-silicide alloys offers substantial potential because of its proximity to existing airfoil manufacturing practices. However, investment casting is not well developed for Nb-silicide composite airfoils. Furthermore, there is only limited understanding of the relationship between composition, processing technique, and properties. Alloying schemes have been developed for an excellent balance of room-temperature toughness, fatigue crack growth behavior, high-temperature creep performance, and oxidation resistance over a broad range of temperatures. Nb-silicide alloys will be described with emphasis on processing, microstructure, and performance.

8:55 AM
Beyond Superalloys: The Role of Ultra-High Temperature Materials in Aero-Propulsion: *Douglas Berczik*[1]; Charles P. Logan[1]; Michael J. McCaffrey[1]; [1]Pratt & Whitney

Ceramic Matrix Composites have the positive attributes of light weight, environmental durability, good creep resistance and high stiffness but have poor transverse properties and manufacturability and are an order of magnitude more expensive then superalloys. Refractory metal/intermetallic systems such as the Mo-Si-B alloys have exceptional thermal conductivity, possess good strength, can be machined into complex shapes and are similar in cost to superalloys but they are dense, 10% greater than superalloys and require coatings for full life. Advanced designs have begun that treat these two materials as complementary rather than rival systems. It is only through careful consideration of the environment and performance requirements of each component in an advanced aero-propulsion system that an optimum choice can be made between these two materials.

9:15 AM Invited

Computational Design of High-Temperature Alloys: *Gregory Olson*[1]; [1]Northwestern University

A systems approach to computational materials design has integrated materials science, applied mechanics and quantum physics in the predictive-science-based creation of high-performance alloys. An ongoing NASA initiative extends the DARPA-AIM methodology of accelerated aeroturbine disc process optimization through high-fidelity computational thermodynamics based simulation of microstructural evolution, employing high-resolution microanalysis for calibration and validation of PrecipiCalc code predictions. A recent AFOSR initiative has employed extensive first-principles calculations in the accelerated design of niobium-based alloys demonstrating protective YAG scale formation for oxidation resistance at 1300C in advanced turbine blade applications.

9:40 AM

Application of Phase Diagram Calculations to the Development of Nb-Silicide Based Alloys: *Ying Yang*[1]; B. Bewlay[2]; S.-L. Chen[1]; F. Zhang[1]; Y. Chang[3]; [1]CompuTherm LLC; [2]General Electric Global Research; [3]University of Wisconsin-Madison

In-situ refractory metal intermetallic composites (RMICs) based on Nb-Silicides are candidate materials for ultra-high temperature applications (T>1200ºC). To provide a balance of mechanical and environmental properties, Nb-Si composites are typically alloyed with Ti, Hf, and Cr. Phase diagrams of Nb-Si-Ti-Hf-Cr, are critically needed as prerequisite knowledge for the development of this family of materials. In this study, a thermodynamic database that compiles the Gibbs energy functions of the phases in the Nb-Si-Ti-Hf-Cr system was developed. It was then coupled with Pandat software for the calculation of phase equilibria and solidification paths. The calculated results were validated using designed experiments. Phase diagram calculation based on the developed Nb-Si-Ti-Hf-Cr thermodynamic database can provide engineers with useful insights on the understanding of the as-cast and heat-treated microstructure of existing alloys. The phase diagram calculations also allow selection of compositions and heat-treatment schedules for promising new alloys.

10:00 AM

First Principles Design of Ductile Refractory Alloys: Ductility Criterion: *Michael Gao*[1]; Omer Dogan[2]; Paul King[2]; [1]National Energy Technology Laboratory/Parsons; [2]National Energy Technology Laboratory

Refractory alloys such as Cr and Mo hold the promises for advanced fossil power generation applications such as oxy-fuel gas turbines. However, improvements to their ductility at low homologous temperatures has been an area of intense interest for decades. Several ductility criteria have been proposed and controversy remains. In this work, we examine two criteria for bcc refractory alloys using first principles calculations: the Rice–Thomson parameter and Poisson ratio. The Rice-Thomson parameter refers to the ratio mu*b/gamma, where mu is the shear modulus of the material in the preferred slip plane; b is the Burgers vector of a dislocation in the preferred slip direction within the slip plane and gamma is the surface energy of the fracture plane. Both Cr- and Mo-based binary alloys are theoretically examined. Alloying strategies to improve their intrinsic ductility are proposed and also compared with available experimental data.

10:20 AM Break

10:30 AM Invited

Fracture and Fatigue of Advanced Nb-Si Alloys: *John Lewandowski*[1]; [1]Case Western Reserve University

The fracture and fatigue behavior of a variety of Nb-Si alloys will be reviewed. Fracture experiments have been conducted on both notched and fatigue-precracked samples. while fatigue crack growth behavior has also been characterized over a range of test temperatures. Microstructures and fracture paths have been characterized via conventional metallography, SEM, and laser confocal microscopy. The mechanical behavior of these advanced Nb alloys will be compared to data available in the literature for a range of high temperature materials.

10:55 AM Invited

NbTiSiMo-X Alloys – Composition, Microstructure Refinement and Properties: *Young-Won Kim*[1]; Menon Sarath[1]; Christopher Woodward[2]; [1]UES Inc; [2]Air Force Research Laboratory

Advanced NbTiSi-X alloys have demonstrated very high RT strength levels (compressive yield strength around 1,800MPa) and excellent high-temperature strength retention (~1,200MPa at 1,000°C and over 500MPa at 1,200°C). Unfortunately, these alloys have a highly inhomogeneous size and spatial distribution of silicides, low fracture strength under tension (<350MPa) at all temperatures, and low oxidation resistance. The high volume fraction and non-uniform size distribution of silicides were considered to be responsible for the poor fracture resistance. In this work we attempt to refine the microstructure of cast alloys and to increase oxidation resistance by introducing Mo and adjusting other alloying additions. In focused efforts, we explored highly refined near-eutectic alloys modified with Mo additions, NbTiSiMo-Y, that showed excellent compression flow behavior. Further chemistry adjustments were made for balanced improvements, and cast alloys in homogenized material forms were evaluated for fracture and oxidation resistance. Results will be discussed.

11:20 AM

High Temperature Oxidation Characteristics of Nb-10W-XCr Alloys: *Maria Moricca*[1]; Shailendra Varma[1]; [1]University of Texas

The effect of Cr content on the static and cyclic oxidation resistance of Nb-10W-XCr alloys has been investigated. Experiments were conducted in air for 24 hours, over a range of temperatures from 700 to 1400°C using static and seven cycle screening tests. The phases present in the alloys and the oxide scales were characterized by XRD, SEM and EDS. Alloy's microstructure consists of Nb solid solution phase regions surrounded by a network of $NbCr_2$ laves phase. The oxidation kinetics follow a parabolic behavior; isothermal experiments indicate a trend of improvement in oxidation resistance with increase of the intermetallic phase with the exception of 30Cr alloy, suggesting the existence of a limit concerning the effective Cr content. The oxidation products are a mixture of Nb_2O_5, Cr_2O_3 and $CrNbO_4$. Results delineate the influence of microstructure and composition on oxidation mechanisms of these alloys that represent a promising base for high-temperature intermetallic alloy development.

11:40 AM

Oxidation Behavior of Nb-15Si-20Mo-5B-20Ti and Nb-15Si-20Mo-5B-20Cr Alloys between 700 and 1300°C: *Benedict Portillo*[1]; Shailendra Varma[1]; Julieta Ventura[1]; Rabindra Mahapatra[2]; [1]University of Texas at El Paso; [2]Naval Air Warfare Center

X-20Ti and X-20Cr (X = Nb-15Si-20Mo-5B and compositions are in atomic percents) alloys have been oxidized in air from 700 to 1300°C for (a) 24 hours (Short Term Oxidation, STO) and (b) cycles of 24 hours for 2 weeks (Long Term Oxidation, LTO). Weight gain per unit area as a function of time (LTO) and temperature (STO) have been used for characterizing the oxidation behavior. Oxidized two and three phase alloys, respectively, have been characterized by XRD, EDS on FESEM, and back scattered electron imaging. Results of oxides formation and phase transformations will be presented. Long term stability of the alloys will be characterized by TEM. The influence of Ti and Cr on Nb-15Si-20Mo-5B alloy will be analyzed.

12:00 PM

Study of the Effects of Hf and Mo Additions on the Microstructure and Properties of Nb Silicide Based Alloys: *Panayiotis Tsakiropoulos*[1]; Jie Geng[2]; [1]The University of Sheffield; [2]University of Surrey

Niobium silicide based alloys could replace Ni superalloys in some structural applications at high temperatures. Their development is aiming to improve their oxidation and mechanical properties at room, intermediate, high and very high temperatures. Alloying and processing strategies are seeking to enhance the performance of the Nbss and to identify optimum microstructures, and to understand how these affect performance at different temperatures. We have studied the role of Hf and Mo in the microstructures of as-cast and heat-treated Nb-24Ti-18Si-5Al-5Cr (at %) based alloys. The phases observed were the Nbss, 3-1 and 5-3 silicides, and Laves phase. The role of Hf and Mo individually or simultaneously regarding the selection/stability of the Nb3Si silicide, the structure of the 5-3 silicide (beta, alpha or gamma), the formation of the Nbss +

Nb3Si eutectic and the Laves phase, the betaNb5Si3 alphaNb5Si3 + (Nb,Ti)ss transformation and the oxidation of the alloys will be discussed.

12:20 PM
Cr-Base Alloys: Current Problems and Future Possibilities for High-Temperature Applications: *Yuefeng Gu*[1]; H. Harada[1]; [1]NIMS

In search of new materials for use as components in gas turbine engines, considerable interest has been shown in chromium (Cr) and Cr-rich alloys because Cr has high melting point (1863°C) and good oxidation resistance. Its low density and high thermal conductivity (two to four times higher than most of Ni-base superalloys) are also attractiveness to the benefit of the increasing efficiency. However, the implementation Cr-rich alloys as a viable substitute for Ni-base alloys has been impeded by their poor ductility at ambient temperature and low strength at high temperature. Recently, we find that adding Ag to Cr can greatly improve its tensile ductility. Some Cr-rich binary alloys show improved tensile ductility at ambient temperature and adequate strength at high temperature. Therefore, new composition design and process would open absolute opportunity for Cr-base alloys as a structural material used at temperatures up to 1300°C.

Materials in Clean Power Systems IV: Clean Coal-, Hydrogen Based-Technologies, and Fuel Cells: High Temperature Materials for Power Generation

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Harvesting and Storage Committee
Program Organizers: K. Scott Weil, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; Ayyakkannu Manivannan, US DOE; Z. Gary Yang, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University Zi-Kui Liu, Pennsylvania State University

Tuesday AM Room: 3005
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: K. Scott Weil, Pacific Northwest National Laboratory; Ayyakkannu Manivannan, US DOE

8:30 AM Introductory Comments

8:35 AM Keynote
Technology Challenges for Advanced Power Generation- A UK Perspective: *Derek Allen*[1]; [1]Alstom Power

The energy landscape is changing dramatically. Climate change, record oil prices, fuel poverty and issues of security of supply are now a reality. Whilst technologies adopted to address these challenges will be dependent on geography, politics and economics, it is also clear that there will be a need to adopt a 'portfolio' approach to energy generation that includes fossil, renewables and nuclear power. Materials engineering is an underpinning technology which can and will help provide solutions to the many issues throughout the energy innovation chain: extraction, processing, conversion, transmission, supply and use. A number of materials research areas need to be addressed and investments need to be made in higher risk, higher potential reward projects for generating clean power. This presentation reviews how the UK is handling this challenge via development of a national strategy for Energy Materials in support of a low-carbon economy.

9:20 AM Invited
Fossil Energy's Advanced Research Materials Program: *Patricia Rawls*[1]; [1]US DOE, NETL

Advanced materials are required for the cleaner, more efficient and more economic electricity power generating systems that are currently being developed. To that end, research is focused on developing high-temperature, corrosion-resistant alloys and protective coatings that are suitable for the extreme environments of the advanced energy systems that are currently being designed. The scope of the Materials Program addresses the need for new materials that can withstand higher temperatures and corrosive environments of advanced power generation technologies including ultrasupercritical steam cycle plants, oxy-fueled combustors, CO2 sequestration, IGCC plants, synthesis-gas-fueled turbines and turbines. Fossil Energy's Advanced Research Materials Program, coordinated by the National Energy Technology Laboratory, is organized into five clusters including Breakthrough Concepts, New Alloys, Coatings and Protection of Materials, Ultrasupercritical Materials and Functional Materials and is implemented through partnerships with academia, industry, non-profit organizations and other national laboratories.

9:55 AM
Chromium Free Nickel Alloys For Hot Sulfuric And Sulfur Environments: *Joseph Newkirk*[1]; Richard Brow[1]; [1]Missouri University of Science and Technology

Resistance to attack by hot, corrosive gases or liquids is important in many energy applications. A new nickel-silicon material based on the intermetallic alloy Ni_3Si has been developed which naturally forms an in-situ glass, amorphous silica, film during exposure to oxidizing conditions. The alloy has an excellent balance of mechanical properties and extremely low corrosion rates. The effects of alloying additions to Ni_3Si on mechanical properties and corrosion resistance will be presented. The mechanisms for the creation of a passive film in oxidizing acids will also be presented. Understanding the film formation mechanism is leading to improved performance through modifications of the alloy chemistry. A total materials approach is necessary to produce an alloy and a film both with good properties, while matching important properties at the interface. Fabrication and welding of the alloy into components will also be addressed.

10:15 AM
Refractory Degradation by Slag Attack in Coal Gasification: *Jinichiro Nakano*[1]; Sridhar Seetharaman[1]; James Bennet[2]; Kyei-Sing Kwong[2]; Tyler Moss[1]; [1]Carnegie Mellon University; [2]National Energy Technology Laboratory

Refractory wear in air cooled slagging coal gasifiers has been identified as one of the top research needs of gasifier users to increase gasifier on line availability. This research investigates Sessile drop interfacial reactions between two refractory materials (high chromia and alumina) and two slags [coal (51SiO2-24Al2O3-15Fe2O3-7CaO-3K2O) and petcoke (24SiO2-46V2O5-7Al2O3-11Fe2O3-11CaO-1K2O)]. Pulverized slag samples were placed at specific microstructure locations on refractory materials and heated to 1500 ºC at log(Po2) = -8. Cross-sections of the slag/refractory interface were evaluated using SEM-EDS for evaluating slag penetration into specific areas of the refractory and refractory grain dissolution into the slag. Initially, the slag attacked grain boundaries and fine microstructure areas, freeing alumina grains into the slag. VOx formation in the petcoke was found to alter the overall slag composition and kinetic behavior.

10:35 AM Break

10:40 AM Invited
A New Oxidation Resistant Ni-Fe-Cr-Al Alloy for Elevated Temperature Applications: *Vinay Deodeshmukh*[1]; Steve Matthews[1]; Henry White[1]; Dwaine Klarstrom[1]; [1]Haynes International

High temperature oxidation of chromia-forming alloys is considerably accelerated in the presence of water vapor in a variety of industrial application such as microturbines, solid oxide fuel cell, ultra super-critical power plants. Alumina-forming alloys may provide adequate long-term protection in harsh oxidizing environments. An issue is the fact that several alumina-forming alloys often encounter fabricability and formability problems due to gamma-prime-Ni3Al precipitation. A new Ni-based superalloy based on the Ni-Fe-Cr-Al system which has excellent fabricability and formability has been recently developed for applications in harsh oxidizing environments. It forms a continuous, adherent, and slow-growing alumina-based scale for high temperature protection. This alloy also exhibits an excellent thermal stability which is needed for long-term use at elevated temperatures. The oxidation behavior and mechanical properties of this alloy were studied and compared to several solid solution strengthened alloys. This paper will discuss some of the major characteristics and benefits of this alloy.

11:15 AM
Controlling Thermal Diffusion by Manipulating Nanoscale Structural Arrangement and Doping in Nanocomposites for High Temperature Applications: Vikas Samvedi[1]; *Vikas Tomar*[1]; [1]University of Notre Dame

Next generation ceramic nanocomposite coatings are required to have the least thermal conductance to prevent exposing the substrates to extreme temperatures. Controlling thermal diffusion and, therefore, thermal conduction is an important requirement in nanocomposites for high temperature applications. Thermal conduction in the nanocomposites occurs across a series of grain boundaries (GBs), second phase particles, and primary matrix phase grains. It is possible that by altering the nanostructural arrangement of GBs, the second phase, and the primary phase, desired control of thermal conduction can be obtained. In the presented research, such analyses for silicon carbide (SiC)-silicon nitride (Si3N4) nanocomposites using non equilibrium molecular dynamics scheme are performed. Analyses show that it is possible to control thermal conduction by manipulating the phase arrangement. Fundamental mechanism to such control is the change in phase arrangement correlated with changes in overall mean phonon wavelengths. Alternate phase arrangements based on biomimetic structures are also analyzed.

11:35 AM

High Temperature Oxidation Of Ti-Si Alloys In Water Vapor Containing Air And Ar-20%O2 Gases: *Jie Yang*[1]; Hugh Middleton[2]; Truls Norby[1]; [1]University of Oslo; [2]University of Agder

The oxidation behavior of Ti-Si based alloys with two different silicon contents (2wt.% and 8.5wt.%) were investigated and compared to pure titanium (grade 2). Isothermal oxidation took place in air-2.5%H2O and Ar-20%O2-2.5%H2O at 800°C and 1000°C for up to 72h. The scale growth kinetics, morphology and composition were studied by thermogravimetry in combination with SEM/EDAX. The oxidation of the Ti-Si alloys at 800°C and 1000°C in both gas mixtures generally displayed parabolic behaviour as did pure titanium. Silicon plays a significant positive effect on the oxidation resistance. The oxidized scales are sub-layered; the existence of SiO2 layer beneath the TiO2 layer ensures better oxidation resistance. Nitriding of the Ti-Si alloys oxidized in air-2.5%H2O leads to the formation of titanium nitride which provides better oxidation resistance compared to Ar-20%O2-2.5%H2O; the diffusion of nitrogen into the oxidized layers decreases the dissolution of oxygen into the substrate.

Materials Issues in Additive Powder-Based Manufacturing Processes: Additive Manufacturing Metals II

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Powder Materials Committee
Program Organizers: David Bourell, University of Texas; James Sears, South Dakota School of Mines and Technology; Pavan Suri, Mississippi State University

Tuesday AM — Room: 3004
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: James Sears, South Dakota School of Mines and Technology

8:30 AM

Overview of Materials Processing with Laser Engineered Net Shaping: *John Smugeresky*[1]; Baolong Zheng[2]; Yuhong Xiong[2]; Jonathan Nguyen[2]; Yizhang Zhou[2]; Enrique Lavernia[2]; Julie Schoenung[2]; [1]Sandia National Laboratories; [2]University of California, Davis

Laser Engineered Net Shaping (LENS®) has combined laser deposition and powder metallurgy technologies with advanced methodologies for rapid manufacturing, converting complex CAD models into functional structural components without the need for part-specific tooling. In addition to fabricating complex geometries of fully dense metals that require minimal finish machining, the rapid-solidification deposition mechanism has produced microstructures with mechanical properties that have typically been superior to those of components made by conventional processes. In this overview, recent research and progress associated with the LENS® process are reviewed, such as laser materials processing for rapid manufacturing; development of metal alloys and composites; the effect of process parameters on microstructure, properties, and build height; thermal behavior measurements, and numerical simulation. This paper is based upon work supported by the National Science Foundation under Grant No. DMI-0423695. Work by Sandia is supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

8:55 AM

Metallic Parts Fabrication with the Selective Inhibition Sintering (SIS): *Behrokh Khoshnevis*[1]; Mahdi Yoozbashizadeh[1]; Yong Chen[1]; [1]University of Southern California

The fundamentals of the SIS-Metal process based on microscopic mechanical inhibition are expolored. In the process, salt solution is printed in the selected area of each metal powder layer; the salt re-crystallizes when water evaporates; salt crystals decompose and grow rapidly prior to sintering; the generated salt particles spread between metal powder particles and prevent the fusing of these particles together, hence inhibiting the sintering process in the affected regions. The SIS-Metal process has numerous advantages including low cost, minimal shrinkage and deformation effects, and non-contamination of sintering furnace because of lack of polymeric binders.

9:20 AM

The Influence of Power Type On Properties of Electron Beam Melted Components: *Denis Cormier*[1]; [1]North Carolina State University

Gas atomization (GA) and plasma rotating electrode (PREP) are two common methods used to produce titanium powders for direct metal additive manufacturing processes. The production method affects the size distribution, the shape and flowability, and the porosity of the powder. These properties, in turn, significantly affect the mechanical properties and the cost of the resulting parts. While PREP powder is currently preferred for the freeform fabrication of critical aerospace components, the cost and availability of GA powder is at present much better than that of PREP. This paper will discuss an extensive study in which static properties as well as low cycle fatigue of coupons fabricated via the Electron Beam Melting (EBM) process using GA and PREP powders are compared. This study aims to determine whether or not parts produced with a finer GA powder can achieve equivalent mechanical properties as parts produced with standard PREP powder.

9:45 AM

Experimental Analysis Of Porosity Formation In Laser-Assisted Powder Deposition Process: *Liang Wang*[1]; Phillip Pratt[1]; Sergio Felicelli[1]; Haitham Kadiri[1]; Paul Wang[1]; [1]Mississippi State University

Porosity has been investigated in samples produced by the Laser Engineered Net Shaping (LENS) process. Different powders were employed including stainless steel SS410, SS316, and carbon steel AISI 4140. The effects of process parameters (e.g. laser power and scanning speed) and the metallic powder characteristics on the porosity formation were studied. The volume fraction, number density, and size distribution of porosity were characterized with x-ray computed tomography and optical metallography. Both interlayer porosity due to lack of fusion and intralayer porosity are identified. The results demonstrate the sensitivity of process parameters and powder quality on the porosity formation. The mechanisms of porosity formation in LENS process are discussed in detail.

10:10 AM

Impact Of Physical Phenomena During Selective Laser Melting Of Ferrous Powders: *Marleen Rombouts*[1]; Jean-Pierre Kruth[2]; Ludo Froyen[2]; [1]VITO; [2]Katholieke Universiteit Leuven

Broadening the range of materials that can be processed by selective laser melting (SLM) to high-density parts in a more systematic way than by trial-and-error requires a fundamental knowledge of the process. In this study the impact of physical phenomena during SLM of ferrous powders has been examined by finite element modeling and experiments. The thermal model reveals the effect of process parameters and of the change in material properties upon melting - like thermal conductivity, density, laser absorptance and heat release – on the temperature profile and on the melt pool dimensions. Experiments have indicated that even small alloying additions like carbon, copper and deoxidizers such as silicon and titanium have a large impact on the melt pool behaviour during SLM. Their impact is evaluated by roughness, density and microscopic analysis after SLM.

10:35 AM Break

10:50 AM

Materials Issues in Indirect Selective Laser Sintering and Post Processing of Ferrous Components: Phani Vallabhajosyula[1]; *David Bourell*[1]; [1]University of Texas

Selective Laser Sintering (SLS) is an additive manufacturing process in which three dimensional parts may be created directly from a computer solid model using part powder and a scanning laser beam. In indirect SLS, powder is mixed with a transient binder. A model for infiltration of an SLSed tool steel part with a cast iron is presented. Included is prediction of the equilibrium solid fraction in the final part at the infiltration temperature based on carbon diffusion from the infiltrant into the brown part particulate. The effects of pre-sintering and excess infiltrant are also developed. The model was tested using Laserformtm A6 tool steel powder infiltrated with ASTM A532 white cast iron. In some cases guided by Ashby densification maps, pre-sintering of the tool steel green part was performed to increase the initial relative density of the solid metal. This research project was sponsored by NSF grant # DMI -0522176.

11:15 AM
Development of Processing Parameters for New Metallic Alloys Built Using Additive Layered Manufacturing Techniques: *Robert Deffley*[1]; Manuelle Carnot[1]; Robert Sudamore[2]; Iain Todd[1]; [1]University Of Sheffield; [2]TWI: The Welding Institute

Additive layer manufacturing in metals and alloys offers much to the engineer in terms of design freedom but its application is, in practice, often restricted by the limited number of metallic materials available for use in the machines. In this paper we apply design of experiments (DOE) to the development of processing parameters for the nickel alloy 718 in order to speed up the definition of suitable processing conditions. The DOE is based on parameters developed by consideration of the fundamental expressions governing heat flow and solidification and the results of this work will be presented and discussed.

11:40 AM
Spatial Control Of Crystal Texture By Laser DMD Process: *J. Choi*[1]; J. Mazumder[2]; B. Dutta[1]; [1]POM Group; [2]University of Michigan

Turbine blades with controlled textures such as directionally solidified and single crystalline have proven to have much improved ductility and longer thermal and fatigue life. It has been further reported that the benefits of single-crystal over conventionally cast as well as directionally solidified components critically depend on avoiding the introduction of defects, such as stray grains, freckles, or deviations from the required crystal orientation. Laser-based direct metal deposition (DMD) process equipped with proper sensors and NC devices helps in overcoming those hurdles to fabricate the blades with controlled texture. It appears that thermal control to provide uniform heat flow as well as spatial control of crystal texture by process feedback control is essential. The paper discusses about how to establish process conditions and thermal control requirements, development of a laboratory scale DMD process for spatial control of crystal texture, and mechanical properties of texture-controlled Ni-based superalloy turbine blade components.

12:05 PM
Thermal Behavior of WC-Co Cermets During the LENS® Process: *Yuhong Xiong*[1]; William Hofmeister[2]; Zhao Cheng[3]; John Smugeresky[4]; Jean-Pierre Delplanque[1]; Baolong Zheng[1]; Jonathan Nguyen[1]; Enrique Lavernia[1]; Julie Schoenung[1]; [1]University of California, Davis; [2]University of Tennessee Space Institute; [3]Earth Mechanics Inc.; [4]Sandia National Laboratories

The Laser Engineered Net Shaping (LENS®) process has been used to fabricate a broad range of materials, including WC-Co cermets. To better explore the potential of this process to cermets, there is a need to understand their special thermal behavior and the relevant effects on the microstructure. In this study, dense WC-Co cermets were produced by the LENS® process. In-situ high-speed thermal imaging was applied to determine temperature gradients and cooling rates in the vicinity of the molten pool created by the laser. The experimental results were compared with finite element method simulation. The entire thermal behavior of this process was predicted by the finite element modeling. The thermal behavior study was also correlated to the microstructure of WC-Co cermets.

Materials Processing Fundamentals: Deformation 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 AM   Room: 2016
February 17, 2009   Location: Moscone West Convention Center

Session Chair: Prince Anyalebechi, Grand Valley State University

8:30 AM
Enhancing Tensile Response of Sn Using Cu at Nano Length Scale and High Temperature Extrusion: *Md. Alam*[1]; Manoj Gupta[1]; [1]National University of Singapore

In the present study, 1.1 volume percent of nano size copper was incorporated into pure tin using hybrid microwave sintering assisted powder metallurgy route. Microwave sintered samples were extruded both at room temperature and at 230 °C. Microstructural characterization studies were conducted on the extruded samples to investigate the distribution characteristics of secondary phase and grain morphology. Room temperature tensile test results revealed that hot extruded Sn-Cu samples exhibited higher strengths (~ 41%, in case of 0.2% yield strength and ~ 38%, in case of ultimate tensile strength) and ductility (~ 15%) when compared to room temperature extruded samples. On the contrary, the tensile properties of pure tin remained independent of extrusion temperature. An attempt is made in this study to correlate the effect of extrusion temperature on the microstructural evolution and tensile properties of Sn-Cu solder.

8:45 AM
Deformation Induced Ferrite Transformation In Low Carbon Cr-V Steels: *Martin Bühler*[1]; Teresa Perez[1]; Victoria Ramos[1]; [1]Tenaris Siderca R&D

The austenite to ferrite phase transformation kinetic of two low carbon steels containing Cr and different levels of V and N was studied. Different tests were performed on a Gleeble 3500 thermomechanical simulator in the temperature range 700°C-1100°C after solution treating. Strain induced transformation tests were performed in order to study the formation of ferrite at high temperatures. Dilatometric measurements were also carried out to analyze the phase transformation kinetic without deformation. DIFT was observed at very high temperatures in the V-N microalloyed steel. The influence of this transformation on the hot ductility was analyzed.

9:00 AM
Effect of Ordering on Cold Workability of Fe-6.5wt%Si Alloy: *Yong Liang*[1]; Junpin Lin[1]; Feng Ye[1]; Yanli Wang[1]; Guoliang Chen[1]; [1]University of Science and Technology Beijing

Fe-6.5wt%Si alloy has excellent soft magnetic properties compared with conventional silicon steel. But it is very brittle at room temperature due to appearance of ordered phases, like B2 and $D0_3$. In this paper, we investigated the effect of ordering on cold workability of this alloy. Methods of rapid quenching and hot rolling were utilized by suction casting system and rolling machine. It is found that rapid quenching and hot rolling could decrease the degree of order due to the very high solidification rate and plastic deformation respectively. The rapid quenched sheet and hot rolled sheet could be cold rolled without crack. Microstructure, structure, micro hardness, and orientation texture were investigated by optical microscope, X-ray diffraction, scanning electron microscope, micro hardness indenter before and after cold rolling.

9:15 AM
Effects Of Process Path On Texture And Microstructure In ECAE Processed And Rolled High Purity Nb: *Shreyas Balachandran*[1]; Karl Hartwig[1]; Derek Baars[2]; Thomas Bieler[2]; [1]Texas A&M University; [2]Michigan State University

High purity Nb is used to fabricated advanced superconducting radio frequency cavities for future particle accelerators. One possible approach to forming cavities efficiently is to use equal channel angle processing to achieve a fine grain size and a desirable texture in the material. Different ECAE processing paths were used on medium and high purity Nb ingots to examine the effects of different ECAE processing routes on recrystallized textures of material rolled to 90% reduction. Three components of texture were commonly observed in widely

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differing volume fractions that depended on the processing history: a punctuated gamma fiber with 6 evenly spaced peaks, an orientation that is near rotated cube, but often split into two symmetric peaks, and symmetric orientations that are just off the alpha fiber. Correlations between the magnitude of shear in particular ECAE process orientations andresulting textures have been identified.

9:30 AM
Elimination Of Crystallinity In Fractured Surface Of Medium Carbon Low Alloy Steel Plates Through Process Improvement: *Ram Avtar*[1]; Gangeshwar Singh[1]; [1]Steel Authority of India Ltd

High tensile thick plates (>80mm) of medium carbon low alloy steel, when heat treated, manifested higher percentage of Crystalline area (>20%) in fractured surface. The genesis of higher Crystallinity in fracture was examined. It was observed that the thicker plate undergone with slower rate from Soaking Temperature to Ms Temperature on cooling after austenising during oil quenching and thereby quenched microstructure had upper bainites. During tempering of as quenched bainite, additional precipitation took place at the lath boundaries of bainite. This led to weakening of grain boundaries and aided easy crack propagation during fracture and resulted in formation of higher percentage of crystalline area in fractured surface. Enhancement in cooling rate of austenised plate by switching over from oil quenching to water quenching practice resulted in complete transformation of austenite into martensite and produced ductile fracture (zero Crystallinity). Plates conformed improvement in mechanical properties between 2-5%.

9:45 AM
Challenges Of Producing Quality Construction Steel Bars In West Africa: Case Study Of Nigeria Steel Industry: Sanmbo Balogun[1]; David Esezobor[1]; Samson Adeosun[1]; *Olatunde Sekunowo*[1]; [1]University of Lagos

The production of quality high-yield reinforcing steel bars has recently received worldwide attention due to its important contribution to GDP index. In developing country such as Nigeria, empirical studies have shown that bars produced through conventional rolling requires appropriate modification of its chemical composition in order to obtain the desired mechanical properties. However, the high cost factor involved in composition adjustment makes such approach unattractive. Rather, the application of the combination of controlled rolling and controlled cooling systems proves to be the best option. This system also, requires some variations in processing parameters to suit individual plant production peculiarities. In this paper attempt is made to study the production challenges and opportunities the steel millers are facing in Nigeria. Previous works in this area were also reviewed with a view to charting the way forward. Experimental studies and process monitoring were carried out at some designated rolling mills in Nigeria.

10:00 AM Break

10:15 AM
Influence Of Ta Microstructure On Co-Deformability Of A Ta Layer Embedded In Cu: *Shreyas Balachandran*[1]; Karl Hartwig[1]; Taeyoung Pyon[2]; Derek Baars[3]; Thomas Bieler[3]; [1]Texas A&M University; [2]Luvata; [3]Michigan State University

Ta sheets are used as diffusion barriers in some niobium-tin superconductors to prevent contamination of stabilizer copper by tin. The ideal diffusion barrier thickness of 2-3 microns is rarely achieved because of Cu-Ta interface instabilities that occur during wire drawing. These instabilities lead to premature thinning and fracture of the Ta layer resulting in poor superconductor performance.Ta sheets produced from ECAE processed Ta bars show improved Cu-Ta co-deformation characteristics leading to lesser interface roughness. Ta with improved co-deformability enables fabrication of wires with thinner Ta layers and a reduction in the amount of Ta needed for the diffusion barrier component. The effects of initial and evolving Ta microstructure on Cu-Ta interface roughening phenomena will be discussed.

10:30 AM
Microstructural Evolution during Spark Plasma Sintering of Ni and W: Matthew Luke[1]; Jeffrey Perkins[1]; William Windes[2]; Darryl Butt[1]; *Megan Frary*[1]; [1]Boise State University [2]Idaho National Laboratory

Spark plasma sintering (SPS) is a novel processing technique for consolidating metal powders. As compared to traditional sintering techniques (e.g., pressureless sintering and hot pressing), SPS can produce fully dense components at lower sintering temperatures and with significantly shorter sintering times. We have studied how processing parameters such as applied pressure, sintering temperature and hold time affect the densification and microstructural evolution in both nickel and tungsten. Electron backscatter diffraction is used to characterize the grain size, grain boundary types, and crystallographic texture of the both materials. The microstructures that result from SPS are found to vary significantly from those of other processing techniques. SPS processing parameters can be adjusted to achieve microstructures with different grain sizes and distributions of grain boundaries. Based on the results, spark plasma sintering can be used to create a wide variety of metal-matrix composite materials with tailored microstructural properties.

10:45 AM
Preliminary Evaluation of Spark Plasma Extrusion: K. Morsi[1]; *A. El-Desouky*[1]; [1]San Diego State University

The interest in spark plasma sintering has been growing considerable over the past few years. This has been brought about by the unique advantages of the process, which include reduced sintering temperatures and times and the production of materials with unique microstructures and properties. Despite its current reputation as an outstanding process that has solved major problems such as nanopowder consolidation, it has so far been largely limited to the manufacture of simple shapes, due to its inherent configuration. In this paper we present preliminary results on spark plasma "extrusion" that can allow the production of extended geometries via electric-current processing. Preliminary results on the processing and microstructure of spark plasma extruded aluminum is discussed.

11:00 AM
Preparation of Cu-12Al Alloy Wires with Continuous Columnar Crystals and the Deformation Behavior in Dieless Drawing: *Xuefeng Liu*[1]; Yuhui Wu[1]; Jianxin Xie[2]; [1]Advanced Materials and Technologies Institute, University of Science and Technology Beijing; [2]State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing

Cu-12%Al alloy (mass fraction) wires with smooth surface and continuous columnar crystals in straight grain boundary can be prepared by continuous unidirectional solidification process under melting temperature 1170°, drawing speed 45mm/min and cooling water flow rate 900L/h. No recrystallization occurs in Cu-12%Al alloy with continuous columnar crystals during continuous dieless drawing under drawing speed 1.0~1.4mm/s and deformation temperature 600~900°. Under the condition of drawing speed 1.0mm/s and deformation temperature increased from 600° to 900°, the grain boundary of Cu-12%Al alloy with continuous columnar crystals gradually turned to be curved during dieless drawing. Under the condition of deformation temperature 600°, no obvious influence took place on the microstructures of the alloy during dieless drawing at drawing speed of 1.1~1.2mm/s, while the grain boundary became curved at drawing speed of 1.3~1.4mm/s. The deformation behavior of dieless drawing of Cu-12%Al alloy wires with continuous columnar crystals present single crystal like deformation behavior.

11:15 AM
The Effect of Cold Working on the Deformation Induced Martensite (DIM) and Degree of Sensitization (DOS) of Austenitic Stainless Steel: *Anil Kumar*[1]; [1]National Institute of Foundry and Forge Technology

Stainless steel possesses good mechanical properties combined with a high corrosion resistant. The cold rolled stainless steel leads high dislocation densities, enhanced residual stress and strain and produced metastable martensite phase. The deformation induced martensite and degree of sensitization behavior of austenitic stainless steel (AISI 304) is greatly influenced by several metallurgical factors, such as the chemical composition, the degree of prior deformation, grain size, and the aging temperature and time. The percentage deformation induced martensite behavior of the austenitic stainless steel (AISI 304) has been investigated after aging at various temperatures from 500°C to 700°C for 1 to 30 hours and also evaluate the degree of sensitization behavior on the cold rolled reduction in thickness from 0% to 60% at 500°C for 30 hours of austenitic stainless steel (AISI 304). This paper investigates the co-relation between deformation-induced martensite and degree of sensitization.

11:30 AM
Seeking Relation Of Dislocation Substructure To Recrystallized Grain Orientation In High Purity Single Crystal Niobium: *Derek Baars*[1]; Kai Wang[1]; Chris Compton[2]; Tom Bieler[1]; Wenjun Liu[3]; Rosa Barabash[4]; Gene Ice[4]; [1]Michigan State University; [2]National Superconducting Cyclotron Laboratory, Michigan State University; [3]Advanced Photon Source, Argonne National Laboratory; [4]Oak Ridge National Laboratory

Manufacturing superconducting radio frequency (SRF) cavities from single crystal niobium is being investigated as an alternative to polycrystalline niobium, as single crystal sheets may be cut directly from the purified ingot, eliminating the cost of forging and rolling ingots into polycrystalline sheet. Normal cavity forming steps are approximated with two groups of samples: 1) different crystal orientations deformed by uniaxial tension, cut, and welded, 2) the same crystal orientation rolled to various reductions and then incrementally heat treated. Active slip systems, dislocation substructures, and recrystallization will be examined by depth-resolved x-ray and orientation imaging microscopy, compared to crystal plasticity model predictions, and the possible influence of dislocation substructure on the orientations of recrystallized grains investigated.

Mechanical Behavior of Nanostructured Materials: Plasticity and Deformation Mechanisms at Small Length Scale 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: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Xinghang Zhang, Texas A & M University; Andrew Minor, Lawrence Berkeley National Laboratory; Xiaodong Li, University of South Carolina; Nathan Mara, Los Alamos National Laboratory; Yuntian Zhu, North Carolina State University; Rui Huang, University of Texas, Austin

Tuesday AM Room: 3024
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Andrew Minor, Lawrence Berkeley National Laboratory; Brad Boylce, Sandia National Laboratory

8:30 AM Invited
Plastic Flow In Nanoscale Pillars: *Evan Ma*[1]; [1]Johns Hopkins University

We have conducted in situ tests of metallic pillars with nanoscale dimensions in a transmission electron microscope. This technique is capable of spatially and temporally resolving the plastic flow, correlating the measured force-displacement response (such as pop-in) at ~0.3 µN and <1 nm resolutions with time-resolved TEM images (movies). Also, one observes the exact sample geometry and punch location, which is important for interpreting the test data. The metallic pillars we tested have nanocrystalline or even sub-nanometer (amorphous) internal structures, and the pillars themselves have dimensions in the nano-regime. We can then reveal what happens to strength and plasticity in such extreme "nanostructured" cases. In addition to the expected high strength, we also observed very large plastic strains, without catastrophic instability that often occurs in large samples and conventional tests. Insights regarding flow defects and flow mechanisms are discussed, together with hints from molecular dynamics simulations.

8:50 AM Invited
Progress in Understanding the Size-Affected Flow Behavior of Microcrystals: *Dennis Dimiduk*[1]; Michael Uchic[1]; Ed Nadgorny[2]; Satish Rao[3]; Christopher Woodward[1]; [1]US Air Force Research Laboratory; [2]Michigan Technological University; [3]UES, Inc

Recently, experimental studies revealed that micrometer-scale crystals show strengthening effects, even at high initial dislocation densities. Their flow behavior is qualitatively characterized by four distinct attributes: (i) a sharply rising flow stress with decreasing sample size that follows power-law scaling (scaling exponent approximately -0.4 to -1.0); (ii) stochastic variation in flow stress that exhibits wider scatter at smaller sample sizes; (iii) intermittent flow as revealed by either repeated load drops or constant-stress flow avalanches; and (iv) an extended (>1% strain) elastic–plastic transition characterized by an average strain-hardening rate, $H > G/200$, where G is the shear modulus. Explanations for these phenomena include "dislocation starvation", formation of an excess dislocation density, strengthening of dislocation sources, size-limited dislocation generation, and a statistical alteration of dislocation forest mechanisms. The present work uses both three-dimensional dislocation simulations and experiments on microcrystals to evaluate the relevancy of these mechanisms to various materials and sample sizes.

9:10 AM
Plastic Size Effects In Ni-W Nanocrystalline Nano-Pillars: *Julia Greer*[1]; Dongchan Jang[1]; [1]California Institute of Technology

When microstructural features or sizes of materials are reduced to nanometer scale, they exhibit different behaviors from bulk. Typical example is "smaller is stronger" manifested by high strengths attained during deformation of nano-sized single crystals. While specific plasticity mechanisms remain controversial, this strengthening is attributed to greater contribution of surfaces and interfaces. Conventional strengthening mechanisms such as Hall-Petch may not be valid at nanometer scale. In this study combined effect of internal nano-structure and sample size on plasticity is investigated. These factors have been investigated separately; combination of their effects has never been reported. We present results of uniaxial compression and tension of Ni-W nano-pillars with grain sizes of 60 nm and diameters down to ~ 100 nm fabricated using Focused Ion Beam (FIB). Pillars are compressed in in-situ nanoindenter inside SEM chamber under displacement rate control. Site-specific TEM analysis reveals microstructural changes occurring as a result of mechanical deformation.

9:25 AM Invited
Size Effects in Micro-Pillar Compression and Nanoindentation: Hongbin Bei[1]; Sanghoon Shim[1]; Michael Miller[1]; George Pharr[1]; *Easo George*[1]; [1]Oak Ridge National Laboratory

Size-dependent strength has been measured both in micro-pillar compression and nanoindentation. Most of the micro-pillars reported on in the literature are produced by focused ion beam (FIB) milling. We report here results on single-crystal micro-pillars produced by a different technique, directional solidification. Since our as-grown pillars behaved like dislocation-free materials and yielded at the theoretical stress, we were able to systematically pre-strain the pillars before compression and study the effects of initial dislocation density and pillar size on pillar behavior. The directionally solidified pillars are also compared to directionally solidified and FIBed pillars to evaluate possible effects of FIB damage. Finally, we discuss an interesting indentation size effect (ISE) that we discovered recently by studying pop-in behavior during nanoindentation with spherical indenters. This ISE is based not on the measured hardness, as in conventional ISE, but rather on the stress to initiate dislocation plasticity.

9:45 AM
Uniaxial Compression of FCC Au Nano-Pillars: The Effects Of Prestraining And Annealing: *Seok-Woo Lee*[1]; Seung Min Han[1]; William Nix[1]; [1]Stanford University

The size dependence of the strength of FCC metals, as revealed by uniaxial compression of nano-pillars, suggests that plasticity is dislocation source-controlled, with fewer sources in smaller pillars producing a "smaller is stronger" effect. To further investigate this phenomenon we have studied the effects of prestraining and annealing on the deformation properties of [001] Au nano-pillars. By making pillars from an epitaxial film of [001] Au on [001] MgO, using focused ion beam machining, we are able to create both puck-shaped pillars that can be stably prestrained and pillars with a high aspect ratio, which can be tested in uniaxial compression. We find that prestraining dramatically reduces the flow strength of nanopillars while annealing restores the strength to the un-prestrained levels. These are unusual effects are not seen in bulk FCC metals, which behave in an opposite way. We discuss their possible causes in terms of dislocation densities.

10:00 AM
Strength In Small Sized Single Crystals: Discrete Dislocation Dynamics Simulations Using ParaDiS: *Meijie Tang*[1]; [1]Lawrence Livermore National Laboratory

The dislocation dynamics method is a numerical tool to quantitatively describe plasticity and strength in crystalline materials. Applications have been made using the Lawrence Livermore National Laboratory large scale parallel dislocation simulator (ParaDiS) for both bulk and finite sized single crystal systems. In these applications, the dislocation dynamics simulations are found to be powerful tools to discover new microstructure [Nature, 440, p1174 (2006)], understand strength in bulk systems, and to explore mechanisms responsible for 'the smaller, the stronger' observation in micro- and sub micro- systems in recent experiments. Latest development in coupling ParaDiS with FEM approach to simulate the strength in micro-sized single crystal pillars will be presented in this talk.

10:15 AM Break

10:25 AM Invited
Oxygen in Grain Boundaries of Aluminum: A Molecular Dynamics Study: Andreas Elsener[1]; Olivier Politano[2]; Peter Derlet[1]; *Helena Van Swygenhoven*[1]; [1]Paul Scherrer Institut; [2]Université de Bourgogne, Dijon, France

One of the important differences between simulation and experiments in grain boundary dominated metallic structures is the lack of impurities such as oxygen in computational samples. A modified variable-charge-method (Modell. Simul. Mater. Sci. Eng. 16,025006(2008)), based on the Streitz and Mintmire approach that incorporates local chemical potentials to efficiently simulate oxidation in a predominantly metallic Al environment is presented. The present work reports on the application of this method to investigate aluminum samples with dilute amounts of oxygen under load. In particular, using aluminum bicrystals with symmetrical tilt grain boundaries, the influence of the presence of Oxygen on coupled grain boundary migration is investigated. It is found that grain boundary migration requires a higher applied shear to activate when Oxygen atoms exist within the boundary. This result is rationalized in terms of the stress signature of the Oxygen within the boundary and the associated atomistic grain boundary migration mechanism.

10:45 AM
Molecular Dynamics and First Principles Studies of Pb Segregation to Al Grain Boundaries and its Influence on Mechanical Properties: *Yojna Purohit*[1]; R. Scattergood[1]; D. Brenner[1]; [1]NCSU

Using ball milling, Koch and co-workers have recently produced nanocrystalline (nc) Al-Pb alloys. The Z-contrast image of consolidated Al-1.0%Pb showed Al grain boundaries coated with Pb atoms. The Pb impurities were found to decrease the hardness of nc Al. To better understand the underlying mechanisms causing the softening, we are using molecular dynamics and first principles methods to calculate segregation energies for Pb on Al grain boundaries. Our modeling shows that the segregation is stress driven, with the Pb tending to substitute for Al sites that are otherwise under hydrostatic tension. Molecular dynamics simulations of bicrystalline and 3-d nanocrystalline structures under high strain rate have shown that Pb can suppress dislocation nucleation while at the same time thickening and disordering the grain boundaries. We believe that the latter contributes to the softening observed experimentally.

11:00 AM Invited
Correlation Between The Deformation Of Nanostructured Materials And The Model Of Dislocation Accommodated Boundary Sliding: *Farghalli Mohamed*[1]; [1]University of California

Very recently, a new model for deformation in nanocrystalline (nc) materials has been formulated. The development of the model was based on the concept that plasticity in nc-materials is the result of grain boundary sliding accommodated by the generation and motion of dislocations under local stresses. By analyzing experimental data on two nc-materials, Ni and Cu, it is shown that this model can account not only for the deformation behavior of both metals over wide ranges of conditions but also for the occurrence of nanoscale softening.

11:20 AM
Micro-Scratch Characterization Of Strength In Nano-Crystalline Metals: Luke Nyakiti[1]; *Alan Jankowski*[1]; [1]Texas Tech University

Tensile testing can provide the detailed plastic behavior from deformation at yielding to the ultimate strength. The power-law dependence of strength on strain rate provides a measure of the rate sensitivity. In general, tensile strength increases for many cubic metals as grain size decreases from the micro-scale into the nano-scale regime. However, many nano-crystalline metals are prone to localized plastic deformation or even brittle failure. As such, tensile strengths may appear well below the true upper-bound values. This drawback can make a quantitative interpretation of the strain-rate sensitivity quite difficult. As an alternative, the method of micro-scratch testing is used to evaluate the upper bound strength through micro-hardness measurements. By varying the velocity of the indent test under constant load, the width of the scratch reveals the rate dependence of hardness. New test results are presented for nanocrystalline cubic metals as gold alloys with grain sizes less than 10 nm.

11:35 AM
Study Of Plasticity In Small Size Tensile Samples: Rick Lee[1]; *Amit Ghosh*[1]; [1]University of Michigan

Investigation of tensile behavior of small size tensile samples were carried out using small tensile stage within the chamber of a scanning electron microscope. Sample cross sections in the range of 300 - 2,000 μm2 were machined by FIB. The micron-size samples exhibited strain bursts of appreciable size in comparison to relatively smooth stress-strain curves for larger samples. The strain bursts were correlated with slip steps observed on sample surface. The surface emergence of slip steps, their arrest, followed by strain hardening, define an intermittent slip process for small size samples. This surface based slip leads to large amount of strain hardening in Inconel 625 and moderate amount of hardening in Ti-1100, but greater than large size samples. The high hardening rate is attributed to increasing fraction of surface atoms in small size specimens to penetrate through during slip, the strength level in the nanoscale size range approaching theoretical strength.

11:50 AM
Synthesis Of Bulk Nanolaminate Materials With Accumulative Roll Bonding: *Rainer Hebert*[1]; Girija Marathe[1]; Jyothi Suri[1]; [1]University of Connecticut

Severe plastic deformation techniques provide an opportunity to study the relationship between mechanical properties and microstructural length scales. During accumulative roll bonding (ARB) of metallic multilayers, for example, the individual layer thickness continuously decreases by as much as four orders of magnitude. Aside from the layer thickness, the microstructure within the layers defines a second length scale. Nanoindentation studies with ARB-processed as-received Mo foils reveal a cyclic hardness change during processing to equivalent strains of about -10. Cu-Ni multilayers reveal necking of the Ni layers that depends on the work-hardening behavior of the Cu and Ni layers. Necking of elemental layers renders the top-down synthesis of nanolaminate materials more difficult. A continuum mechanics model along with experimentally determined strain hardening data enable the prediction of the onset of diffuse necking. The results highlight the relation between mechanical properties and microstructure evolution for the ARB synthesis of bulk nanolaminate materials.

12:05 PM
Tensile Deformation and Fracture Mechanism of Bimodal Al-Mg Alloy: *Zonghoon Lee*[1]; Velimir Radmilovic[1]; Byungmin Ahn[2]; Enrique Lavernia[3]; Steven Nutt[2]; [1]Lawrence Berkeley National Laboratory; [2]University of Southern California; [3]University of California, Davis

Bimodal bulk Al-Mg alloys, which were comprised of nanocrystalline grains separated by coarse grains, achieved balanced mechanical properties of enhanced strength and reasonable ductility and toughness compared to conventional counterparts and other nanocrystalline metals. However, the underlying deformation and fracture mechanism of the bulk bimodal metals have not been fully elucidated because of lack of unambiguous evidence based on direct observations in various scale range. We investigated cross-sections of tensile fractures of bimodal Al-Mg alloys at the micro and macro-scale using TEM, SEM equipped with FIB and optical microscopy. The direct observation revealed nanoscale voids and preserved micro-cracks near the tensile fracture surfaces successfully. It is evident that the incorporation of ductile coarse grains effectively impedes propagation of micro-cracks and results in enhanced ductility and toughness while retaining high strength. The findings may provide insights of further design of bimodal and moreover multiscale microstructures in ultra-fine grained and nanoscale regime.

12:20 PM
The Effect of Starting Microstructure on the Creation of Ultra-Fine Grained Ti-6Al-4V via Multi-Axis Forging: *Richard Didomizio*[1]; Andrew Deal[1]; Judson Marte[1]; P.R. Subramanian[1]; Steve Buresh[1]; Radhakrishna Bhat[1]; [1]GE Global Research

Multi-axis forging (MAF) under near-isothermal conditions was used to produce ultra-fine grained (UFG) structures in Ti-6Al-4V alloys. Mill-annealed and globurized microstructures were used as the starting material for the MAF processing. The resulting ultra-fine grained structures were compared with high resolution scanning electron microscopy and electron backscatter diffraction (EBSD). Using EBSD, the evolution of colonies of the alpha phase and the texture of both the alpha and beta phases were tracked from the starting structure through final processing. The flow responses of the UFG materials were obtained in both tension and compression. The salient microstructural features and flow behavior will be presented, along with resulting implications for superplastic behavior of the UFG Ti-6Al-4V alloys.

Microstructural Processes in Irradiated Materials: Advanced Oxide Dispersion Strengthened Ferritic Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee
Program Organizers: Christophe Domain, Electricite De France; Gary Was, University of Michigan; Brian Wirth, University of California, Berkeley

Tuesday AM — Room: 2008
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Akihiko Kimura, Kyoto University; Brian Wirth, University of California, Berkeley

8:30 AM Invited
Recent CEA Results on the Development of Nanoscale Oxide Dispersion Strengthened Ferritic Alloys for Nuclear Applications: *Yann de Carlan*[1]; Mathieu Ratti[1]; Marie-Hélène Mathon[1]; Patrick Olier[1]; Cyril Cayron[1]; Joël Ribis[1]; Philippe Pareige[2]; Arnaud Monnier[1]; Laurent Forest[1]; Xavier Averty[1]; [1]CEA; [2]GPM

Ferritic/martensitic ODS alloys are considered as promising materials for different nuclear applications. They exhibit very low swelling under irradiation and very good creep properties. In the seventies, different alloys were developed to be used as cladding materials in Sodium Fast Reactors (SFR) but the qualification of this type of materials has appeared long and difficult. In the framework of the studies on GENIV reactors, important means are now dedicated at CEA for the development of new F/M ODS alloys. The aim of this paper is to present the design of these new materials, their manufacture and the dedicated program to assess them as cladding materials. It includes the basic studies on the formation mechanisms of nano-oxides, the definition and the optimization of the fabrication route, the welding studies to evaluate the different joining processes and also all the experiments to insure the stability of the materials under irradiation.

9:00 AM Invited
Analytical Electron Microscopy of Nano-Structured Ferritic Alloys: *James Bentley*[1]; David Hoelzer[1]; [1]Oak Ridge National Laboratory

At a scale intermediate to those of atom probe tomography and “bulk” techniques such as small-angle neutron scattering, analytical (transmission) electron microscopy (AEM) of mechanically alloyed nano-structured ferritic alloys (NFAs) has provided much useful information for structure-property-processing correlations. The NFAs include MA957, ORNL-developed 14YWT (Fe-14.2%Cr-1.95%W-0.22%Ti-0.25%Y_2O_3) and prototypical 12YWT (12%Cr). Energy-filtered transmission electron microscopy (EFTEM) methods have been emphasized for reliably characterizing the oxide nano-clusters (typically with diameters less than 4 nm and concentrations exceeding 10^{23} m^{-3}) that are responsible for the exceptional mechanical properties of these materials. X-ray microanalysis, especially spectrum imaging in the scanning transmission mode, has been a useful complement to EFTEM methods. AEM characterization of irradiated specimens and of tensile- and creep-tested specimens will be discussed along with the role of AEM in identifying undesirable processing conditions and aiding the selection of more optimum fabrication protocols.

9:30 AM
Microstructural Characterization of Irradiated ODS and Fe-Cr Alloys: *Vanessa de Castro*[1]; Sen Xu[1]; Sergio Lozano-Perez[1]; Emmanuelle Marquis[1]; Mike Jenkins[1]; [1]University of Oxford

Reduced activation ferritic-martensitic steels are candidate structural materials for fusion reactors. Strengthening the steel with a homogeneous dispersion of oxide nanoparticles such as Y_2O_3 could help to rise the operating temperature of these materials and lower the rate of damage accumulation in the steel. These oxide dispersion strengthened steels(ODS)are produced by pulvimetallurgical routes which lead to complex microstructures. This work describes the microstructure after ion irradiation of an ODS/Fe12Cr and a reference Fe12Cr alloy, produced by pulvimetallurgy, with the microstructure present in a Fe11Cr alloy produced by casting. The alloys were irradiated at 300 and 500°C with 0.5 and 2 MeV Fe^+ ions up to doses of 10^{16} ions/cm^2. The distribution, size and density of the defects induced in these materials are compared. The stability of the oxide nanoparticles dispersion is also discussed. This research has been supported by FP6 Euratom Research and Training Programme on Nuclear Energy.

9:50 AM
Radiation Induced Segregation to Nano-Particles and Grain Boundaries in 9CrODS Steel after Low Dose Irradiation: *Alicia Certain*[1]; Kevin Field[1]; Kumar Sridharan[1]; Todd Allen[1]; Jeremy Busby[2]; Mike Miller[2]; [1]University of Wisconsin-Madison; [2]Oak Ridge National Laboratory

9Cr ferritic-martensitic (F-M) alloys show promise for nuclear applications due to their resistance to void swelling compared to austenitic alloys and improved toughness compared to 12Cr F-M alloys. However, these alloys tend to have low creep strength. This can be circumvented by adding Y-Ti-O nanoclusters that act as pinning points for dislocations. A 9Cr oxide dispersion strengthened (ODS) steel was irradiated with protons to 1 dpa at 525°C to investigate oxide stability. The radiation-induced segregation response of the alloys was investigated using EDS and atom probe tomography along prior austenite grain boundaries and in the yttrium-titanium oxide nano-particles contained within the matrix of the ODS steel. No segregation was observed at the particle or grain boundary interfaces in the unirradiated condition, but Cr enrichment was observed at the particle-matrix interface for the 1 dpa irradiated steel.

10:10 AM Break

10:30 AM Invited
Atomic Scale Characterization of ODS Steels by Atom Probe Tomography: *Emmanuelle Marquis*[1]; [1]University of Oxford

Reduced activation ferritic and ferritic-martensitic steels (RAFMS) are promising structural materials for the first wall and blanket of future fusion reactors. In order to improve the high temperature properties of these steels, oxide-dispersion strengthened (ODS) versions were processed with the addition of oxide precipitates that provide dislocation pinning points and remain stable up to temperatures close to the melting point. Controlling material properties during irradiation requires detailed understanding of the role of the defect sinks, i.e. nanoscale particles, grain boundaries, dislocations, etc. which in turn implies a detailed knowledge about the internal structure, chemistry, and interfacial structure of these microstructural features. The role of atom probe tomography for the 3-D atomic scale characterization of ODS steels will be discussed, focusing on the internal structure of the nanoscale particles and grain boundary chemistry before and after irradiation in different ODS Fe-Cr alloys.

11:00 AM
Irradiation of Nanoclusters: *Michael Miller*[1]; David Hoelzer[1]; Kaye Russell[1]; Chong Long Fu[1]; [1]Oak Ridge National Laboratory

Atomic displacement cascades produced during neutron or ion irradiations can induce mechanisms that can potentially destabilize or destroy nanoclusters and precipitates, change the vacancy and interstitial atom distribution, and thereby degrade the desired properties of materials. Atom-probe tomography has been used to determine, with atomic scale resolution, the solute distribution associated with titanium-, oxygen-, and yttrium-enriched nanoclusters in mechanically-alloyed, nanostructured ferritic alloys before and after high dose irradiation. This is the initial stage towards a fundamental understanding of the remarkable stability of these alloys when exposed to extreme conditions. 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.

11:20 AM
A Density Functional Theory Study of Formation of Y-Ti-O Nanoclusters in Nanostructured Ferritic Alloys: *Yong Jiang*[1]; John Smith[1]; G. Robert Odette[1]; [1]University of California, Santa Barbara

Atom probe tomography shows that nanostructured ferritic alloys are dispersion strengthened by far from equilibrium Y-Ti-O nanofeatures. The nanofeatures and large excess quantities of dissolved O persist even after prolonged high temperature aging. Density functional theory (DFT) calculations were used to study the energies, structures and formation mechanisms of dissolved Y, Ti and O solutes and small Y-Ti-O nanoclusters (NC). Y and O dissolve during mechanical alloying of Y_2O_3 with metal powders, requiring solution energies of about 4 eV/atom provided by the ball milling. Substitutional Ti and Y and interstitial O have high solution energies, but O-O, Y-O and Ti-O pairs are strongly bound, and constitute NC building blocks. The energy decreases upon further clustering and is about -5.1 eV for a Y_2TiO_3 NC. NC formation can take place without the energetic assistance of pre-existing vacancies. The O-O pairs and O-O-Y/Ti complexes also increase the solubility of O.

11:40 AM
Comparison of Microstructures of Commercial ODS Alloys Using Local Electrode Atom Probe and Transmission Electron Microscopy for Irradiation Applications: *Peter Hosemann*[1]; Erich Stergar[2]; Christiane Vieh[2]; Patricia Dickerson[1]; Nicholas Cunningham[3]; Robert Odette[3]; Harald Leitner[2]; Stuart Maloy[1]; [1]Los Alamos National Laboratory; [2]Montanuniversität Leoben; [3]University of California Santa Barbara

Nanostructured ferritic/martensitic alloys have been shown to be promising candidate materials for high dose irradiation applications. The main reason for these materials irradiation tolerance is a distribution of nanometer sized stable oxide particles in the material. The work presented here used Local Electrode Atom Probe (LEAP) and Transmission Electron Microscopy (TEM) to investigate the aluminum and chromium alloyed materials PM2000 (two different grain sizes) and MA956 as well as the chromium alloyed material MA957 and an experimental alloy. The exact composition of the nanostructured oxide particles as well as their shape and distribution are discussed and compared to LEAP and TEM measurements on conventional reactor steels like HT-9. The new knowledge of these measurements are discussed in relation to radiation tolerance in comparison with results from the literature. In addition LEAP results as measured on these same materials after an ion beam irradiation experiment (1dpa, room temperature) are discussed.

12:00 PM
Effects of Atypical Particle Distributions on Grain Growth: *Zachary Royer*[1]; Ralph Napolitano[1]; Richard Lesar[1]; [1]Iowa State University

The thermal stability of oxide dispersion strengthened (ODS) steels is highly dependent on the distribution of the oxides in the matrix. We employ 2-D phase-field simulation to examine the effects of atypical particle distributions on grain growth. By varying the initial concentration and distribution of particles relative to the initial grain structure, we are developing scaling relationships to describe the interrelationship of particle distribution and the evolution of the microstructure.

Nanocomposite Materials: Characterization and Modeling of Nanocomposites I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee, TMS: Nanomaterials Committee

Program Organizers: Jonathan Spowart, US Air Force; Judy Schneider, Mississippi State University; Bhaskar Majumdar, New Mexico Tech; Benji Maruyama, Air Force Research Laboratory

Tuesday AM — Room: 3018
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Nikhilesh Chawla, Arizona State University Nathan Mara, Los Alamos National Laboratory

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8:30 AM Introductory Comments

8:35 AM Invited
Length-Scale Dependent Failure of Hierarchical Composites: *Shailendra Joshi*[1]; Yeong Sung Suh[2]; K.T. Ramesh[3]; [1]National University of Singapore; [2]Hannam University; [3]Johns Hopkins University

Nature tends to design multi-scale microstructures, using growth mechanisms to develop several levels of hierarchy, and these microstructures are efficient (strong and ductile) from the structural viewpoint. Motivated by this concept, we define hierarchical composites as heterogeneous materials comprising two or more constituent phases where at least one phase is itself a composite at a finer scale. Using these bio-inspired concepts, coupled with recent advances in nanostructured materials, we present explicit finite element analyses of artificial microstructures with multiple elasto-plastic phases, accounting for length-scale effects. The results provide insight in to topological influences on the strengthening and failure of hierarchical microstructures.

9:00 AM
A Multi-Scale Statistical Model of the Dynamic Mechanical Response of Natural Composites: *Mark Jhon*[1]; Daryl Chrzan[1]; [1]University of California, Berkeley and Lawrence Berkeley National Labratory

Nacre is a natural composite material consisting of brittle mineral platelets and an organic adhesive. It has a very high toughness relative to the properties of its component materials. The reason for this lies in nacre's rich hierarchy of structural features. For instance, individual molecules in the organic have been shown to unfold in discrete steps on the order of 10s of nanometers. In the present study, a multi-scale statistical model is introduced to address the consequences of such nanometer length-scale features on the deformation of the microstructure of nacre. A dynamic fiber-bundle model models the rate-dependent mechanical behavior of the organic, while a random fuse model connects the local and macroscopic mechanical response. Faster loading rates are found to increase the microscopic strength. Introducing microscopic hardening is found to spread the spatial extent of damage. This work was supported by the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

9:20 AM
Electron Instabilities in Inhomogeneous Nanoclusters and Nanostructured Materials: *Armen Kocharian*[1]; Gayanath Fernando[2]; Kalum Palandage[2]; James Davenport[3]; [1]California State University, Los Angeles; [2]University of Connecticut, Storrs; [3]Brookhaven National Laboratory, Upton

Exact calculations of thermodynamic properties in various cluster geometries yield level crossing degeneracies driven by interaction strength, coupling strength and temperature. The electronic configurations of the lowest energy levels control the physics of electronic instabilities and magnetic phase transitions. Rigorous conditions are found for phase transitions and crossovers which resemble a number of inhomogeneous, coherent and incoherent nanoscale phases seen recently in high Tc cuprates, manganites and CMR nanomaterials. Small bipartite and frustrated nanoclusters exhibit instabilities and phase diagrams in many respects typical for nano and heterostructured materials.[1] The calculated phase diagrams in various cluster geometries may be linked also to atomic scale tunneling experiments in high Tc cuprates, manganites and other transition metal oxides. [1]A.N. Kocharian, G.W. Fernando, K. Palandage, and J.W. Davenport, cond.-mat.: arXiv:0804.0958 (2008); Phys. Lett. A364, 57 (2007); Phys. Rev. B74, 024511 (2006).

9:40 AM
Molecular Dynamic Simulations for Effect of Polymer Chain Morphology on Mechanical Properties of Carbon Nanotube-Polymer Composites: *Zhongqiang Zhang*[1]; Don Ward[1]; Yibin Xue[1]; Hongwu Zhang[2]; Mark Horstemeyer[1]; [1]Mississippi State University; [2]Dalian University of Technology

The influence of chain length and morphology of polyethylene on the constitutive properties of single walled carbon nanotube (SWCNT) reinforced polyethylene composites is investigated using molecular dynamics simulations. Molecular models of nanocomposites are developed by embedding SWCNTs into both an amorphous and a semi-crystalline polyethylene matrix at the thermodynamic equilibrium state, in which the carbon nanotubes can be pristine or functionalized. The mechanical properties of bulk polyethylene and nanocomposites are evaluated by simulating a series of tension, compression and shear tests at various loading rates and temperatures. For pure polymer, the results show that an increase in chain length has induced non-linear proportional increases in tensile strength and Young's modulus; the elongation and viscoelastic hardening of the polymer are significantly enhanced with the increase in chain length; The crystalline morphology varies as the deformation increases, which is a novel observation in simulations and is consistent with the assumptions in open literature.

10:00 AM
Modeling of Indentation Behavior in Nanolayered Al/SiC Composites: Guanlin Tang[1]; *Yu-lin Shen*[1]; Danny Singh[2]; Nikhilesh Chawla[2]; [1]University of New Mexico; [2]Arizona State University

The indentation behavior of multilayered Al/SiC composites is studied numerically. The numerical model features the explicit composite structure on top of a Si substrate indented by a conical diamond indenter. Attention is devoted to the evolution of stress and deformation fields in the layered composite during the indentation loading and unloading processes. It is found that the layered composite, consisting of materials with distinctly different mechanical properties, results in unique deformation patterns. Significant tensile stresses can be generated locally along certain directions, which offers a mechanistic rationale for the indentation-induced internal cracking observed experimentally. The unloading process also leads to an expansion of the tension-stressed area, as well as continued plastic flow in parts of the Al layers. Implications of these numerical findings to the nanoindentation response of metal-ceramic laminates will be discussed. Simulation results on microcompression of the pillar structure will also be presented.

10:20 AM Break

10:35 AM Invited

Deformation and Failure Mechanisms of Cu/Nb Nanoscale Composites: Microstructural Analysis at the Nanoscale: *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 using 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 (5 nm Cu/Nb case) of nearly 3 GPa, and deformation during micropillar compression exceeding 20%. Through the use of Focused Ion Beam (FIB) milling, post-deformed microstructures of micropillars are examined via Transmission Electron Microscopy (TEM). Shear banding, as well as homogeneous deformation of over 10% true strain is evident at individual layer thicknesses as low as 5 nm. 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%. Plastic behavior of nanolayered composites at large plastic strains will be discussed in terms of interfacial effects on dislocation motion.

11:00 AM

Mechanical Characterization of Nanolayered Al/SiC Composites through Indentation and Microcompression Testing: Danny Singh[1]; *Nikhilesh Chawla*[1]; Guan Lin Tang[2]; Yu-lin Shen[2]; [1]Arizona State University; [2]University of New Mexico

Multilayered Al/SiC composites exhibit extremely high strength and toughness. In this paper we discuss the processing, microstructural characterization, and mechanical behavior of this novel system. The nanolaminates were processed by physical vapor deposition (PVD) using magnetron sputtering. Layer thickness and morphology was studied using a dual beam focused ion beam (FIB). The mechanical properties were characterized by nanoindentation and microcompression of "pillars." The pillars were fabricated and characterized by FIB. The effect of pillar size on the mechanical response of these materials was studied. Mechanical properties derived from microcompression were compared to conventional nanoindentation results. Finally, post-deformation microstructural analysis and modeling was carried out to provide insight into the observed deformation mechanisms.

11:20 AM

Nanomechanics of Cellulose Nanocrystal Composites: *Reza Shahbazian Yassar*[1]; Anahita Pakzad[1]; Patricia Heiden[1]; [1]Michigan Technological University

Cellulose nanocrystals are theoretically estimated to have mechanical properties comparable to carbon nanotubes. Being one of the most abundant materials in the world, cellulose has several appealing characteristics such as low cost, eco-friendly, and low density. As such, cellulose nanocrystals have attracted scientists to devote considerable efforts in order to develop cellulose-based nanocomposite materials for automotive applications. Yet no direct experimental work has been performed to measure the mechanical properties of the individual cellulose nanocrystals. In this research, we use a novel in-situ nanomechanical testing based on atomic force microscope (AFM) that operates inside a transmission electron microscope (TEM) and as a result makes the simultaneous quantitative and qualitative analysis possible. By this method, for the first time, deformation parameters including the elastic modulus, total amount of deformation, the amount of strain prior to fracture, and failure mechanisms of cellulose nanocrystals are determined.

11:40 AM

Nanoscale Near-Surface Deformation in Polymer Nanocomposites: *Devesh Misra*[1]; Qiang Yuan[1]; [1]University of Louisiana

The objective of the presentation is to elucidate the nanoscale near-surface deformation response of two polymer nanocomposite systems with significant differences in ductility during nanoscratching with a Berkovich indenter. An accompanying objective is to investigate the commonality in surface deformation behavior between nano- and microscale deformation to reinforce the underlying fundamental principles governing surface deformation. An understanding of surface deformation response is accomplished through determination of physical and mechanical properties, structural characterization and electron microscopy analysis of surface deformation tracks and residual plastically deformed structures. The deformation behavior is described in terms of physical and mechanical properties of materials notably percentage crystallinity and elastic recovery.

12:00 PM

Investigation of Nano SiC-Nickel Composite Coatings: Gholamreza Heidari[1]; mohammad mosavi khoei[1]; *Alimehran Shahhosseini*[2]; Masoud Toghraie[1]; [1]Amirkabir University of Technology; [2]University of Louisville

Composite plating is a method through which the fine particles of metallic or non-metallic compounds are co-deposited in a plated layer to improve such properties like lubrication, wear resistance and corrosion resistance. In this study, nanosize SiC particles were co-deposited with nickel from sulfamate bath using pulse and DC currents. Scanning electron microscopy, micro hardness and wear tests were carried out to characterize the coating properties. The effect of SiC nano particles on friction and wear resistance was investigated under dry sliding wear. The results showed that micro hardness and wear resistance of Ni-SiC composite coatings were increased compared to those of Ni films.

Neutron and X-Ray Studies of Advanced Materials: Small Scale and Thin Film Studies

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Titanium Committee

Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter Liaw, The University of Tennessee; Jaimie Tiley, US Air Force

Tuesday AM — Room: 3016
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Paul Zschack, Advanced Photon Source; Carol Thompson, NIU

8:30 AM Keynote

Micro-Mechanical Insights From In-Situ X-Ray And Neutron Diffraction: *Helena Van Swygenhoven*[1]; [1]Paul Scherrer Institute

With the high intensities of neutron and X-ray sources, new detector developments and X-ray micro-focusing techniques, time resolved studies of mechanical behavior of interface/surface dominated structures becomes one of the new powerful methods in materials science. In this talk recent results obtained from in-situ powder diffraction at the Swiss Light Source and at the Swiss neutron source are presented for a variety of nano-materials. In the second part of this talk, results obtained from the new in-situ Laue micro-compression device at the SLS to study the dynamics of single crystal plasticity in micron-sized single-crystals will be presented. In most studies it is assumed that the pillars made by the FIB method do not contain pre-existing strain gradients. In-situ Laue has however revealed the presence of strain gradients and misorientations at the pillar base extending well into the pillar body – all features that are known to contribute to classical hardening.

9:00 AM Invited

Cation Ordering In Thin LaSrCoO Films: *Wolfgang Donner*[1]; [1]TU Darmstadt

Cathode materials for low-temperature solid oxide fuel cells should exhibit a high oxygen permittivity at low temperatures (< 500 centigrades). It has been found that the oxygen diffusion through the cathode can be enhanced by using cation-ordered perovskites. We will present a Synchrotron x-ray study of thin (40 nm) epitaxial films of LaSrCoO on SrTiO that has been carried out under variable temperature and oxygen partial pressure. We found that the La / Sr cations undergo chemical ordering under reduction. This cation ordering is not present in the bulk material and induced by epitaxial strain from the substrate.

9:20 AM Invited

3D Spatially-Resolved Measurements of Strain Gradients in Embedded and Free Standing Mo Micro-Pillars and NiAl Matrix: *Rozaliya Barabash*[1]; Hongbin Bei[1]; Gene Ice[1]; J. Tischler[1]; Wenjun Liu[2]; Easo George[1]; [1]Oak Ridge National Laboratory; [2]Argonne National Laboratory

Spatially resolved strain distributions in the NiAl matrix and the ~550-nm Mo fibers of a NiAl-Mo eutectic were investigated by micro-beam X-ray diffraction. Position sensitive d-spacings for the individual phases were obtained from Laue patterns. For embedded Mo fibers, the measured elastic strain is consistent with the predicted thermal mismatch strain between the NiAl and Mo phases. However, when the NiAl matrix is etched back to expose Mo micro-pillars, the d-spacing increases to that of unconstrained Mo, indicating release of the compressive residual strain in the Mo fibers.

9:40 AM Invited

Gold As A Model System For Nanoscale Interconnects: Hillocking, Temperature Dependent Strength And And Low Temperature Ductility: *Ralph Spolenak*[1]; [1]ETH Zurich

Diffusion barriers limit the downscaling of classical interconnect materials such as aluminum and copper. Gold, on the other hand, does not require passivating layers and can thus be scaled down further. With regards to probes for interaction with biological material gold is also well suited. We report an in-situ synchrotron based study on gold interconnects as narrow as 20 nm, and thin films of similar dimension. Whereas thicker films exhibit hillocking under compressive stress as investigated by Laue microdiffraction, nanoscale films show an unexpectedly strong dependence of yield strength on temperature. A high low temperature yield strength is found to be correlated to an improved ductility.

10:00 AM Invited

Characterization of Group-III Nitrides with X-Ray Diffraction: *Alois Krost*[1]; Juergen Blaesing[1]; [1]Magdeburg University

During the last 15 years group-III nitrides have developed one of the most important and technological relevant semiconductor family. Unfortunately, due to the lack of homoepitaxy, most of such layer structures are far from being perfect single crystalline films but exhibit strong mosaicity.In this talk it will be shown, how a lot of information on such layers can be gathered with conventional and advanced laboratory equipment. In order to get lateral resolution down to 1 µm in the laboratory, we have designed a new convergent beam, concurrent detection diffractometer. The instrument was built by Bruker and is equipped with a rotating anode generator, a Johannson monochromator crystal for beam focusing, and a Soller slit arrangement in combination with a knife edge in front of the sample which defines the illuminated area on the sample. A large area detector from Vantec allows for rapid simultaneous detection of the diffracted intensity.

10:20 AM Break

10:30 AM Invited

In-Situ Synchrotron X-Ray Studies of (In,Ga)N Growth: *Gregory Stephenson*[1]; Marie-Ingrid Richard[1]; Fan Jiang[1]; Matthew Highland[1]; Tim Fister[1]; Carol Thompson[2]; Stephen Streiffer[1]; Paul Fuoss[1]; Ken Elder[3]; Anneli Munkholm[4]; [1]Argonne National Laboratory; [2]Northern Illinois University; [3]Oakland University; [4]Philips Lumileds Lighting Company

In-situ, time-resolved x-ray techniques can provide unique insight into the atomic-scale mechanisms occurring during materials synthesis and processing. In this talk we discuss studies of metal-organic chemical vapor deposition of (In,Ga)N thin films. While high In content is desirable for several applications, InN has a 10% lattice mismatch with the GaN substrate, and is metastable at ambient pressure, requiring a chemically active nitrogen species for growth. Using x-ray scattering and fluorescence, we have studied the coupled strain and composition changes that occur during (In,Ga)N film growth and relaxation. During InN growth, we observe that self-sustaining oscillations in phase stability can occur: islands of relaxed InN nucleate and grow; the InN islands collectively transform into elemental In droplets; the liquid In evaporates; and then another cycle of InN growth begins. These observations indicate key synthesis mechanisms for these metastable materials. Work supported by DOE under contract DE-AC02-06CH11357.

10:50 AM Invited

Spatially Resolved Structure Investigation of Periodic Arrays of GaAs Nanowires Grown on Pre-patterned GaAs[111] and Ge[111] Substrates: *Ullrich Pietsch*[1]; Andreas Biermanns[1]; Anton Davydok[1]; Jörg Grenzer[2]; Hendrik Paetzelt[3]; Volker Gottschalch[3]; Gerald Wagner[3]; [1]University of Siegen; [2]Research Center Dresden-Rossendorf; [3]University Leipzig

Nanowires (NW) with uniform thickness and defined separation can be grown on pre-patterned substrates created by etching through a thin SiNx mask. Using this route regular hole pattern with lateral spacing between 2-3 µms have been created on GaAs and Ge[111] substrates. Using these templates GaAs NWs have been grown by MOVPE at 750°C and 50mbar. The periodicity of NW ensembles has been inspected by high-resolution x-ray diffraction. The structure properties of individual NWs were studied using a focused x-ray beam provided by beamline ID1 of ESRF. The spot size of 1x2 µm² was small enough to record reciprocal space maps (RSM) of single NWs and small NW ensembles. RSMs of NWs on GaAs[111]B display the individual strain field at the bottom of NWs caused by the SiNx mask. This strain influence is negligible using Ge[111] as substrate. Here the shape of individual NWs dominates and can be probed individually.

11:10 AM Invited

X-Ray Studies of Thin-Film Thermoelectric Materials: *Paul Zschack*[1]; Colby Heideman[2]; Qiyin Lin[2]; Ngoc Nguyen[2]; Mary Smeller[2]; Clay Mortensen[2]; David Johnson[2]; [1]Argonne National Laboratory; [2]University of Oregon

Layered materials fabricated with the Modulated Elemental Reactant (MER) technique have demonstrated extremely low thermal conductivity and hold great promise for effective thermoelectric applications. These thin-films and multilayers incorporate ordered stacking of 2D hexagonal sheets that are highly textured with [00L] along the surface normal and random crystalline orientation in the plane parallel to the substrate. The extremely low thermal conductivity of these disordered, layered crystals is related to the nano-scale structural arrangement, due to enhanced interface scattering and the localization of lattice vibrations within the randomly distributed nano-crystalline regions. X-ray diffraction and imaging techniques at the Advanced Photon Source have been used to characterize the structures.

11:30 AM Invited

In Situ, Time-Resolved X-Ray Scattering Studies Of Morphology Evolution And Kinetic Relaxation During Layer-By-Layer Growth Of Complex Oxides Via Pulsed Laser Deposition: Gökhan Arikan[1]; John Ferguson[1]; Arthur Woll[1]; *Joel Brock*[1]; [1]Cornell University

Obtaining time-resolved, atomic-scale structural information of thin film deposition remains an on-going challenge. Here, we present recent studies of layer by layer (LBL) homoepitaxy of SrTiO3 (001) via pulsed laser deposition (PLD). First, we discuss single shot specular reflectivity measurements using an avalanche photodiode which provide access to changes in the surface roughness with temporal resolution comparable to the plume duration (~50e-6 sec.). Next, we discuss diffuse scattering measurements using a CCD detector operating in streak-camera mode. We simultaneously capture both the specular reflectivity and the diffuse scattering parallel to the substrate with a temporal resolution of ~0.2 seconds. These rich data sets provide information on the surface roughness, coverage, island density and size as a function of time. For example, the time scale for interlayer transport grows over the course of the first 5-10 layers and is correlated with an increase in the average distance between islands.

11:50 AM Invited

In Situ X-Ray Scattering Investigations Into The Growth Of Nanostructured Surfaces: *Paul Miceli*[1]; Chinkyo Kim[2]; Shawn Hayden[1]; Michael Gramlich[1]; Edward Conrad[3]; Rui Feng[3]; Michael Tringides[4]; Myron Hupalo[4]; Craig Jeffrey[1]; Philip Ryan[5]; [1]University of Missouri-Columbia; [2]Kyung Hee University; [3]Georgia Institute of Technology; [4]Ames Laboratory; [5]MUCAT

Because it is sensitive to both the surface and the subsurface of a sample, x-ray scattering possesses unique capabilities for exploring atomic-scale mechanisms that control the growth and formation of nanostructures at surfaces. Using the in-situ scattering facility that we developed at the Advanced Photon Source, our research has revealed unexpected behavior. For example, the formation of large vacancy clusters was discovered during the homoepitaxial growth of noble metals. Despite intense interest in film-growth mechanisms, conventional "surface-only" tools have missed these buried defects. Their existence is important for understanding the atomic-scale growth-mechanisms. Our studies of nanoscale Pb islands on Si(111) reveal anomalously fast surface kinetics as well as novel coarsening due to the breakdown of classical ripening processes, all of which derive from quantum-size-effects. These studies will be presented for a general audience in order to illustrate the utility of in-situ x-ray scattering methods. Funding: NSF, PRF, DOE

12:10 PM Invited
Using X-Ray Microbeams to Assess Long Range Internal Stresses in Materials: *Michael Kassner*[1]; Peter Geantil[1]; Lyle Levine[2]; Bennett Larson[3]; Jon Tischler[3]; Wenjun Liu[4]; [1]University of Southern California; [2]National Institute of Standards and Technology; [3]Oak Ridge National Laboratory; [4]Argonne National Laboratories

The presence of counterbalanced stresses within microscopic volumes in deformed materials was predicted more than two decades ago and inferred from numerous indirect experiments. Yet, direct proof of their existence had been elusive, as spatially resolved measurements of the stress magnitudes and distributions critical for testing theories and computer modeling were not possible until recently. Researchers using the intense submicron x-ray beams at the Advanced Photon Source made the first quantitative, spatially resolved measurements of elastic strains within dislocation cells in plastically deformed Cu single crystals. The measurements indicated that the dislocation cell interiors were under significant and variable long range internal stresses. Additional measurements are uncovering other aspects of these stresses such as the statistical distribution of magnitudes. The most recent results of long range internal stress measurements in plastically deformed Cu single crystals as a function of position in the heterogeneous dislocation substructure will be presented.

Pb-Free Solders and Emerging Interconnect and Packaging Technologies: Effects of Surface Finishes and Advances in Interconnects

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Sung Kang, IBM Corp; Iver Anderson, Iowa State University; Srinivas Chada, Medtronic; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Albert Wu, National Central University

Tuesday AM — Room: 2020
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Srinivas Chada, Medtronic; Fay Hua, Intel Corp

8:30 AM Invited
Electroless Ni Contamination Induced ENIG Corrosion: *John Osenbach*[1]; John Delucca[1]; Frank Baiocchi[1]; Ahmed Amin[1]; [1]LSI Corporation

Corrosion of ENIG surface finish has been known for almost 2 decades. There has been significant progress toward eliminating this corrosion problem. However, corrosion, often referred to a black pad, is still sometimes found. Using FIB cross sectioning and STEM/EDS analysis we have identified a second phase particle contamination in the electroless Ni, Ni(P), layer on substrates that have corroded ENIG but not on substrates with non-corroded ENIG. To the authors knowledge it is the first reported observation of this effect. These particles are almost the same composition as the Ni(P) layer. When present, the particle leads to a modification in the growth habit of the Ni(P) layer which ultimately lead to a modification in the surface topology as well as creating low density interfaces. This change in topology and microstructure ultimately leads to Ni(P) corrosion. The data and corrosion model will be presented in the talk

8:50 AM
Effect of Cu Surface Finishes of Printed Circuit Board on the Microstructure of Lead-free Solder Joint: *Dai-hong Xiao*[1]; [1]Central South University

Effect of Cu surface finishes, including organic solderability preservatives (OSP) and immersion Ag (I-Ag), on the microstructure of lead-free solder joint was investigated with scanning electron microscope and energy dispersive X-ray spectroscopy). It was found that Cu surface finishes affected the microstructure of lead-free solder joint. The thickness of intermetallic compounds (IMC) layer with OSP was higher than that of I-Ag. Comparing with OSP, there are more plate-like Ag_3Sn intermetallic compounds between solder joints and Cu pad after I-Ag surface finishing. However, there are more voids found in the solder joints which distributed close to the solder interface and reduced the strength of solder joints after OSP surface finishing.

9:05 AM
Fluxless Ultrasonic Lead-Free Soldering For Electronics Packaging Applications: *Shankar Srinivasan*[1]; Tim Frech[1]; Karl Graff[1]; [1]Edison Welding Institute

Microstructure and mechanical properties of Sn-3.0Ag-0.5Cu (SAC305) solder joints fabricated using fluxless ultrasonic soldering is reported. Single-lap copper-copper joints with two different surface finishes, electroless nickel immersion gold (ENIG) and immersion silver (ImAg), were fabricated by ultrasonic soldering using SAC305. Tensile shear strengths of SAC305 in Cu/Cu, Cu/Cu-ENIG, and Cu/Cu-ImAg joints were similar to each other and in the range 30-35 MPa. Fractographic characterization indicated failure occurring through the solder, and exhibiting typical shear failure mechanism. Furthermore, the shear strength of SAC305 in the fluxless solder joints was similar to that obtained by conventional flux-based soldering. The results of the present study clearly demonstrate the potential for using ultrasonic soldering as a fluxless joining technology in electronics packaging applications requiring structural integrity of the joint.

9:20 AM
OSP PCB Via Hole Crack Effect Factors Analysis and Improvement: *Xie Na*[1]; Park ChangYong[1]; Jin Xing[1]; Chung Won Seok[1]; Guo Shi Da[1]; [1]Samsung Electronics(Suzhou) Semiconductor Co., Ltd.

OSP(Organic Solderability Preservative) PCB(Printed Circuit Board) has been widely used for its preferable character and more competitive cost. In the field of semiconductor, OSP has taken place of ENIG (Electroless Nickel/Immersion Gold) gradually. However, because of the process difference between ENIG and OSP, OSP PCB is prone to cause via hole crack, it is a headache problem puzzled us very much which brings us and our customers many reliability troubles. In this paper, through thermal cycling test, analyze the defect samples and contrast the structure difference for OSP & ENIG, to find reasons why OSP PCB via hole crack. Another purpose of this paper is by the means of DOE to select the optimal parameter for important effect factors, after applying to OSP PCB relative manufacture process, make improvement, reduce OSP via hole crack defect rate and control the quality of our products.

9:35 AM
The Properties of Novel Lead-Free Solder Composites: *Ainissa Ramirez*[1]; Brian Lewis[1]; [1]Yale University

Although ubiquitous in use, solders are endowed with basic properties that are limiting and require design compromises to be made. Specifically, the mechanical strength is influenced by the high homologous temperature of solder, and the transport properties are generally lower than desired. The transition to lead-free solders has brought further challenges. Their higher processing temperatures can be detrimental to nearby components. Here, we report lead-free composite solders designed to address not only these important properties but also their processing. A range of composite lead-free solders were produced by a combination of blending and vacuum mixing. It has been found that the mechanical properties can be altered by the amount, size, and degree of alignment of magnetic particle additions. Further, we examine induction heating as a means to localize heating in the magnetically-enhanced solder thereby minimizing thermal damage to adjacent components. Applications for the magnetically-enhanced and other composite solders are reviewed.

9:50 AM
Transverse Ultrasonic Bonding Of Electrodes Coated With Pb-Free Solder Between Rigid And Flexible Printed Circuit Board: *Jong-Bum Lee*[1]; Ja-Myeong Koo[1]; Jong-Gun Lee[1]; Seung-Boo Jung[1]; [1]Sungkyunkwan University

Recently, electrical and electronic equipment manufacturers have shown more interest in the development of electrical and mechanical bonding techniques for electrodes between a flexible printed circuit board (FPCB) and a rigid PCB (RPCB). The transverse ultrasonic bonding has been used for connecting the electrodes between RPCB and FPCB. There are attractive aspects that are rapid, low temperature and environmentally friendly process. However, there are many voids and un-bonded areas that have caused the reliability problems. Pb-free solder on the electrodes of the FPCB was melted and reacted with those of RPCB by ultrasonic vibration at optimized bonding condition. Thermal cycle, high temperature storage and high temperature/humidity test was evaluated to understand if there was vulnerability. Cross-section features in a bonding interface were also inspected by using a high resolution transmission electron microscope.

10:05 AM
Developing of Sn-Bi Eutectic Alloy Soldering Behavior by Al Addition: *Huseyin Adanir*[1]; [1]New Mexico Tech.

Sn-Al eutectic added Sn-Bi eutectic-based alloy was investigated on soldering of Cu-Cu components. Sn-Bi eutectic alloyed with Sn-Al eutectic at 25, 50 or 75 weight percentages. Al amount was increased 1-to-3 weight percentages in final compositions of three alloy samples. X-ray diffraction (XRD), differential scanning calorimeter (DSC), metallography and hardness testing were used to characterize the solder alloys. The joining of Cu-to-Cu substrates was investigated at 300, 350 and 400oC. The specimens were successfully soldered in a butt joint configuration and four-point bend tests were applied to determined soldered joint strength. The solder alloy containing eutectic-eutectic composition in weight percentages 14.5Bi+82.5Sn+3Al and soldered at 350oC performed highest joint value, as 72 MPa, among solder alloys. XRD and Scanning Electron Microscopy (SEM) were used to characterize the fracture surfaces after the four-point bend tests.

10:20 AM Break

10:35 AM
Effect Of Rare Earth Addition On Physical And Mechanical Properties Of Sn-Bi-Ag Lead-Free Solders: *Miguel Neri-Flores*[1]; Alberto Martinez-Villafañe[1]; Caleb Carreño-Gallardo[1]; [1]CIMAV, S.C.

The effect of rare earth element addition (Nd and Pr) on the Physical and mechanical properties of the Sn-Ag-Bi alloy was investigated, specially the wettability of the evaluated solders trough the contact angle measurements, applied on a copper substrate using two different RMA fluxes, with a higher chemical activity. The melting points of the alloys were determined using the Differential Scanning Calorimetry Technique (DSC). The effect of rare earth addition on the microstructure and tensile strength of the alloy Sn-Ag-Bi was investigated. Addition of 0.5 weight percent of rare earth elements Nd and Pr , refines the microstructure of the modified alloy Sn-Ag-Bi, obtaining finer particles of the formed intermetallic compounds, uniformly distributed on the alloy. The Pr addition on the Sn-Ag-Bi alloy increase the tensile strength up to 113 MPa, meanwhile the Nd addition on the alloy increase the tensile strength to 97 MPa.

10:50 AM
Effects of Surface Pre-treatment and Various Bonding Temperatures on Interfacial Toughness of Cu-Cu Direct Bonds: *Eun-Jung Jang*[1]; Jae-Won Kim[1]; Sarah Pfeiffer[2]; Bioh Kim[2]; Thorsten Matthias[2]; Seungmin Hyun[3]; Hak-Joo Lee[3]; Young-Bae Park[1]; [1]Andong National University [2]EV Group; [3]Korea Institute of Machinery and Materials

Cu–to-Cu thermo-compression bonds for three dimensional(3D) integrated circuits(ICs) have several advantages such as low electrical resistivity, high EM resistance, and reduced interconnect RC delay. However, the process temperature is limited up to 400° to prevent CMOS devices from being thermally damaged. High temperature bonding is a key bottleneck for 3D ICs applications due to a deadly impact on device reliability.Cu and Ti films were deposited by sputtering on thermally oxidized Si(100) wafers and then the deposited films were bonded by direct Cu-to-Cu thermo-compression bonding for evaluating the effect of the native oxide on the bonding toughness. The bonding toughness will be evaluated as a function of acid cleaning time on Cu surface with varying bonding temperatures. The effect of post-annealing under oxygen and nitrogen environment at 200, 300, 400 and 500°C on the bonding toughness at the Cu bonded interface will be evaluated by four point bending test.

11:05 AM
Microstructure and Mechanical Properties of Density-Matched Particle Reinforced Composite Pb-Free Solder Joints: *James Lucas*[1]; [1]Michigan State University

Mechanical properties of Pb-free solder joints were investigated using nanoindentation testing. Indentation elastic moduli and hardness properties were determined for matrix and intermetallic compounds phases. Functional-gradient particle reinforcement was used to attain better gravity matching with the base solder alloy density. Density matching between the matrix and reinforcement allows for more uniform and consistent particle distribution within the composite solder joint. To better achieve matrix/particle density matching, Cu, Ag and Ni coated particles were introduced into the solder matrix. Indentation creep properties were assessed in localized regions of the solder joint microstructure. The stress exponent, n, associated with secondary creep differs widely with the microstructure features probed. Early investigation of the mechanical properties of density-matched particle composite solder shows promise improved electronic packaging.

11:20 AM
Interfacial Reaction of Sn-based Solder/Cu System with Zn Addition after Heat Treatment: *Chi-Yang Yu*[1]; Jeng-Gong Duh[1]; [1]National Tsing Hua University

Intermetallic compounds (IMCs), Cu6Sn5 and Cu3Sn, usually formed at the interface between Sn-based solders and Cu substrate during reflow. After long time aging, the total thickness of IMCs increased and kirkendall voids were in Cu3Sn layer. Recently, it was reported that addition of minor Zn in Sn-Ag-Cu solder could suppress the growth rate of Cu6Sn5 and Cu3Sn. To understand the mechanism of Zn addition in the formation of IMCs, the experimental design was carried out to fabricate Sn-based solder/Cu joint with and without minor Zn addition. After reflow and long time aging, Zn atoms accumulated at the interface between IMCs and Cu substrate, and incorporated into Sn sublattice of Cu6Sn5. The specific composition and structure of IMCs were obtained with the aid of FE-EPMA and TEM. IMCs growth mechanism was correlated to the microstructure feature. Besides, the mechanical properties of the solder joint with Zn-content was investigated and discussed.

11:35 AM
Characterization of the Effect of Ni-Ti Shape Memory Alloy on Solder Joint Reliability through Modeling and Testing: *Chia-Yen Tan*[1]; Jeng-Gong Duh[1]; [1]National Tsing Hua University

Nowadays, the most common problem that surface mount technology faces is the warpage and the inelastic strain concentration accumulated in the solder joint during thermal cycling due to the mismatch of thermal expansion coefficient between package side and chip side. The NiTi shape memory alloy (SMA) UBM can suppress the inelastic strain in the solder joint. The objective of this research is to investigate how SMA applied in UBM can affect solder joint reliability during thermal cycling. A BGA component with silicon chip, deposited multi layer UBM, SAC305 solder ball, and the adhered PCB side was prepared and employed for thermal cycling test. Meanwhile, a finite element model of the exact component was also set up for simulation of stress and strain distribution in the solder joint under different temperatures. Cross section observation of fracture in solder joint also provided the direct evident of SMA effect on the tested component.

11:50 AM
Intermetallic Formations In Rapidly Solidified Pb-Free Solder Bonds Formed Via The Solder Jet Bonding Technique: John Wagner[1]; *Peter Ladwig*[1]; Douglas Riemer[1]; Galen Houk[1]; [1]Hutchinson Technology

The majority of the research published on Pb-free solders is concerned with BGA or other applications where the reflow time allows significant solder to pad reaction time. The hard disk drive industry predominately uses solder jet bonding to electrically connect the read/write sensor. This technique does not use flux and has cooling times on the order of milliseconds. Therefore, the intermetallic formation is highly non-equilibrium and is localized near the pad interface. Surface finish thicknesses and compositions have a significant influence on the intermetallic phases and morphologies that are formed. The intermetallic microstructure, along with voids, can significantly impact joint reliability. This study highlights unique intermetallic formations that occur with rapid solidification and probes how these are influenced by underlying pad metallization. From this understanding, recommendations for ideal surface finishes for solder jet bonding can be made.

12:05 PM
Silver-Bismuth Alloys as High Temperature Lead-Free Solders: Anthony Muza[1]; Mark Cooper[1]; Carol Handwerker[1]; [1]Purdue University

Under pressure from RoHS, microelectronics companies are exploring possible high temperature lead-free solders to replace the 95Pb-5Sn tin-lead alloy used for chip interconnects and hierarchical soldering processes. This study investigated a range of Bi-Ag binary eutectic alloys as suitable high temperature lead-free solders and as partially molten joining materials. Results will be presented describing the wetting of these alloys as a function of alloy composition, temperature, and flux type, their microstructures, and their resulting electrical and mechanical properties. Of greatest importance for use in interconnects is that, as the Ag concentration increases, the Ag dendritic primary phase provides not only a more electrically conductive path than high Bi alloys, but also improved mechanical properties.

12:20 PM
Reliability Examination Of Mixed Assemblies: *Rishi Kaila*[1]; Doug D. Perovic[1]; [1]University of Toronto

Two problems have become apparent in the use of Pb-free solders. First, suppliers of electronic components are producing RoHS compliant versions of some area array components with only certain Pb-free solder balls. In use, these may be mixed in forming a joint with solder paste of a different composition, thereby forming a joint with an altered microstructure with unknown properties. Second, it has been found that the new Pb-free solders are susceptible to failure under impact loading conditions.Metallographic samples will be made and their microstructure examined under the optical and scanning electron microscope. Microhardness characteristics and the effect of ageing on microstructure will also be examined. This study will allow choosing the parameters for successful reflow and rework processes and provide industrial guidance on how to manage through the issues and concerns with incorporating new components in Pb-free assemblies.

Peirce-Smith Converting Centennial Symposium: Injection Techniques, Modeling and Process Control

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
Program Organizer: Joel Kapusta, Air Liquide Canada Inc

Tuesday AM — Room: 2009
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

8:30 AM
Peirce-Smith Converting: Another 100 Years?: Thomas Price[1]; *Cameron Harris*[2]; Albert Wraith[3]; Skip (I.E.) Hills[2]; Wayne Boyd[4]; [1]TKTV Tedhnologies; [2]Worley Parsons M&M Toronto; [3]Retired; [4]Worley Parsons Canada

Most Peirce-Smith converters inject air through tuyeres, with nitrogen representing the majority of gas passing through the process. The nitrogen controls the temperature of the vessel by carrying away heat from the reactions, both globally and locally at the tuyere tip. However, nitrogen adds energy to the bath and contributes to splashing and limits the blowing rate. High oxygen smelting has made streams of gas containing high concentrations of sulphur dioxide commonplace, which can be used to substitute SO2 for nitrogen and drastically changes Peirce-Smith converting (and all other smelting/converting processes as well). Less SO2 is required to carry away the same quantity of heat, which allows higher concentrations of oxygen and leads to the possibility of increased production.The paper investigates this substitution, its impact on converter productivity, and on the remainder of the plant.

8:55 AM
Pneumatic Injection of Process Materials to the Peirce Smith Converter: *Mark Coleman*[1]; Gavin Money[1]; [1]Clyde Materials Handling Ltd

Clyde Materials Handling is an established customer-driven solutions provider, with over 30 years experience in the process improvement industry, Clyde Materials Handling has helped their global customer base transform processes, enabling them to generate sustainable economic benefit and maintain their positions as leaders in their respective markets. Clyde Materials Handling has worked closely with their customers, such as Codelco in Chile, where pneumatic solutions have been used to inject a consistent, pulseless and accurate flow of copper concentrate into a bath smelter, increasing operational capacity, availability and productivity. This paper will discuss and highlight the ways in which pneumatic injection technologies, have improved the operational performance with specific reference to applications in the Peirce Smith Converter. Clyde Materials Handling has applications injecting Electronic Scraps, Dusts, Reverts, and other additives to the Peirce Smith Converter. They also have experience of modified PS Units used for Slag Cleaning.

9:20 AM
Full-Scale Converter Experiments on High Pressure Tuyère Injection at the Horne Smelter: *Phillip Mackey*[1]; [1]Xstrata

This paper describes the design, operation and process outcomes of groups of three sonic high pressure (HP) tuyères set in the tuyere line panels of the Noranda Continuous Converter (NCV) over a period of several converter campaigns. The NCV treats high grade Noranda Reactor matte and therefore operates in continuous 'copper-blow' mode, which is the most severe environment for tuyère line erosion. The tuyères, spaced 0.3m (1 ft.) apart, were of two sizes delivering approx. 1300 and 5000 Nm3/hr (~800 and 3100 scfm) respectively at pressures up to 650 kPa abs. (~80 psig). Measurements of tuyère length through each test campaign showed a profound reduction in erosion at the mouth of HP tuyères compared to conventional tuyeres elsewhere in the tuyère line. Tuyère life was extended by a factor of three. Accretion conditions, their relationship to flow resistance and the implications of high pressure injection for refractories performance are discussed.

9:45 AM
Technology and Operational Improvements in Tuyère Punching, Silencing, Pyrometry and Refractory Drilling Equipment: *Michael Marinigh*[1]; [1]Heath and Sherwood, Ltd.

Increased safety requirements, higher operational costs, greater environmental restrictions, and new competitive processing techniques are the challenges smelter operators must confront in order to remain economically viable. On converting furnaces which have tuyères such as the Noranda Reactor and the Teniente, Peirce-Smith and Hoboken converters, efficient tuyère line management is critical to optimizing blowing rates, increasing refractory life, and improving safety. This paper will describe recent improvements made to equipment used for tuyère punching, tuyère silencing, tuyère pyrometry and refractory drilling of tuyères and how the proper application of the equipment can lead to operational and safety improvements.

10:10 AM Break

10:25 AM
Changing Reality: Continuous Production Control for Optimized Productivity: Wilhelm Wendt[1]; *Willy Persson*[1]; [1]Semtech Metallurgy AB

With rizing price of raw materials and increasing energy and environmental restrictions the optimization of pyrometallurgical processes beyond what can be achieved via modelling and operator experience has come more and more into focus and led to a quest for methods for dynamic process control. Independent of the details of the control mechanism it has to be based on real-time information on the status of the process. Since mid 1990s Semtech OPC Systems are in routine use at a number of smelters to provide continuous on-line information on the instantaneous status of converting processes in Peirce-Smith converters. This information offers an opportunity for the operators to maintain / bring back the process to the desired route. This presentation will discuss experiences from applications to converting of copper and nickel in Peirce-Smith converters. Special emphasis will be laid on de-bottlenecking, e.g., in concentrate injection, revert accumulation, magnetite handling and oxide-skim transfer.

10:50 AM
A Dynamic Simulation for the Validation Tests of the Codelco-Chile Continuous Converting Process: Carlos Caballero[1]; Alex Moyano[1]; Pedro Morales[1]; Claudio Toro[2]; *Hugo Jara*[2]; Leandro Guzmán[2]; Rodrigo Díaz[2]; [1]Codelco; [2]IM2-Codelco

During 2007, Codelco-Chile carried out industrial tests for validating the Continuous Converting Process in the Teniente Converter of the Codelco Norte smelter. For planning and starting up the tests, a predictive, analytical and dynamic model was developed to support operational decisions. The dynamic model was developed using Metsim® platform and dynamic data exchange (DDE) to operate in a friendly way along the MSExcel®. The main feature of this real-time model was the on/off-line ability to work gathering data from either the smelter's PI system® or database, and simulate the operation with or without scheduled events and controls.The main model development considerations which include thermodynamics, metallurgical, kinetics, slag-type and on-line mass and heat balance of the process, and also the successful validation of the model are presented and discussed in the present paper.

11:15 AM

Advanced Metallurgical Modeling of Ni-Cu Smelting at Xstrata Nickel's Sudbury Smelter, Ontario, Canada: *Nagendra Tripathi*[1]; Pascal Coursol[1]; David Tisdale[2]; Phillip Mackey[1]; [1]Xstrata Process Support; [2]Xstrata Nickel

Xstrata Nickel's Sudbury smelter operation is based on fluid bed roasting of Ni-Cu sulphide feed, followed by electric furnace smelting and Peirce-Smith converting to produce a Bessemer matte as the final product. The present smelter configuration has been operational since 1978, and currently has a nominal capacity of about 65,000 tpy of contained nickel and is ramping up to 80,000 tpy in the next few years. As part of a review of plant capacity options, a new metallurgical model of the plant was developed in order to examine a number of processing alternatives. Based on the Metsim platform, the present model includes the ability to examine the impact of a number of plant parameters on potential plant performance. This paper provides a description of the model and provides an overview of the results.

11:40 AM

Key Performance Indicators of Peirce-Smith Converting – Choosing the Correct Parameters to Monitor: *Benjamin Hogg*[1]; Jared Ball[1]; Muthuraman Ramanathan[1]; [1]Xstrata

There are over 100 process parameters that can be monitored during a single Peirce-Smith (PS) converter batch operation. To support a consistent operation it is important to select the correct Key Performance Indicators (KPI's) to monitor. They should be strategically chosen to drive the desired behaviour with enough flexibility to endure process variations. At the Xstrata Copper Smelter in Mount Isa most of the PS converter operators have very little experience and are often lacking in the appropriate education or training to properly understand the process. For this reason 10 simple KPI's were chosen that could be easily comprehended and monitored by all smelter personnel. KPI compliance has been supported by relevant project work in the converter area. The online measurement of full and empty ladle weights has enabled an immediate indication of skull size, skim/slag carryover, and metal loss.

12:05 PM

Modeling Peirce-Smith Converter Operating Costs: *Alessandro Navarra*[1]; Joël Kapusta[2]; [1]École Polytechnique de Montréal; [2]Air Liquide Canada

The cost-modeling software introduced by Ng et al. in 2005 [1] has been extended to consider the remelting of copper scrap and revert. The original model evaluated the operational costs of a Peirce-Smith converter; algebraic relationships were implemented with arrays (to represent matrices, vectors and scalar ratios). The new software brings flexibility to the underlying thermochemical balances, by using linked-lists instead of arrays. It is now possible to consider the blending of several feeds, including scrap and revert. The software computes the length of the converting cycle from the blowing capacity and the times required for other operations (charging, skimming and idle time). Downtime, labor, and materials for converter lining repair are also considered. The software is easily extended to examine the costs of alternative operating strategies or injection technologies such as high-pressure, shrouded injection. The cost benefits of changing operating procedures and technology are demonstrated through example calculations.

12:30 PM

Minimum Numerical Model of a Pierce-Smith Converter: Adriana Cervantes-Clemente[1]; *Cesar Real-Ramirez*[1]; Manuel Palomar-Pardave[1]; Luis Hoyos-Reyes[1]; Marco Gutierrez-Villegas[1]; Jesus Gonzalez-Trejo[1]; [1]Universidad Autonoma Metropolitana - Azcapotzalco

Recent improvements on Pierce-Smith Converters (PSC) have combined numerical and physical simulations. However, most of the physical simulations have been carried out in cold-water models with only one tuyere. Several authors have proved that 2D numerical simulations do not reproduce the hydrodynamic behavior observed in physical simulations. On the other hand, most of the 3D numerical models have used a PSC thin slice with only one tuyere, but symmetric boundary conditions on the virtual walls have been imposed in order to reproduce the behavior of the whole converter. Therefore, there is no coincidence with the rigid walls of the physical models. The aim of this work is to quantify the effect of the virtual walls boundary conditions of a PSC thin slice and to determine the minimum number of tuyers to characterize the fluid flow behavior of the entire converter.

12:55 PM

Applications of Thermo-Chemical and Thermo-Physical Modeling in the Copper Converter Industries: *Pengfu Tan*[1]; [1]Xstrata Copper

A thermo-chemical model of the copper P-S converter and a viscosity model for the converter slag have been developed to predict the behavior of magnetite in the converter slag, amounts of slag and matte, compositions of slag and matte, volume of the bath, bath temperature, slag blow endpoint determination, SEMTECH OPC signals, and the viscosity of converter slags. The predictions of bath temperature, slag and matte compositions, and magnetite content in the slag have been validated by the industrial data. The effects of fluxing strategy, returns and skim charges, oxygen enrichment, and temperature on the magnetite formation in the slag have been predicted and discussed. The effect of oxygen potential, SiO_2/Fe in slag, detection of slag blow endpoint, and temperature on slag viscosity, liquidus temperature of converter slag and skimming operation has been modeled and discussed as well. Some applications of the industrial operations have been presented.

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

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic 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; Daniel J. Lewis, Rensselaer Polytechnic Institute; Kejun Zeng, Texas Instruments; Wojciech Gierlotka, AGH University of Science and Technology; Yee-wen Yen, National Taiwan University of Science and Technology

Tuesday AM Room: 2022
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Kejun Zeng, Texas Instruments Inc; Albert Tzu-Chia Wu, National Central University

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8:30 AM Invited

Dynamic Reactive Wetting of Sn-Ag-Cu Solder Alloys on Cu Substrates Coated by Ni and Au: *Joonho Lee*[1]; Jong-Min Kim[2]; [1]Korea University; [2]Chung-Ang University

In the electronic components and device packaging process, the conductor Cu surface is generally coated by Au and Ni. Au coating is applied to protect the oxidation of the Cu surface and enhance the solderability, while Ni coating is applied as a diffusion barrier between the solder alloy and the Cu substrate to restrict the formation and growth of the intermetallic compound. Dynamic reactive wetting characteristics of Sn-Ag-Cu alloys are related to the properties of the coating materials. This presentation will report the observation results on the reactive wetting behavior of Sn-Ag-Cu alloys on Cu substrates coated by Ni and Au in millisecond scale.

8:50 AM Invited

Phase Equilibria for Understanding the Reaction between Sn-based Solders and Ni(P) Substrates: *Clemens Schmetterer*[1]; Herbert Ipser[1]; Simona Delsante[2]; Gabriela Borzone[2]; [1]University of Vienna; [2]Università degli Studi di Genova

Ni(P) coated substrates are widely used in the electronics industry due to their ease of production and their favorable properties. In order to understand the reactions that occur between such a substrate and Sn-based solders, knowledge on the phase equilibria is important, because ternary Ni-P-Sn compounds are brittle and detrimental to the joint quality. The ternary alloy system Ni-P-Sn and its sub-systems have thus been the subject of extensive investigations. Five ternary compounds are known to exist in the Ni-rich corner. The related phase equilibria are shown as a consistent set of isotherms, isopleths and a liquidus surface. Emphasis will be placed on the thermal behavior.Due to the experimental difficulties caused by phosphorus, CALPHAD modelling of this system is desirable. Therefore enthalpies of formation of binary Ni-P and ternary Ni-P-Sn compounds have been determined by Direct Reaction Calorimetry.

9:10 AM

Effect Of Cu Lead-Frame Microstructure On Solder/Cu Interfacial Reaction And Soldering Wettabilty: *Huang Kuan Chih*[1]; Hsiao Yu Hsiang[1]; Lee Chih Ming[1]; Liu Cheng Yi[2]; Shieu Fuh Sheng[1]; [1]National Chung Hsing University; [2]National Central University

Cu, with a high thermal conductive property, is used as Lead Frame (LF) for high power IC bond pad. The wettability and interfacial reactions of solders highly affect the reliability of power IC on Cu LF. Our preliminary results show that the solder wettability significantly depend on the microstructure of Cu LF, such as, preferred orientation of Cu grains, and Cu grain size. In this study, the correlation between the soldering wettability and interfacial reaction on Cu LF and the microstructure of Cu LF was investigated. The Cu LF having (220) preferred orientation show the best wettability, comparing to other preferred orientations. Also, we found microstructure of Cu LF greatly influences the solder/Cu interfacial reaction, for example, Cu dissolution during solder reflow. Different grain size and grain orientation of Cu LF materials were investigated. The correlation between soldering interfacial reaction and Cu LF microstructure will be present in this talk.

9:25 AM

Interfacial Reactions on Pb-Free Solders with Pd/Au/Ni/Cu and Pd/Au/Ni/Brass Multilayer Substrates: *Yee-Wen Yen*[1]; Yang-Kai Fang[2]; Chiapyng Lee[2]; [1]Graduate Institute of Mterials Science and Technology, National Taiwan University of Science and Technology; [2]Department of Chemical Engineering

Interfacial reactions on Sn, Sn-3.0Ag-0.5Cu, Sn-0.7Cu, Sn-58Bi and Sn-9Zn with Pd/Au/Ni/Cu and Pd/Au/Ni/Brass at 240-270° for 20 minutes to 20 hours were investigated. The experimental results present that the (Ni,Cu)3Sn4 phase converted to the (Cu,Ni)6Sn5 phase and the Cu3Sn was formed in the Sn/Pd/Au/Ni/Cu. In Sn-3.0Ag-0.5Cu/Au/Pd/Ni/Cu and Sn-0.7Cu/Au/Pd/Ni/Cu, the (Cu,Ni)6Sn5 and Cu3Sn phase were observed. Only the Ni3Sn4 phase was observed in the Sn-58Bi/ Pd/Au/Ni/Cu. In the Sn-9Zn/Au/Pd/Ni/Cu, the Pd2Zn9 and NiZn phases were formed. The Pd2Zn9, NiZn, and Ni5Zn21 phases were formed for 4 hours later. The (Cu,Ni)6Sn5 and CuZn phase were found in Sn/Au/Pd/Ni/Brass, Sn-3.0Ag-0.5Cu/Au/Pd/Ni/Brass, and Sn-0.7Cu/Au/Pd/Ni/Brass. In the Sn-58Bi/Au/Pd/Ni/Brass, only the Ni3Sn4 phase was observed. However, the (Ni,Cu)3Sn4, (Cu,Ni)6Sn5, and CuZn phases were formed after 8 hours later. In the Sn-9Zn/Au/Pd/Ni/Brass, the Pd2Zn9 and Ni5Zn21 phases were formed. Aging for 20 hours, the CuZn5, Pd2Zn9, Ni5Zn21, and Cu5Zn8 phases were formed.

9:40 AM

The Growth Of Intermetallic Compounds Between SnAgBiIn Pb-Free Solders And Copper Substrates During Reflow And Solid State Aging: *Albert Wu*[1]; Ming-Hsun Chen[2]; [1]National Central University; [2]National Taipei University of Technology

SnAgBiIn solder systems are one of the Pb-free candidates to replace eutectic SnPb solder. The addition of indium can lower the melting point of the alloy but will not reduce the mechanical strength of the joints. In this study, liquid solders were reflowed on Cu substrates at different temperatures and times. In addition, the solder systems were solid state aged for up to 40 days. The interfacial reactions between the SnAgBiIn solders and Cu substrates of the systems were investigated; the composition of the compounds was studied by EPMA. In this paper, the kinetics of the growth of the intermetallic compounds is discussed.

9:55 AM

Volume Effect on the Solid-State Reaction between Sn-Ag-Cu Solders and Ni: *Su-Chun Yang*[1]; C. Robert Kao[1]; [1]National Taiwan University

With the continuous push for device miniaturization, solder volume effect in electronic devices should be taken into account. In our previous study, strongly volume effect during soldering had been reported. In this study, solid state reaction would be considered. Sn3AgxCu (x = 0.3, 0.5 and 0.7 wt.%) were soldered on Ni and aged at 160oC for 1000 hrs. Three different sizes of solder spheres (300, 500, and 760 micrometer diameter) were used. The study revealed that the type of intermetallic compound transformed from (Cu,Ni)6Sn5 to (Ni,Cu)3Sn4 as the residual Cu concentration in solder dropped below 2 wt.%. In addition, during soldering (Cu,Ni)6Sn5 spalled massively from the interface under certain conditions, including the smaller joints and those with lower Cu concentration. However, the spalling was not observed during aging. The reason for these observations would be discussed in this talk.

10:10 AM Break

10:30 AM Invited

Detailed Phase Evolution of Phosphorous-Rich Layer and Formation of Ni-Sn-P Compound in SnAgCu/Electroplated Ni-P Solder Joint: Yung-Chi Lin[1]; Kai-Jheng Wang[1]; *Jeng-Gong Duh*[1]; [1]National Tsing Hua University

Interfacial microstructure of Sn-3Ag-0.5Cu/Ni-P with various phosphorous contents was investigated by TEM and FE-EPMA. It was revealed that as the Ni-Sn-P compound was formed between the solder matrix and Ni-P UBM, the conventionally so-called phosphorous-rich layer was transformed to a series of layer compounds, including Ni3P, Ni12P5 and Ni2P. The relationship between Ni-Sn-P formation and evolution of P-rich layers was probed by electron microscopic characterization with the aid of the phase diagram of Ni-P. It was also demonstrated that the thickness of Ni-P UBM would affect the Ni-Sn-P formation. On the basis of the TEM micrograph, selected area diffraction pattern as well the FE-EPMA results, the detailed phase evolution of P-rich layers in the SnAgCu/Ni-P joint was revealed and proposed. Moreover, in consideration of the mechanical property for the joint, Ni-Sn-P phase formation and fabrication feasibility of Ni-P UBM, the phosphorous content and suitable thickness of Ni-P UBM were discussed.

10:50 AM Invited

Effect of Cu in SnAgCu Solder on Interfacial Reliability of Solder Joints: *Kejun Zeng*[1]; [1]Texas Instruments Inc

Effect of Cu content in solder is studied on the interfacial reliability of SnAgCu solder joints on Ni/Au plated pads. Solder ball composition was Sn3.0Ag0.5Cu. Cold ball pull test was performed to assess the BGA joint reliability after ball attachment process and also after preconditioning reflows. Test after BGA assembly (one reflow) did not generate any interfacial failure, but after the three more reflows of preconditioning many joints showed the failure mode of interfacial cracking by pull test. Cracking occurred between IMC layers of Cu6Sn5 and Ni3Sn4. Formation of the bilayer IMC structure is explained from the perspective of interfacial equilibrium. Its effect on interfacial reliability of solder joints is discussed. It is suggested that, if the Cu content in the SnAgCu solder is reduced to a certain level, formation of the Cu6Sn5 layer can be avoided and thus the interfacial reliability is improved.

11:10 AM

Nano-sized Induced Low Temperature Alloying Behaviour in Interconnection Applications: Tzu-Hsuan Kao[1]; *Jenn-Ming Song*[2]; In-Gann Chen[1]; Weng-Sing Hwang[1]; Teng-Yuan Dong[3]; [1]National Cheng Kung University [2]National Dong Hwa University [3]Kaohsiung Medical University

A concept of nanosize induced liquid-solid reaction and thus interdiffusion behavior between NPD (nanoparticle deposition) and metallic substrates has been proposed recently. The supercooled liquid reacts with the substrate and provides a fast atomic mobility, which results in low temperature alloying between NPD and substrate materials. Even though the reaction duration is very short, this liquid-solid reaction has a considerable effect on the mutual interdiffusion between the NPD and substrates, leading to a firm bonding. This study investigates the low temperature alloying behavior in binary and ternary systems. Several kinds of NPs and substrate materials are chosen to investigate the effect of the differences in lattice mismatch and electronegativity between the elements. Experimental results of elemental distribution and phase identification by XPS and Nano-AES, as well as the evaluation of mechanical properties such as adhesion strength and nanoindentation, will be given in this presentation.

11:25 AM

Interfacial Reactions in the Sn-9Zn+Cu Solder with Ni Substrate: *Wei-Kai Liou*[1]; Yee-wen Yen[1]; [1]Graduate Institute of Materials Science and Technology, National Taiwan University of Science and Technology

This study investigates the interfacial reactions between (Sn-9Zn)+xCu/Ni systems. The sequences of IMC evolutions in the (Sn-9Zn)+xCu/Ni system aged at 255° for 1-3 hours were: (i) Ni5Zn21 and Zn phases at Sn-9Zn/Ni couples; (ii) Cu5Zn8, Cu5Zn8 phases, as x (the Cu content) was 1 wt%; (iii) (Ni,Zn,Cu)3Sn4 and Cu5Zn8 phases, as x was 4 wt%; (iv) (Cu,Ni)6Sn5 and CuZn phases, as x was 7 wt%, and (v) Cu6Sn5 and CuZn phases at Sn-9Zn+10wt%Cu/Ni couples. As the reaction time was prolonged from 5 to 24 hour, the Cu5Zn8 phase would convert to the (Cu5Zn8+Ni5Zn21) mixture at interface, as x was 1 wt%. When 10 wt% Cu was added into the Sn-9Zn solder, the (CuZn+Cu6Sn5) replaced the CuZn phase in the solder. Experimental results indicate that IMCs formation in

(Sn-9Zn)+xCu/Ni systems dramatically changes with various reaction time and Cu contents.

11:40 AM
Wetting Properties and Interfacial Reactions of Cu5Zn8-Bearing Pb-Free Solders on the Cu Substrate by Mechanical Alloying: *Inyu Jung*[1]; Moon Gi Cho[1]; Hyuck Mo Lee[1]; [1]KAIST

Cu5Zn8-bearing solders are proposed to enhance the wetting properties of the Zn-doped solder alloys. The Cu5Zn8-bearing solder powders with a diameter of 50-70 μm were fabricated successfully by the mechanical alloying process in which the milling time, the rotational speed and the ball-to-powder weight ratio were controlled. Their composition was identified as Sn-0.31Cu-0.48Zn (in wt.%) by the inductively coupled plasma atomic emission spectroscopy (ICP-AES). After making the powders into a paste with a rosin activated type flux, the wetting angles of the Cu5Zn8-bearing solder paste on the Cu substrate were compared with those of Sn-0.31Cu-0.48Zn and Sn-0.7Cu bulk solder alloys. As a result, the wetting properties of the Cu5Zn8-bearing solder paste were better than the bulk Sn-0.31Cu-0.48Zn and similar with the bulk Sn-0.7Cu. The reason of the enhanced wetting properties is explained through thermodynamic calculations, and interfacial reactions with Cu substrates are also discussed.

Recent Advances in Thin Films: Metal Films and Integration Schemes

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Thin Films and Interfaces Committee
Program Organizers: Nuggehalli Ravindra, New Jersey Institute of Technology; Gregory Krumdick, Argonne National Laboratoryoratory; Choong-un Kim, University of Texas; Narsingh Singh, Northrop Grumman, ES

Tuesday AM Room: 3011
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Bhushan Sopori, National Renewable Energy Laboratory; Nuggehalli Ravindra, New Jersey Institute of Technology

8:30 AM Introductory Comments

8:35 AM
Study of Surface Electromigration in Au Thin Films: *Liangshan Chen*[1]; N. Michael[1]; C. U. Kim[1]; U. Chul[2]; J. S. Cho[2]; J. T. Moon[2]; [1]Department of Materials Science and Engineering, The University of Texas at Arlington; [2]MK Electron Co. Ltd

The mechanism of electromigration in Au thin films is one of the most illusive subjects in the related field primarily because of vastly differing results presented by previous studies. While some studies present that electromigration in Au occurs by grain boundary migration in the direction of electrons, others state that it occurs by surface in the opposite direction. With technical importance of electromigration mechanism, especially for Au wirebond and interconnects used in electronic devices, it is important to understand how such varying results are possible. In our study, we investigate the nature of electromigration using "cross-strip" configuration. Our study finds that electromigration of Au occurs in two routes, grain boundaries and surface, with different directionality. This paper presents the results leading to such a conclusion and discusses their technological impacts.

8:55 AM
Texture Control During Growth Of Copper Thin Films: Atomic-Scale Simulations: *Moneesh Upmanyu*[1]; Haiyi Liang[2]; Hanchen Huang[3]; [1]Colorado School of Mines; [2]Harvard University; [3]Rensselaer Polytechnic Institute

We have performed molecular dynamics simulations of the <111>-<110> texture competition during low energy copper film growth. For an initially bi-textured thin film, we find that the competition can be controlled by optimizing three accessible deposition conditions: in-plane strain, deposition rate, and angle of deposition. The variables modify the interplay between thermodynamic and surface kinetic anisotropies in copper which directly affect the texture evolution. The evolving surface morphology is also sensitive to the form and rate of texture evolution. The control paradigm should be applicable for thin film texture control in metallic thin films in general.

9:15 AM
The Effect of temperature on In-Situ Intrinsic Stress Behavior in Cu Thin Films: *Moohyun Cho*[1]; Sang Ryu[1]; Youngman Kim[1]; [1]Chonnam National University

The intrinsic stress in thin film evolves during nucleation and growth of atoms on the substrate. Cu thin films, which grow in Volmer-Weber type, have unique stress behavior of three stages, such as initial compressive, tensile and incremental compressive. The tensile stress evolution was reported from the volume contraction though island coalescence. The mechanism of compressive stress is still in controversy even though extensive research efforts are being made. Incremental compressive stress may be related to the mobility of adatom on the substrate. To control the mobility of depositing Cu atoms, the substrate temperatures are changed from room temperature to 250° during depositon. We observed the in-situ stresses behavior of Cu thin films during deposition using multi-beam curvature measurement system attached to a thermal evaporation device. When temperature of substrate increased, the thickness at tensile maximum and the slop of incremental compressive stress showed a tendency to decrease.

9:35 AM
A Novel Method for Parallel Assembly of Microcomponents: Nuggehalli Ravindra[1]; *Rene Rivero*[1]; Michael Booty[1]; Anthony Fiory[1]; [1]New Jersey Institute of Technology

Pick and Place is the current industry standard for the heterogeneous assembly of microcomponents. However, because Pick and Place is a serial processing method, which requires significant expenditures of resources, it is not the most efficient way to assemble devices. Several alternative and parallel techniques have been proposed but those methods are limited by geometric, material, and statistical issues. The method outlined in this paper will be shown to circumvent the drawbacks that plague existing parallel assembly techniques; it represents a versatile and scalable method which is able to conform to any manufacturing situation and to produce a 100% yield.

Recycling of Electronic Wastes: Hydrometallurgical Recycling

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS Materials Processing and Manufacturing Division, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Lifeng Zhang, Missouri University; Fay Hua, Intel Corp Corp; Oladele Ogunseitan, University of California; Gregory Krumdick, Argonne National Laboratory

Tuesday AM Room: 2024
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Lifeng Zhang, Missouri University Science Technology; Christina Meskers, Umicore

8:30 AM Introductory Comments

8:35 AM
Extraction and Separation of Metals Using LIX 84 and D2EHPA Diluted in Kerosene from Sulfate Solution: *Vinay Kumar*[1]; Manoj Kumar[1]; Manis Jha[2]; Jae-chun Lee[2]; [1]National Metallurgical Laboratory, Jamshedpur, India; [2]Korea Institute of Geoscience and Mineral Resources

The disposal of large quantities of electronic scraps generated world wide is causing not only environmental problem but also loss of resources. Therefore, R&D efforts have been made to develop a suitable process for extraction and separation of metals viz. copper, zinc, cadmium and nickel expected from the leaching of e-scraps using solvent extraction process. Different process parameters viz. pH, A/O ratio, contact time, simulation studies to establish stage requirement for extraction in continuous mode etc have been studied to optimise the condition for metals separation. The studies showed selective extraction of copper after iron precipitation from the sulfate leach solution of printed circuit boards containing 1.0 g/L Cu and minor impurities with 2-hydroxy-5-nonylacetophenoneoxime (LIX 84) above pH 2.0 in single stage. A scheme for separation of cadmium, zinc and nickel has also been proposed using di(2-ethylhexyl) phosphoric acid (D2EHPA) under controlled pH of the aqueous solution.

8:55 AM Question and Answer Period

9:05 AM
Metal Recovery from Waste Electrical and Electronic Equipment (WEEE) by Leaching and Electrodeposition IV: *Geoff Kelsall*[1]; Chun-yee Cheng[1]; Anna Robson[1]; [1]Imperial College London

A novel process is being developed to recover metals from waste electrical and electronic equipment (WEEE) and other secondary sources using acidic aqueous chloride electrolyte. Chlorine is generated at the anode of a membrane-divided electrochemical reactor, absorbed into the solution and used to dissolve metals (Ag, Au, Cu, Pd, Sn, Pb, etc.) from shredded WEEE in an external leach reactor. The metals are electrodeposited at the cathode, enabling their subsequent recovery and refining. Hence, the overall process involves inputting electrical energy to move the metals from WEEE to cathode and, in principle, additionally produces only de-metallised WEEE, for further processing. Finite element models were developed for a packed bed metal leach reactor and the effects computed of particle shape, liquid flow rate and reaction rate coefficients, on metal conversions and efflux chlorine concentrations. Predictions were validated against experimental data; metal conversions >0.9 were achieved in 8 hours.

9:25 AM Question and Answer Period

9:35 AM
Metals Recover Of Obsolete Mobile With Emphasis In Recycling: *Viviane Tavares*[1]; Mariana Maioli[1]; Denise Espinosa[1]; Jorge Tenorio[1]; [1]Escola Politecnica da Universidade de São Paulo

The tecnology advance of mobile devices makes consumers of these equipment do more constant exchanges, with this the obsolete devices discarding becomes an environmental problem, due its landfill accumulation. In order to minimize the environmental impacts caused by these equipment there's the recycling necessity of polymers, ceramic and metallic materials. The main goal of this recycling's to study the printed circuit board of device mobile processing through ore treatment in order to recoup copper. Initially were processed printed circuit in a knives mill, in order to liberate the ferrous material, after that was done the magnetic separation of the reduced residue. With the not magnetic fraction it was done the grain sized analysis and sulfuric acid and hydrogen peroxide leaching analysis. With the chemical analysis results done with the leaching aliquot it was possible to observe that the leaching method with sulfuric acid and hydrogen peroxide presented greater copper recovery

9:55 AM Question and Answer Period

10:05 AM Invited
Recovery Of Components And Valuable Metals From Printed Circuit Boards: Young Park[1]; Robert Gibson[1]; *Derek Fray*[1]; [1]University of Cambridge

Printed circuit boards, free of iron and aluminium, were submerged in fluoroboric acid, selectively dissolving the solder and allowing the components, in working order, to be harvested. The boards were then shredded and the copper dissolved in ammonia/ammonium carbonate and subsequently electrowon. Aqua regia was used to leach the remaining metals and 95 wt% of the silver, 93 wt% of the palladium and 97 wt% of the gold were recovered. Zinc and nickel were also recovered from the aqua regia solution.

10:25 AM Break

10:45 AM
Recovery of Metals from Electronic Scrap by Hydrometallurgical Route: *Nikhil Dhawan*[1]; Vinay Kumar[2]; Manoj Kumar[2]; [1]Punjab Engineering College; [2]NML

Electronic waste is a collective name given to discarded electronic devices such as Television, Cellular Phones and Computers. Among the heterogeneous metals present in the e-waste are the base metals: copper, aluminium, nickel, tin, iron and precious metals: gold, silver and platinum apart from several hazardous and halogens metals. Hence there is a need to recover these metals by recycling and then re-use to meet large amount of metals demand. Hydrometallurgical processing was employed for the recovery of metals from TV PCB. Different lixiviants such as hydrochloric acid, Sulphuric acid, and nitric acid were used to understand the dissolution behavior of copper, iron and lead present in the TV PCB. Effect of increasing concentration of nitric acid was also studied. Selective recovery of tin over 95% in the form of tin oxide was precipitated and was identified by XRD technique. Tin oxide of purity more than 99.9% was obtained.

11:05 AM Question and Answer Period

11:15 AM Invited
Leaching Behaviour Of Metals From Waste Printed Circuit Boards (PCBs) In Acidic Medium: Manis Jha[1]; *Jae-chun Lee*[1]; Nghiem Nguyen[1]; Kyoungkeun Yoo[1]; Jinki Jeong[1]; [1]Korea Institute of Geoscience and Mineral Resources

The leaching behaviour of metals from waste printed circuit boards (PCBs) in acidic medium has been reported. The waste PCBs obtained from personal computer (PC) contains Cu, Ni, Fe, Pb, Sn as major metallic constituents and precious metals as minor. Batch experiments were carried out to investigate the leaching behaviour of metals from crushed PCBs in various acidic medium viz. hydrochloric, nitric and sulphuric acids with hydrogen peroxide as additive. Further, studies have been carried out by varying various process parameters viz. temperature, particle size, leaching time, pulp density, acid concentration etc. Lead forms unstable complex with nitric acid during the leaching. Therefore, leaching and precipitation behaviour of Pb with nitric acid was also studied. The obtained leach liquor could be used for the recovery of valuables as metal or salt by electrolysis or crystallization, respectively.

11:35 AM Question and Answer Period

11:45 AM
A Recovery Technology Of Ag From Composite Ag-Cu Electronic Wastes: *Jinhui Li*[1]; Xinhai Li[1]; Daoling Xiong[2]; Qiyang Hu[1]; Zhixing Wang[1]; [1]Central South University; [2]Jiangxi University of Science and Technology

Silver is a very important metal used in a wide range of applications. It is necessary to recovery of Ag from electronic wastes for increasing of Ag price and circumstance protection. For separating and recycling Ag from Ag-Cu composite materials, thermodynamic data of reaction is calculated and some experiments have been proceeded with Ag-Cu composite material scraps, time, temperature, nitric-sulfuric mixed acid volume ratio and the volume ration of mixed acid vs. water have been discussed. The results show that the optimization conditions is that nitric-sulfuric mixed acid volume ratio 5:95, volume ration of mixed acid vs. water 10:1, temperature 55°, time 25min. In the conditions, Ag can be selectively leached from Cu-based composite material, Cu base is not eroded nearly.

12:05 PM Question and Answer Period

Shape Casting: Third International Symposium: Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Solidification Committee, TMS: Aluminum Processing Committee
Program Organizers: John Campbell, University of Birmingham; Paul Crepeau, General Motors Corp; Murat Tiryakioglu, Robert Morris University

Tuesday AM — Room: 2011
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Sumanth Shankar, McMaster University; Srinath Viswanathan, University of Alabama

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8:30 AM Introductory Comments

8:35 AM
Oxide Film and Porosity Defects in Magnesium Alloy AZ91: *Liang Wang*[1]; Hongjoo Rhee[1]; Sergio Felicelli[2]; Adrian Sabau[3]; John Berry[2]; [1]Center for Advanced Vehicular Systems, Mississippi State University; [2]Center for Advanced Vehicular Systems and Mechanical Engineering Department, Mississippi State University; [3]Metals and Ceramics Division, Oak Ridge National Laboratory

Porosity is a major concern in the production of light metal parts. This work aims to identify some of the mechanisms of microporosity formation during the gravity-poured castings of magnesium alloy AZ91. Two graphite plate molds and a ceramic cylindrical mold were selected to produce a wide range of cooling rates. Temperature data during cooling was acquired with type K thermocouples at 60 Hz in two locations of each casting. The microstructure of samples extracted from the regions of measured temperature was then characterized with x-ray computed tomography and optical metallography. The gathered data was analyzed to search for correlations between cooling rate, dendrite arm spacing, pore volume fraction and pore size. The experimental outcomes were compared with simulations performed with a finite element continuum model of dendritic solidification. The results of this study confirm some of the findings observed in similarly cast aluminum alloys.

9:00 AM
Assessing Casting Quality Using Computed Tomography with Advanced Visualization Techniques: *Georg Geier*[1]; Joerdis Rosc[1]; Markus Hadwiger[2]; Laura Fritz[2]; Daniel Habe[1]; Thomas Pabel[1]; Peter Schumacher[1]; [1]Austrian Foundry Research Insitute; [2]VRVis Research Center for Virtual Reality and Visualization, Ltd

Increasing demand for high quality castings has increased the importance of the use of computed tomography (CT) in the casting industry. The major advantages of computed tomography include its ability to cover the whole sample-volume with respect to apparent differences in density within the object and being able to determine their size and position in three dimensions. The possibility of the detection and quantification of varied casting defects makes it a valuable tool.This paper deals with the possibilities and limits of the use of computed tomography for quality control and assessment in the casting industry from the materials to finished castings. Considerable improvements can be achieved using volume rendering with novel multi-dimensional transfer functions for the visualization of the volume data. In particular the quantification of casting defects will be addressed and compared to standard metallographic procedures and common CT analysis-tools.

9:25 AM
Reconstruction, Visualization, and Quantitative Characterization of Multi-phase Three-Dimensional Microstructures of Cast Aluminum Alloys: Harpreet Singh[1]; *Arun Gokhale*[1]; Yuxiong Mao[1]; Asim Tewari[2]; [1]Georgia Institute of Technology; [2]General Motors Corporation

Serial sectioning technique is well known for reconstruction of three-dimensional microstructures of opaque materials. During the recent years, techniques have also been developed for reconstruction of high fidelity large volume segments of three-dimensional microstructures using montage serial sections; robot assisted automated acquisitions of montage serial sections are also reported. Nonetheless, the past work of three-dimensional microstructure reconstruction from serial sections is restricted to microstructures containing at the most two phases, or in the multi-phase microstructures, the three-dimensional geometry of only one or two phases is reconstructed. In this contribution, we present three-dimensional reconstruction of multi-phase microstructures of a series of cast Al-alloys containing porosity, Si particles, and numerous intermetallic inclusion phases. All the phases are segmented and separately reconstructed, rendered, and quantitatively characterized in three-dimensions, which clearly brings out the complex three-dimensional morphologies of all phases. The technique is useful for characterization of any multi-phase three-dimensional microstructure.

9:50 AM
Correlation of Thermal, Tensile and Corrosion Parameters of Zn-Al Alloys with Columnar, Equiaxed and Transition Structures: Alicia Ares[1]; Liliana Gassa[2]; Sergio Gueijman[3]; *Carlos Schvezov*[1]; [1]CONICET/National University of Misiones; [2]CONICET/INIFTA; [3]National University of Misiones

The columnar to equiaxed transition (CET) has been examined in different wrought and casting alloys for many years and the metallurgical significance of CET has been treated in several articles. Experimental observations in the literature have focused on thermal parameters like cooling rate, velocity of the liquidus and solidus fronts, local solidification time, temperature gradients and recalescence. The objective of the present research consist on studying the influence of solidification thermal parameters on the type of structure (columnar, equiaxial or with the CET); and on the dendritic spacing (primary and secondary) in Zn-Al alloys (Zn-1%Al to Zn-4wt%Al, weight percent). Also, correlate the thermal and structure parameters of these alloys with tensile and corrosion behaviour. The results show that the CET zone and the equiaxed structures presented a better tensile and corrosion resistance than the columnar zone.

10:15 AM Break

10:25 AM
Solidification, Macrostructure and Microstructure Analysis of Al-Cu Alloys Directionally Solidified from the Chill Face: Alicia Ares[1]; *Carlos Schvezov*[1]; [1]CONICET/Univ De Misiones

The understanding of the phenomenon of the columnar to equiaxed transition (CET) is very important for metallurgical applications. In the present study the CET was observed in aluminum-copper alloys of different compositions covering a range from 2wt%Cu to 33.2 wt%Cu, which were solidified directionally from a chill face. The main parameters analyzed include cooling rates, temperature gradients, solidification velocities of the liquidus and solidus fronts, recalescence, heat flow, grain size, primary and secondary dendritic arm spacing and eutectic spacing. The temperature gradient and the velocity of the liquidus front reach low critical values before the transition. These critical values are between 0.35 to 3.12 mm/s for the velocity and -0.44 to 0.17 K/mm for the temperature gradient. The temperature measurements indicate that solidification in the transition region is far from equilibrium given by the lever rule and the phase diagram.

10:50 AM
The Modification of Cast Al-Mg2Si Metal Matrix Composite by Li: *Raheleh Hadian*[1]; Mahmood Emamy[2]; John Campbell[3]; [1]Sharif University of Technology; [2]University of Tehran; [3]University of Birmingham

The effects of both Li modification and cooling rate on the microstructure and tensile properties of an in-situ prepared Al-15% Mg2Si composite were investigated. The size of Mg2Si particles was refined and tensile properties were improved as a result of both 300 ppm Li additions and cooling rate increases, and these effects were additive. The refinement by Li and enhanced cooling rate is discussed in terms of an analogy with the effect of Sr and cooling rate in Al-Si alloys, and is ultimately attributed to the effect of the alkali and alkaline earth metals deactivating extrinsic (entrained oxide bifilms) suspended in Al melts as favoured substrates for intermetallics.

11:15 AM
Effect of Strontium on Viscosity and Liquid Structure of Al-Si Eutectic Alloy: Sumanth Shankar[1]; *Srirangam VS Prakash*[1]; Minhajuddin Malik[1]; Manickaraj Jeyakumar[1]; Michael Walker[2]; Mohamed Hamed[1]; [1]LMCRC - McMaster University; [2]General Motors

This study aims to present conclusive evidence that trace level addition of Sr in Al-Si hypoeutectic alloys change the liquid melt characteristics and alter the nucleation environment of the eutectic phases. High temperature rheological experiments measuring viscosity of Al-Si eutectic melt with and without Sr addition show that Sr significantly alters the melt viscosities at various shear rate regimes. Further, liquid diffraction experiments have been carried out on Al-12.5wt%Si (eutectic) alloy using high-energy synchrotron X-ray beam source to determine the effect of Sr on various liquid structure parameters such as structure factor, pair distribution function and coordination numbers at various melt superheat temperatures. The analysis of the data suggests that Sr changes the nucleation environment of the eutectic Si phase. Further, the effect of Sr on the atomic arrangement of the Si atom with respect of Si and Al atoms in the liquid will be quantified and presented.

11:40 AM
Characterization of the Melt Quality and Impurity Content of an LM25 Alloy: *Katharina Haberl*[1]; Peter Schumacher[2]; Georg Geier[3]; Bernhard Stauder[4]; [1]University of Leoben; [2]University of Leoben and Austrian Foundry Research Institute; [3]Austrian Foundry Research Institute; [4]Nemak

The melt quality of an LM25 aluminium casting alloy has been examined using reduced pressure test (RPT) measurements, porous disc filtration analysis (PoDFA), and fatigue and tensile tests. The aim of this study was to determine existing melt quality and thus evaluate methods used with respect to monitoring and improving melt cleanliness. Special emphasis was given to the influence of oxides. It was found that the melt quality has varying degrees of effect on the tests used. Results in particular indicate, that it was necessary to distinguish between "new" oxides and "hard" inclusions in the melt, as new oxides impact porosity whilst hard inclusions impact on ductility. Based on the results of this study, suggestions for the measurement of the melt quality have been proposed.

Structural Materials Division Symposium: Advanced Characterization and Modeling of Phase Transformations in Metals in Honor of David N. Seidman on his 70th Birthday: Structure Property Relationships

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Robert Averback, University of Illinois, Urbana-Champaign; Mark Asta, University of California, Davis; David Dunand, Northwestern University; Ian Robertson, University of Illinois at Urbana-Champaign; Stephen Foiles

Tuesday AM   Room: 3000
February 17, 2009   Location: Moscone West Convention Center

Session Chair: Stephen Foiles, Sandia National Laboratories

8:30 AM Invited
Mechanical Consequences of Grain Boundary Structure: *John Cahn*[1]; V. Ivanov[2]; Y. Mishin[2]; [1]University of Washington; [2]George Mason University
Most moving grain boundaries (GBs), not just small-angle dislocation and twin boundaries, deform and rotate the material traversed with important consequences for such processes as grain growth and recrystallization. Applied stresses couple to these GBs, leading to their motion and the deformations. For any given GB the structure and bicrystal symmetry play important roles, but there always are multiple sol utions t n for the GB dislocation content. GB structure is important for understanding the atomic mechanisms for the GB motions that are realized in molecular dynamics simulations, including for the multiple coupling modes and abrupt changes in the direction of the GB motion. We discuss recent progress in understanding the stress-driven GB motion and its role in mechanical behavior of materials.

9:00 AM Invited
A New Paradigm For Designing Strong Ductile Alloys With High Peierls Stress: *Morris Fine*[1]; Semyon Vaynman[1]; [1]Northwestern University
Interaction of coherent misfit centers with dislocations to locally lower the Peierls stress is the concept for making metals and alloys more ductile below a ductile to brittle transformation temperature such as occurs in steels. Hans Weertman, circa 1958 developed the basic theory. The misfit centers catalyze the formation of double kinks such as in screw dislocations in iron at low temperatures. We used Han's theory to explain why low carbon ferritic steels with low nanoscale coherent Cu alloy precipitates can have outstanding ductility and high Charpy impact fracture energies at cryogenic temperatures, below the usual ductile to brittle transformation temperature. These ideas are being extended to very high strength steels and to other metals and alloys. David Seidman and his group did the precipitate characterization using atom probe tomography, a key part of the research.

9:30 AM Invited
Grain Boundary Dissociation in Low Stacking Fault Energy Metals: *Douglas Medlin*[1]; John Hamilton[1]; Gene Lucadamo[1]; [1]Sandia National Laboratories
Grain boundaries in metals that possess low stacking fault energies can reconstruct into three-dimensional configurations by the emission of stacking faults. An important question is how the arrangement of these faults, and hence the structure of the interfacial layer, depends on the orientational parameters of the interface. Here, we present electron microscopic observations and modeling of two boundary misorientations in gold that both reconstruct to form a nanometer-scale layer of hexagonal-close-packed (HCP) material. In both cases, the HCP layer and its relationship to the grain misorientation is directly explained and predicted by the arrangement of Shockley partial dislocations at the interface. A comparison of the two boundary structures, one of which has partials paired as full lattice dislocations and the other, which does not, provides insight concerning the formation of other stacking arrangements, such as 9R, that have been observed at other grain misorientations in low SFE FCC metals.

10:00 AM Break

10:15 AM Invited
Modeling Point, Line, and Planar Defects in Metal Alloys: *Christopher Woodward*[1]; [1]Air Force Research Laboratory
Often solution and precipitation strengthening strategies are used to optimize properties of structural materials for aerospace applications. Much can be learned about these alloys by studying defects in model simple, binary and ternary systems. We review first principles predictions of point, line and planar effects in simple bcc metals and several ordered intermetallics. These include: enthalpy of point defects in \\947-TiAl-X and \\947-Al_3Sc-X; dislocation core structure in bcc and fcc metals (including solute-dislocation interactions), and interfacial boundaries (IFB) in Ni-Ni_3Al and Al-Al_3Sc. Using thermodynamics we can predict concentration and temperature dependence of ternary-solute site selection in the ordered intermetallics. Predicted solute-dislocation interactions in Mo-X alloys are used to develop new model of solution hardening and softening. Finally, cluster expansion and lattice gas methods are used to study composition profiles and free energies of IFB's. Results for Mg impurity segregation to (100)Al-Al_3Sc IFB have been verified by atom probe tomography.

10:45 AM Invited
Structural Stability Issues in Nanocrystalline, Ultrafine-Grain and Nano-Twinned Copper: Carla Shute[1]; Kai Zhang[1]; Andrea Hodge[2]; Benjamin Myers[1]; Sujing Xie[1]; *Julia Weertman*[1]; [1]Northwestern University; [2]University of Southern California
The mechanical behavior of pure nanocrystalline and ultrafine-grain (UFG) materials are of interest because of likely changes in deformation mechanisms in these grain size regimes. However it is found that the internal structure is unstable, especially under stress. Localized stress has been found to increase average grain size by a factor of more than 5 in high-purity nanocrystalline Cu. Fatigue loading of UFG Cu increases the grain size by orders of magnitude. However aligned nano-twinning considerably stabilizes samples subjected to fatigue or localized stresses. Recent experiments on nano-twinned Cu will be discussed. Characterization was performed in the EPIC facility NUANCE Center, supported by NSF-NSEC, NSF-MRSEC, Keck Foundation, State of Illinois, and Northwestern University.

11:15 AM Invited
Stability of Nanocrystalline Ni-W Electrodeposits: *Christopher Schuh*[1]; T. Rupert[1]; T. Ziebell[1]; [1]Massachusetts Institute of Technology
Electrodeposited Ni-W alloys are used as coatings for improved mechanical performance, and derive their desirable properties from very fine nanoscale structures. This talk will survey our work to characterize the coating structure both in the as-deposited state, and after exposure to thermal treatment and mechanical deformation. In the as-deposited condition, these alloys exhibit tensile residual stresses, disordered grain boundaries, as well as some degree of grain boundary segregation. For thermal exposures of technological relevance, we observe grain boundary relaxation and hardening without any other structural changes, including grain growth, additional segregation, or precipitation of second phases. Severe mechanical deformation imposed by wear and abrasion also leads to structural relaxation that has broad implications for the use of these coatings in wear applications.

11:45 AM Invited
Defect Generation And Stabilisation As A Route To Nanostructured And Amorphous Materials: *Reiner Kirchheim*[1]; [1]University of Goettingen
Willard Gibbs Adsorption Isotherm and Carl Wagner's definition of excess solute at surfaces and grain boundaries were both extended to include other crystalline defects like dislocations and vacancies [1]. Thus solute segregation to dislocations and vacancies and other crystalline defects gives rise to a reduction of their formation energies, too. The Gibbs Adsorption Isotherm remains to be unchanged by its generalization. Thus defect formation requires less energy for positive excess solute, i.e. attractive interaction between solutes and defects. In this context special attention is paid to the intriguing question whether defect energies might become zero or negative leading to metastable equilibrium or instable crystalline phases. However, the corresponding high chemical potentials may not be reached as solute A may precipitate as pure A or an A-rich compound [2]. [1]R. Kirchheim, Acta Mater. 55 (2007) 5129-5138 and 5138-5148. [2]R. Kirchheim, Acta Mater. 50 (2002) 413-419.

12:15 PM
Forced Chemical Mixing In Alloys At Elevated Temperatures: *Nhon Vo*[1]; Samson Odunuga[1]; Robert Averback[1]; Pascal Bellon[1]; [1]University of Illinois

Severe plastic deformation of alloys at low temperatures generally leads to the homogenization of alloying components regardless of their thermochemical properties. At elevated temperatures this is no longer the case as thermally activated jumps of point defects act in competition with the shearing until a dynamic equilibrium is established. In the present work we investigate this behavior by using molecular dynamics computer simulation to calculate the response of a series of immiscible alloys to high shear-rate deformation as a function of temperature, deformation rate, and the heat of mixing. We demonstrate that systems with large heats of mixing indeed undergo phase separation at high temperatures. For the high strain rates employed, however, $5 \times 10^{8} – 5 \times 10^{9}$ s^{-1}, we find that point defects have only minor impact on the behavior and that the phase separation results directly from the shearing events, themselves.

Synergies of Computational and Experimental Materials Science: Three-Dimensional Materials Science III

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Katsuyo Thornton, University of Michigan; Henning Poulsen, Risoe National Laboratory; Mei Li, Ford Motor Company

Tuesday AM — Room: 3003
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Alexis Lewis, Naval Research Laboratory; David Rowenhorst, Naval Research Laboratory

8:30 AM Invited
Experimental and Modeling Synergies in High Temperature Materials: *Tresa Pollock*[1]; Jonathan Madison[1]; Sara Johnson[1]; [1]University of Michigan

High temperature materials experience complex, aggressive environments during processing and in service. Two examples of the benefits of a combined computational-experimental approach will be discussed for nickel base superalloys. In the first, three-dimensional serial sectioning of the growth front of a superalloy single crystal is utilized to provide a computational mesh of a realistic dendritic structure. This provides the basis for a 3-D fluid flow model that permits assessment of the permeability of the dendritic structure and the tendency for convective instabilities to develop during single crystal solidification. In the second example, materials thermodynamic and strengthening models are combined with a thermostructural code to search for materials design solutions to the complex problem of actively cooled structural panels for hypersonic flight vehicles. New experimental materials motivated by this approach will be discussed.

9:10 AM Invited
Integration of 3D Structure Information for a Ni-Base Superalloy into Computational Models for Behavior Prediction: *Michael Groeber*[1]; Dennis Dimiduk[2]; Michael Uchic[2]; Chris Woodward[2]; [1]UTC/Air Force Research Laboratory; [2]Air Force Research Laboratory

There is a drive to utilize modern advances in computational power to expedite development and enhance utilization of materials. Thus, there is a demand for virtual representations of material structure. Predicting material response using computational tools demands that microstructure is accurately described, either statistically or explicitly. Additionally, descriptions of microstructure must be integrated with property simulators. Applications of any such characterization-representation framework may well span from material design to life prediction. This talk will focus on the experimental collection of the 3D structure information for a Ni-Base superalloy turbine blade. Further, the talk will present developments in the integration of this information within computational models. Details of the serial-section collection methods will be presented, followed by descriptions of tools used to identify and measure features of interest. Lastly, the development of computational tools to represent the microstructure both explicitly and statistically will be discussed

9:50 AM
Modeling Fluid Flow Within an Experimentally Obtained Three-Dimensional Solid-Liquid Interface in Directionally Solidified Nickel-Base Superalloy: *Jonathan Madison*[1]; Jonathan Spowart[2]; David Rowenhorst[3]; Katsuyo Thornton[1]; Tresa Pollock[1]; [1]The University of Michigan; [2]US Air Force; [3]Naval Research Laboratory

Convective flow within the mushy zone of directionally solidified superalloys can result in the formation of freckles and misoriented grains. These defects signal not only a disruption in the columnar or single crystal nature of the component but also a tendency toward reduction in life and performance. Approximations of the onset of convective flow in the mush have primarily used the Rayleigh criteria as a predictor for the occurrence of freckles. However, a detailed understanding of fluid flow at the scale of the dendritic structure is still lacking. This research utilizes 3-D dendritic structures obtained from the solid-liquid interface of directionally solidified nickel-base superalloys as direct inputs to fluid flow models. These models have been utilized to assess the permeability of the dendritic array. Implications of simulations will be discussed with reference to the Rayleigh criteria and freckle prediction.

10:10 AM
3-D Moment Invariants For Description Of Precipitate Morphology And Evolution In Nickel Based Superalloys: *Jeremiah MacSleyne*[1]; Marc DeGraef[1]; [1]Carnegie Mellon University

The quantitative description of 3-D shapes is of fundamental importance to microstructural characterization. One method to describe a microstructure is to characterize the shapes of individual precipitates. This characterization has typically been limited to particle size, aspect-ratio, and qualitative descriptors. In general, these are insufficient and do not provide an adequate characterization in a way that allows for direct comparison between microstructures. This is evident during microstructure evolution when changes in precipitate morphology occur or when precipitates exhibit complex shapes. We will show how moment invariants (combinations of second order shape moments that are invariant w.r.t. affine or similarity transformations) can be used as sensitive shape discriminators in 3-D. As an application of 3-D moment invariants, we will examine the morphological evolution of gamma-prime precipitates in a Ru containing single crystal nickel-based superalloy. Experimental data has been collected using automated FIB-based serial sectioning for different aging times.

10:30 AM Break

10:50 AM
Utility of Experimental 3D Microstructure Reconstructions for Simulations of Realistic 3D Microstructures: *Arun Gokhale*[1]; Youxiong Mao[1]; Harpreet Singh[1]; Arun Sreeranganathan[2]; [1]Georgia Institute of Technology; [2]Stress Engineering Services, Inc.

Current methodologies for microstructure simulations involve idealized simple particle/feature shapes; uniform-random spatial distribution of microstructural features; and isotropic feature orientations. However, the corresponding "real" microstructures often have complex feature shapes/morphologies; non-random/non-uniform spatial distributions; and partially anisotropic feature orientations. Consequently, such simulations do not capture these aspects of microstructural reality. In this contribution, we present a methodology that enables simulations of "realistic" 3D microstructures where feature shapes/morphologies, spatial arrangement, and feature orientations are statistically similar to those in the corresponding real microstructures. The realistic complex feature shapes/morphologies are obtained via experimental reconstructions of 3D microstructures from serial sections. The methodology is applied for simulations of realistic 3D microstructures discontinuously reinforced Al-alloy composites. The methodology enables generation of a set of "virtual" microstructures that cover a wide range of process conditions, which can be implemented in finite element (FE) based computations to simulate mechanical response of the corresponding virtual materials.

11:10 AM
The Use Of Correlation Functions To Identify Features In Ti-Based Alloys: *Stephen Niezgoda*[1]; Peter Collins[2]; David Turner[1]; Surya Kalidindi[1]; Hamish Fraser[2]; [1]Drexel University [2]Ohio State University

The need to robustly and automatically identify certain features within two-dimensional and three-dimensional titanium microstructures has led to the development of novel techniques, based on 2-point and higher order microstructure correlations (n- point statistics) and local neighborhood statistics

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calculated via fast Fourier transforms (FFT) and other fast integral transforms, including Radon and Hough transforms. These methodologies have been successfully used to automatically identify critical microstructural features from two-dimensional micrographs of different Ti-based alloys, including colony boundaries - an often difficult feature to automatically identify. Microstructural features such as chord length distributions and interface area, typically estimated by stereological techniques, can be directly calculated from these correlations. These methodologies have been applied to existing datasets of Ti-based alloys, including Ti-6Al-4V and Ti-5553 (Ti-5Al-5V-5Mo-3Cr). The results will be compared with those results obtained using traditional manual or semi-automated procedures.

11:30 AM
Morphological Analysis of 3D Grain Topology in Ti-21S: *David Rowenhorst*[1]; Alexis Lewis[1]; George Spanos[1]; [1]Naval Research Laboratory

Using serial sectioning, the 3D morphology of a statistically significant number of β grains was determined in Ti-21S. Over 200 sections were collected, with a total of 4700 grains within the collection volume. Using the 3D information, direct comparisons are made between this experimental data and theory and simulation of grain growth and topology. This will include discussions of traditional measurements such as grain size distribution, but also the relationships in the topology, including the number of faces and edges within the grains, and their relationship to the the interfacial curvature. We will also show that this type of experimental data is ideal for inclusion into simulations (both FEM and Phase-Field Modeling) as initial conditions, removing many assumptions in the modeling process.

11:50 AM
3D Phase Field Simulation on Beta" Precipitation Kinetics in Al-Mg-Si Alloys: *Ruijie Zhang*[1]; Mei Li[1]; John Allison[1]; Longqing Chen[2]; [1]Ford Motor Company; [2]The Pennsylvania State University

Beta" precipitates always appear at the peak aging condition and act as the most effective strengthening phase during heat treatment process in Al-Mg-Si alloys. In this paper, a phase field model for studying the growth kinetics of beta" precipitates was developed. An experimental nucleation model was adopted to predict the nucleation behavior of beta" precipitates. The Gibbs energy for solid solution and solute diffusion behavior were obtained from thermodynamics and diffusion mobility database using CALPHAD method. The Gibbs free energy, interface energy and elastic constants of beta" precipitates were selected from the results of first-principles calculations. Because there is no direct experimental data on the interface mobility, this parameter was optimized by several published experimental results. Good agreements were achieved between predictions and experimental results, such as precipitate size and volume fraction. These parameters are key factors for the description of ageing behavior and for the mechanical properties predictions.

Transformations under Extreme Conditions: A New Frontier in Materials: High Rate Deformation

Sponsored by: The Minerals, Metals and Materials Society, ASM International, ASM Materials Science Critical Technology Sector, TMS Materials Processing and Manufacturing Division, TMS/ASM: Phase Transformations Committee
Program Organizers: Vijay Vasudevan, University of Cincinnati; Mukul Kumar, Lawrence Livermore National Laboratory; Marc Meyers, University of California-San Diego; George "Rusty" Gray, Los Alamos National Laboratory; Dan Thoma, Los Alamos National Laboratory

Tuesday AM — Room: 3001
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Marc Meyers, University of California; Naresh Thadhani, Georgia Institute of Technology

8:30 AM Invited
A Path to Materials Science above 1000 GPa (10 Mbar) on the NIF Laser: *Bruce Remington*[1]; Hye-Sook Park[1]; Shon Prisbrey[1]; Stephen Pollaine[1]; Luke Hsiung[1]; Robert Rudd[1]; Robert Cavallo[1]; Stefan Hau-Riege[1]; Justin Wark[2]; Marc Meyers[3]; [1]Lawrence Livermore National Laboratory; [2]University of Oxford; [3]University of California, San Diego

Solid state dynamics experiments at extreme pressures, P > 1000 GPa (10 Mbar), and strain rates (1.e6–1.e8 1/s) are being developed for the National Ignition Facility (NIF) laser, and offer the possibility for exploring new regimes of materials science. These extreme, solid state conditions can be accessed with a ramped pressure drive. Velocity interferometer measurements (VISAR) establish the high pressure conditions. Constitutive models for solid state strength under these conditions are tested by comparing 2D simulations with experiments measuring perturbation growth from the Rayleigh–Taylor or Richtmyer-Meshkov instabilities in solid state samples of V and Ta. Radiography techniques using synchronized bursts of 20-40 keV x-rays have been developed to diagnose this perturbation growth. Time resolved lattice response and phase can be measured with dynamic X-ray diffraction and modeled with large scale molecular dynamics (MD) simulations. Methods proposed for inferring deformation mechanism (slip vs. twinning vs. phonon drag) will also be discussed.

9:05 AM Invited
Shock Deformation in Cubic Metals: *Neil Bourne*[1]; [1]AWE

There is a current need to solve design problems experienced where structures experience dynamic and impact loading. To do this, requires valid, physically-based, analytical laws that describe the deformation behaviour of materials. Populating material descriptions found in such codes with suitable analytical descriptions, generally requires knowledge of operating physical mechanisms at the mesoscale. This work will attempt to provide an overview of present work concerning the shock response of metals focusing upon work done on cubic materials. The materials chosen are pure nickel, pure tantalum and the ordered fcc material Ni3Al. Additionally TiAl is also considered. A range of results from complementary techniques is presented casting light on the operating mechanisms giving rise to the observed phenomena. The behaviour of these metals is discussed in terms of the materials' Peierl's stress, stacking fault energy of the microstructure and twinning prevalence.

9:40 AM
High Rate Plasticity under Pressure Using an Oblique-Impact Ramp Compression Experiment: *Jeffrey Florando*[1]; Louis Ferranti[1]; Grant Bazan[1]; Richard Becker[1]; Roger Minich[1]; Dave Lassila; Tong Jiao[2]; Steve Grunschel[2]; Rodney Clifton[2]; [1]Lawrence Livermore National Laboratory; [2]Brown University

An experimental technique has been developed to study the strength of materials under conditions of moderate pressures and high strain rates. The technique is similar to the traditional pressure-shear experiments except that window interferometry is used to measure both the normal and transverse particle velocities at the sample-window interface. Additionally, the sample is impacted with a graded density impactor, which imposes a ramp compression wave and controls the strain rate to between 10^4 – 10^6. Both simulation and experimental results on copper samples with a sapphire window will be presented to show the utility of the technique to measure the strength properties under dynamic loading conditions.

10:00 AM

Material Strength and Microstructural Effects in Beryllium during Nanosecond Heating by Hard X-Rays: *Eric Loomis*[1]; Scott Greenfield[1]; Shengnian Luo[1]; Randall Johnson[1]; Tom Shimada[1]; Jim Cobble[1]; David Montgomery[1]; [1]Los Alamos National Laboratories

Understanding material behavior at high strain-rates and high temperatures is a formidable problem, requiring complex simulations of dislocation kinetics or experiments. From the experimental standpoint, lasers are a useful tool for inducing such states due to their ability in producing extreme conditions on nanosecond timescales. I will present recent data showing the response of single and polycrystalline beryllium exposed to nanosecond hard x-rays produced by laser-irradiated gold foils. Velocity measurements showed that a suddenly established temperature profile through the target resulted in high temperature tensile ramped loading. Plastic flow played a diminished role in single crystals compared to lower temperature behavior where the plastic deformation is more anisotropic. A large difference in single crystal behavior compared to polycrystalline behavior was observed in velocity measurements as well as surface displacement measurements where anisotropic thermal expansion resulted in observable differential grain expansion at the free surface.

10:20 AM Break

10:35 AM Invited

Characterization of Deformation Bands, Adiabatic Shear Bands, and Crack Formation and Propagation in Ti-6Al-4V Ballistic Plug Targets: *Lawrence Murr*[1]; A. Ramirez[1]; S. Gaytan[1]; M. Lopez[1]; E. Martinez[1]; D. Hernandez[1]; E. Martinez[1]; [1]University of Texas

The microstructures and microstructure evolution associated with deformation bands and adiabatic shear band (ASB) formation in ballistic plugging in thick, Ti-6Al-4V targets impacted by cylindrical, flat-nose 4370 steel projectiles at velocities ranging from 633 m/s to 1027 m/s were investigated by optical and transmission electron microscopy. Deformation bands were composed of transformed alpha-prime (hcp) platelets with spacing decreasing with impact velocity. Horizontal ASB spacing decreased with impact velocity while the ASB width increased. The deformation band microindentation hardness increased with an increase in impact velocity while the ASB microindentation hardness, although 16% higher than the surrounding matrix, remained constant; indicative of a consistent dynamic recrystallization (DRX) grain structure. The deformation bands were not precursors to ASB formation, and cracks nucleated and propagated preferentially in the ASBs, with crack length (0.02 mm at 633 m/s to 10 mm at 1006 m/s) and crack density increasing with impact velocity.

11:10 AM Invited

Influence of the Shock-Induced α-ε Transition in Fe and the α-ω Transitions in Ti and Zr on Post-Shock Substructure Evolution and Mechanical Behavior: *George Gray*[1]; Ellen Cerreta[1]; [1]Los Alamos National Laboratory

Shock loading of materials is well known to induce a range of defects in metals and alloys, including dislocations, deformation twins, and point defects. In addition to the defects generated to accommodate the plasticity of impact loading, some materials exhibit additional structure / property changes due to a pressure-induced phase transition in the material. In this paper, the manner by which the shock-induced α-ε transition in Fe and the α-ω transition in Ti and Zr alters the post-shock substructure evolution and mechanical behavior will be presented. Enhanced defect generation and storage mechanisms, including deformation twinning, and a commensurate increase in shock hardening is shown to occur upon crossing both the α-ε transition in Fe and the α-ω in Ti and Zr. Shock recovery experiments are shown to provide an invaluable window, when coupled with "real-time" diagnostic techniques, into the defect generation and storage processes operative during shock loading of materials.

11:45 AM

Laser Shock Induced Residual Stress and Microstructural Changes in Aero Engine Alloys: Amrinder Gill[1]; Yixiang Zhao[1]; Vibhor Chaswal[1]; Ulrich Lienert[2]; Jonathan Almer[2]; Yang Ren[2]; David Lahrman[3]; Seetha Mannava[1]; Dong Qian[1]; *Vijay Vasudevan*[1]; [1]University of Cincinnati; [2]Argonne National Laboratory; [3]LSP Technologies, Inc.

Laser shock peening (LSP) is a novel surface treatment that generates deep compressive residual stresses and near-surface microstructural changes through shockwaves, thereby leading to dramatic improvements in fatigue strength and crack propagation resistance of aircraft engine parts. In this study, coupons of IN718 and Ti-6Al-4V were LSP-treated at a range of beam energies. Depth-resolved characterization of the residual strains and stresses was achieved using high-energy synchrotron x-ray diffraction at the APS/ANL. The near-surface and through-the-depth changes in microstructure were studied using EBSD/OIM and by TEM of thin foils fabricated using the FIB method. Local property changes were examined using microhardness and micropillar compression tests. Finally, analytical and finite element modeling and simulation were utilized to predict the laser shock induced residual stress and spallation. The results showing the relationship between shock parameters and the residual strain/stress distributions, near-surface microstructure, mechanical properties and tendency for spallation are presented and discussed.

12:05 PM

Microstructural Evolution of Ti-6Al-4V during High Strain Rate Conditions of Metal Cutting: *Lei Dong*[1]; Judy Schneider[1]; [1]Mississippi State University

The microstructural evolution following metal cutting was investigated within the metal chips of Ti-6Al-4V. Metal cutting was used to impose a high strain rate on the order of ~105 s-1 within the primary shear zone as the metal was removed from the workpiece. The initial microstructure of the parent material (PM) was composed of a bi-modal microstructure with coarse prior ß grains and equiaxed primary a located at the boundaries. After metal cutting, the microstructure of the metal chips showed coarsening of the equiaxed primary a grains and ß lamellar. These metallographic findings suggest that the metal chips experienced high temperatures which remained below the ß transus temperature.

12:25 PM

A Technique for Yield-Strength Experiments at Ultra-High Pressure and Strain Rate Using High-Power Laser Pulses: Paul DeMange[1]; J. D. Colvin[1]; H. S. Park[1]; S. M. Pollaine[1]; R. Smith[1]; [1]Lawrence Livermore National Laboratory

High-power laser systems have made it possible to achieve Mbar pressure and MHz strain-rate. A laser pulse drives a shock through a reservoir material which then unloads onto the target specimen. Laser velocimetry measurements at the back surface of the specimen are used to infer the material response. With the advent of this recent capability, a technique for material strength experiments has been proposed in which the pressure wave reverberates within the specimen. The velocity amplitude due to the reflections at the back surface is recorded and an amplitude decrease due to the cumulative resistance to compression by material strength is measured. In this work, a general approach for optimizing the reverberation technique is explored that also includes eliminating the risk of shock and spall. Hydrocode simulations are used to develop a direct-drive target design for yield strength experiments at ultra-high pressure and strain rate.

2009 Functional and Structural Nanomaterials: Fabrication, Properties, and Applications: Nanoscale Fabrication and Devices: Concepts, Approaches and Scale-Up

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

Program Organizers: Gregory Thompson, University of Alabama; Amit Misra, Los Alamos National Laboratory; David Stollberg, Georgia Tech Research Institute; Jiyoung Kim, University of Texas at Dallas; Seong Jin Koh, University of Texas at Arlington; Wonbong Choi, Florida International University; Alexander Howard, Air Force Research Laboratory

Tuesday PM Room: 3020
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Amit Misra, Los Alamos National Laboratory; David Stollberg, Georgia Tech Research Institute

2:00 PM Invited

Functional Nanomaterials and the Birth of Ionic Memory: *Michael Kozicki*[1]; [1]Arizona State University

Scalable devices that switch between widely-separated non-volatile resistance states at extremely low power are highly desirable for applications in nanoscale memory and logic. One promising approach involves the use of nanostructured ion-conducting films. A mobile ion-containing electrolyte sandwiched between two electrodes can constitute a device which reversibly transitions between high and low resistance states. The resistance reduction occurs by the formation of a nanoscale conducting region created by redistribution of the ions. A reverse bias (or in some cases a forward bias) returns the device to its high resistance state. In addition to possessing the speed, endurance, retention, and CMOS compatibility required of future switching elements, such devices can also have excellent scaling prospects due to their low operational energy and demonstrated physical scalability. This paper reviews the materials and functionality of a variety of ionic memory technologies and shows how nanostructure is critical to device operation.

2:30 PM

Diffuse-Interface Field Approach to Simulation of Self- and Guided-Assembly of Charged Particles of Various Shapes and Sizes: *Paul Millett*[1]; Yu Wang[2]; [1]Idaho National Laboratory; [2]Virginia Tech

Recent advances in the ability to control the size, shape, and composition of nanoparticles has significantly broadened the possibilities to create novel mesoscale structures as a result of their "bottom-up" assembly. A particularly efficient approach to facilitate various assembly dynamics is to control the collective electrostatic interactions by tuning the particle charge density, dipole moment, and/or an external electric field. Here, we present a novel mesoscale simulation approach that utilizes diffuse interface fields to capture the dynamic assembly processes for arbitrarily shaped particles with arbitrary charge density and/or dipole moment. We will present results illustrating 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. We find that varying the shapes, relative charge density ratio, as well as the relative number density of each particle type results in vastly different assembly dynamics.

2:45 PM

Large-Scale Fabrication of CMOS Based Single-Electron Transistors: *Vishva Ray*[1]; Ramkumar Subramanian[1]; Pradeep Bhadrachalam[1]; Seong Jin Koh[1]; [1]The University of Texas at Arlington

We present a new scheme of fabricating room-temperature single-electron transistors on a large-scale, in parallel processing, and using CMOS based processes. The nanometer scale gap between the source and the drain electrodes, a critical requirement in single-electron devices, was created by employing a vertical electrode configuration. Coulomb islands (10 nm Au nanoparticles) were positioned in the gap between the source and the drain electrodes using a combination of colloidal and surface chemistry. Addressable gate electrodes were also incorporated to fabricate single-electron transistors in complete parallel processing. Single-electron transport phenomena (Coulomb blockade/staircase and Coulomb oscillations) have been demonstrated at room temperature (295K) as well as at low temperature (10K). A shift in the Coulomb staircase due to application of a gate bias, a definitive signature of single-electron transistor behavior has also been demonstrated. Simulations based on the orthodox theory are in very good agreement with the experimental results. (NSF-CAREER(ECS-0449958), ONR(N00014-05-1-0030), THECB(003656-0014-2006).

3:00 PM

Low Temperature Photonic Curing of Nano-particles for Printed Electronic Conductors and Dielectrics: *James Sears*[1]; Steve Smith[1]; Michael Carter[1]; Jeffery West[1]; [1]South Dakota School of Mines and Technology

Photonic Curing is being developed to cure or sinter metal nano-particle based films by exposing them to a brief, intense pulse of light from a xenon flash lamp. This photonic curing technology allows for rapid and selective heating that fuses nano-scale metallic ink particles into functional components. This technology allows the curing or sintering of nanoscale metallic ink patterns on low-temperature substrates including flexible circuit boards, flat panel displays, interconnects, RFID tags, and other disposable electronics without the use of heat. This paper reports on the results obtained after sintering conductive, magnetic, and dielectric nano-particle inks. Sintering was performed with the photonic curing technique developed by NovaCentrix, 2W frequency doubled Nd:YAG CW laser, and a conventional muffle furnace. Sample thickness, micro-structural details, resistivity, and sintering characteristics are also examined and compared for the sintering techniques.

3:15 PM

Nano-Scale Trench Filling Using Atomic Layer Deposition: *Tae Wook Kim*[1]; Jiyoung Kim[1]; Duncan MacFarlane[1]; [1]University of Texas at Dallas

Minimizing the distance needed to redirect a light wave is a key enabling technology for integrated photonic circuits; a recent photonic nanocoupler proposal aims to achieve this through splitting incident light by disrupting total internal reflection with a trench. Fabrication of the trench, requiring a sub-100nm width and a very high aspect ratio, becomes a manufacturing challenge. This constraint, however, is substantially alleviated by filling the trench with a metal oxide. Atomic layer deposition (ALD) is particularly well suited due to excellent conformality on structures with high aspect ratios, and its level of fine control on the film thickness. This study will explore the use of ALD to deposit conformal films consisting of several types of materials including Al2O3 and HfO2, chosen for their high refractive indices necessary for the nanocoupler trenches. Emphasis will be placed on the effects of process parameters, such as cycle time and dose of precursors.

3:30 PM Break

3:45 PM Invited

Rapid Formation Reactions in Nanolayered Foils: Scientific Studies and Commercial Applications: *Timothy Weihs*[1]; [1]Johns Hopkins University

Over the last 15 years we have investigated exothermic formation reactions that self-propagate in nanolayered foils where the layers alternate between materials with negative heats of mixing. These exothermic reactions can reach temperatures as high as 3300 K and can travel at velocities greater than 30 m/s. Using results from ignition experiments, velocity and temperature measurements, and continuum modeling, the physical parameters that control the ignition and the propagation of the reactions will be identified for multiple material systems. Commercial applications of these reactive foils will also be described. Emphasis will be placed on the use of the foils as local heat sources that melt solder and metallurgically bond components without thermal damage. Examples such as the bonding of LEDs to PCBs and the bonding of large sputter targets to backing plates will be provided.

4:15 PM

A Novel Ceramic High Secondary Yield Microchannel Plate: *Raghunandan Seelaboyina*[1]; Indranil Lahiri[1]; Kinzy Jones[1]; Wonbong Choi[1]; [1]Florida International University

In this presentation we will present our recent results on a novel ceramic microchannel plate with high secondary electron yield. Microchannel plates are electron multipliers utilized primarily as an amplification element in various applications. We have employed this unique property of theirs to enhance the field emission current from carbon nanotube emitters. When the microchannel plate was placed above the nanotube cathode, an enhancement of ~18 times in field emission current was achieved. This is attributed to the giant electron multiplication from our novel high secondary emission material inside the channels of the microchannel plate. A bright field emission image also confirmed the field emission enhancement. The current density achieved with the microchannel plate and the nanotube cathode system was ~7.1 mA/cm2. We will also discuss our ongoing work to further improve the performance of our novel system.

4:30 PM
Precise Placement of Single Nanoparticles on a Large Scale: *Pradeep Bhadrachalam*[1]; Hong-Wen Huang[1]; Vishva Ray[1]; Seong Jin Koh[1]; [1]University of Texas at Arlington

The capability of manipulating single nanoparticles with nanoscale precision is one of the key requirements for the fabrication of various nanoparticle-based devices and sensors. We present a novel technique to place exactly one single nanoparticle onto a desired substrate location with nanoscale precision. Importantly, the single-nanoparticle placement has been demonstrated by parallel processing over a large area with success rate over 90%. The 20nm gold nanoparticles were positioned onto the target locations through electrostatic guiding structure which was defined using CMOS-compatible technology. The electrostatic guiding structure was made by functionalizing the substrate using self-assembled monolayers (SAMs). The precision of the nanoparticle placement was measured to be ~12±7nm. We also theoretically studied the forces exerted on the nanoparticles that are responsible for single-nanoparticle placement. This was done by calculating electrostatic potential through numerically solving the non-linear Poisson-Boltzmann equation. Very good agreement was found between the calculations and experiments. (NSF-CAREER(ECS-0449958), ONR(N00014-05-1-0030), THECB(003656-0014-2006)).

4:45 PM
Piezoresistive Effect in Nickel Nanostrand - Polymer Composites: *Calvin Gardner*[1]; Oliver Johnson[1]; George Hansen[2]; Brent Adams[1]; David Fullwood[1]; [1]Brigham Young University; [2]Metal Matrix Composites Corp

Piezoresistive effects are observed when using nickel nanostrands as a conductive additive to a polymer matrix. Nickel nanostrands are elemental crystalline filaments engineered with diameters ranging from 50-1000nm and typical aspect ratios exceeding 50:1, and, most importantly, an interconnected highly bifurcated structure. When combined with a pliable polymer, either by mixing or infusion, unique piezoresistive properties result. We present experimental data exploring the decrease in resistivity and its directional dependence under compressive and tensile strain. A nickel nanostrand-silicone matrix composite in particular demonstrated a very large piezoresistive effect, with resistivity decreasing three orders of magnitude under comparatively small strain. The piezoresistive effect alters with changes in temperature, the resistivity decreasing as temperature increases. Further, the impacts of changing the volume fraction, magnetic alignment of the filaments, and fatigue loading are each examined to characterize the distinctive effect. Additionally, we present a preliminary microstructure based finite element model for the piezoresistivity.

5:00 PM
Ultrasonic Processing of Ultrafine Materials: *Qingyou Han*[1]; Clause Xu[2]; [1]Purdue University [2]Hans Tech

Ultrasonic vibration has been used to generate oscillating strain and stress fields in solid materials, and to introduce varying pressure fields in the liquids. This article reports novel techniques for producing nanostructures in bulk materials using ultrasonic vibrations. Two approaches were tested. The first one was to use ultrasonically induced plastic deformation to produce dislocations and vacancies in materials for the formation of nanostructures. This approach was similar to the production of nanostructures using severe plastic deformation. The second approach was to use ultrasonically induced oscillating stress fields to induce repeated phase transformations in the solid materials. Experiments were carried out in 1010 steel using these two approaches. Initial experimental results indicate both approaches led to the formation of nanostructures in bulk materials. The size of the nanostructures obtained using the first approach is smaller than 200 nm and possibly in the range of 100 nm.

5:15 PM
Nano-Manufacturing by Electroforming - A Near Net Forming Process for Manufacturing Complex Parts: *Mohammad Hussain*[1]; [1]KACST

This paper describes the application of electroforming in the synthesis of nano-crystalline nickel coatings/components by very high movement of the electrolyte. A high speed plating equipment has been designed and constructed. Nanocrystalline nickel coatings were formed at a speed of 600 µm per hour, by high speed plating, the grain size of the electrodeposited nickel was considerably reduced by high speed movement of the plating solution. Other processes such as casting, forging, stamping, deep drawing and machining may serve well for most applications. However, when requirements specify high tolerances, complexity, lightweight and miniature geometry, electroforming is a serious contender and in certain cases may be the only economically viable manufacturing process. The electroplated nickel deposits were characterized using SEM, XRD and AFM. The focus of this study is primarily in the application of nano-composites in industrial gas turbines in the synthesis of TBC (Thermal Barrier Coatings).

Alumina and Bauxite: Process Improvements and Experiences - Red Side

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Everett Phillips, Nalco Co; Sringeri Chandrashekar, Dubai Aluminum Co

Tuesday PM — Room: 2002
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Ashish Jog, Dubai Aluminium Company

2:00 PM Introductory Comments

2:05 PM
Advanced Process Control in Alumna Digestion Unit: *Ayana Oliveira*[1]; Jefferson Batista[1]; Jedson Santos[1]; Márcia Ribeiro[1]; Rafael Lopes[2]; Jorge Charr[3]; [1]ALUNORTE - Alumina do Norte do Brasil S.A.; [2]Honeywell do Brasil; [3]Honeywell Venezuela

The most competitive environment generated the need for performance optimization, for this reason a new control technologies seems a challenges for increase yield in alumina plant, using existing infrastructure and requiring a reduced support team. Robust Multivariable Predictive Control Technology becomes one of the main tools to optimize this class of plants. This paper will discuss the application and benefits of this technology to alumina digestion units, implemented in 3 interconnected digesters. The APC philosophy is based on process variability reduction, and consequently operations optimization, against plant constraints. Since alumina – caustic ratio (A/C) is the key plant variable, it has a fundamental role in this variability reduction. The main challenge in this project was to coordinate the use of 5 grinders to the 3 digesters. The implementation for phase I and II, are finished, respectively, in eight and six months generated more than 1.00% increase in production, rather than A/C variability reduction.

2:30 PM
The Red Mud Recycles on Bayer Process and Its Effect on the Iron Content on Liquor: *Ayana Oliveira*[1]; Eliomar Ferreira[2]; Tamara Ribeiro[2]; Angela Avelar[2]; Dorival Santos Jr.[3]; [1]ALUNORTE - Alumina do Norte do Brasil S.A.; [2]VALE - Department of Mineral Project Development; [3]VALE - DIAL

It is well known that as the content of iron on the bauxite decreases its concentration on the liquor is increasing and, as a consequence, its content on alumina increases. It is believed that this soluble iron on the liquor could be due to the presence of Al-goethite and its transformation of hematite during the digestion process. In order to minimize the content of iron on the liquor (DBO), the digestion process was carried out with two bauxite samples with recycles of red mud, fine and coarse part, called sand, at different percentages to reach the Fe_2O_3 grade from 10 to 15% at the process feed. The bauxite and red mud mixtures were digested at 145°C, during 60 minutes, with caustic soda concentration at 280g Na_2CO_3/L and initial an A/C of 0.38 and the final A/C reached the value of 0.74. The results demonstrated that fine red mud added to the bauxite did not contribute to a decrease in the iron content of the liquor, instead, the iron content increased. However, the coarse part of red mud, called sand, decreased the iron in the liquor from 0.027g/L to less than 0.004/L.

2:55 PM
Effect of Lime Quality on Slaking: *David Kirkpatrick*[1]; Don Williamson[1]; Lynn Blankenship[1]; Shawn Kostelak[1]; [1]Gramercy Alumina LLC

At Gramercy Alumina, both water and liquor-slaked lime are used in the Bayer Process. After laboratory slaking and filtration properties testing, two sources were approved as lime suppliers. In 2007, economics drove a change in the source ratio, and soon the slaking operation began experiencing problems with excessive scaling and large pebbles in the slaker discharge. These problems decreased the operational life of the slakers, consequently decreasing the amount of lime available to the process. Gramercy Alumina began investigating the lime properties by conducting laboratory analyses, site visits and plant trials to determine the cause of the scaling. Although standard lime testing could not highlight the problem, additional tests found hydrocarbon residue after slaking from one source. Plant trials determined the lime source of the excessive scaling and the proper blend of the two limes to obtain the best processing characteristics at the best economics.

3:20 PM
Selection of Sedimentation Equipment for the Bayer Process——An Overview of Past and Present Technology: *Tim Laros*[1]; Frank Baczek[1]; [1]FLSmidth Minerals

The Bayer Process relies heavily on sedimentation Equipment in Desilication, Liquor Clarification, Residue Disposal, Tertiary Seed Classification, as well as Caustization and Oxalate Removal. Over the past 20 years, sedimentation equipment design and operating philosophies have changed dramatically with the advent of feed slurry dilution, new flocculants, and robotic descaling. This paper will present and overview of the progression of Bayer Process sedimentation technology and current equipment options available.

3:45 PM Break

4:05 PM
A Novel Chemistry for Improved Aluminate Scale Control in Bayer Process: *Jing Wang*[1]; Harry Li[1]; Kevin O'Brien[1]; [1]Nalco

In the Bayer Process for the production of alumina, auto-precipitation of alumina trihydrate in the decantation, security filtration, and wash circuit results in both alumina losses and problematic deposit buildup on vessel surfaces. Moreover, auto-precipitation is a key limiting factor in improving plant production by limiting the A/C ratio of digestion. As a result, reduction of this scaling and/or prevention of auto-precipitation in these vessels can significantly improve overall plant performance. While chemical methods to control scale formation, such as Nalco's SCFA (Scale Control Filtration Aid) programs, are well established and widely used within the industry, significant opportunity for additional improvement still remains. In this work, a new, more effective, polymer based chemistry for the prevention of trihydrate scale in thickeners, washers and filters has been developed. This paper provides details on this experimental chemistry to further reduce auto-precipitation without downstream effects in the Bayer process.

4:30 PM
Using a Statistical Model in the Red Mud Filtration to Predict the Caustic Concentration in the Red Mud: Américo Borges[1]; *Jorge Aldi*[1]; [1]Alunorte – Alumina do Norte do Brasil S.A

Alunorte began its operation in 1995 with a nominal capacity of 1.1mi tpy and after three expansions in a row the production in 2009 will be around 6.3mi tpy. Alunorte uses dry-stacking technology to dispose the red mud, deep-thickeners in the mud washing circuit and drum filters for mud filtration, reducing the caustic concentration from 65 g/L in the last washers down to 7 g/L at filters discharge. This paper aims to present a DOE pro gram with two levels and five factors in the red mud filtration area. Dilution, rotation, condensate, level in the basin and vacuum has been considered as control variables. The output variable considered was the caustic concentration in the red mud. The program measured the magnitude of the control variables and the influences on the output variable, making possible to model the filtration process, controlling the caustic concentration in the red mud below the target of 7 g/L.

4:55 PM
Formation of Scales in Pipes and Equipments with Kelly Filter Liquor: *Ricardo Galarraga*[1]; Rodolfo Diaz[1]; [1]CVG Bauxilum

All Alumina plants suffer from the problem of scale formation in equipment and pipes. The scale blocks pipes and adds additional resources and time diverted to descaling equipment. Plants have created working groups to control scaling by regular cleaning, changing equipment, and replacing sections of pipe. CVG Bauxilum's "Security Filtration Area" has been able to determine how the scale is created over the years by constant inspections. We have seen a correlation between the build-up of scale in relation to the speed of fluid. We have been able to maintain our operations and equipment by planning and predicting the formation of scale based on the speed of the fluid and the correlation of scale build up. Through our many years of experience we have been able to evaluate the formation of scale and maintain our equipment and operational level thus reducing wear, oxidation, and protecting our equipment against chemical attack.

5:20 PM
The Application of Nepheline in Alumina Industry: *Zhanwei Liu*[1]; Wangxing Li[1]; Wenmi Chen[2]; Bin Liu[1]; [1]Zhengzhou Research Institute of CHALCO; [2]Central South University

The manufacturers of alumina and aluminum manage to make use of local resources to solve the problem of resource shortages because of the rapid increase of alumina productivity. There are amounts of nepheline resources distributed widely in China, the future for nepheline resources is bright. If nepheline resources are exploited and utilized to produce alumina, the byproduct of potash and sodium salt can be attained, so we can get remarkable economic returns. The physicochemical property, geographical distribution and the application of nepheline in alumina industry are reported in this paper.

5:45 PM Concluding Comments

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

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

Tuesday PM Room: 2001
February 17, 2009 Location: Moscone West Convention Center

Session Chair: Subodh Das, Phinix LLC

2:00 PM
Influence of Microstructure of 5754 Aluminum Sheet on Localization Under Uniaxial Loading: *Sooho Kim*[1]; Raja Mishra[1]; Anil Sachdev[1]; Asim Tewari[2]; Pinaki Biswas[2]; Swamydha Vijayalakshmi[2]; Shashank Tiwari[2]; [1]Materials and Processes Laboratory,General Motors R&D Center; [2]India Science Laboratory, General Motors R&D Center

This study compares the material characteristics and influence of microstructure on strain localization in four 5754 aluminum sheets made by direct chill (DC) casting, twin belt continuous casting (TBC) and twin roll casting (TRC). Yield and tensile strengths of the four alloys were essentially similar, but the fracture and forming strains of all sheets were different, decreasing in the order of DC, TBC, and TRC. The particle distribution in the four sheets varied considerably; stringers were observed throughout the sheet thickness in both the TBC and TRC sheets, while centerline segregation was observed only in the TRC sheets. The DC cast sheets showed only randomly distributed particles. The difference in fracture strain was correlated to the spatial distribution of the second phase intermetallic particles. The talk will also discuss grain size and texture differences in the various sheets, and compare actual forming behaviors.

2:20 PM
Development of a Three-Point Bend Test to Evaluate Hemming Performance of Aluminum Sheet: *Susan Hartfield-Wunsch*[1]; John Carsley[1]; [1]General Motors Corp

General Motors' mid-size car platform is the first attempt to manufacture a common, global aluminum hood. To enable the use of common materials, a new global specification was created based on performance rather than on composition. A new three-point bend test procedure was developed to evaluate aluminum sheet alloys in severe bending deformation analogous to hemming. This new three-point bend test was compared to a previously used wrap bend test, and both tests were effective for evaluating and categorizing different aluminum alloys. The three-point bend test offers several advantages including bending the sample to 180 degrees, and automated testing with a controlled punch rate and load measurement. New aluminum sheet alloys with improved bending performance were compared to current production alloys. A pass-fail criterion was established based on visual inspection of bent samples.

2:40 PM
Recrystallization and Texture Evolution in Al-Cu-Li Alloys: *Soonwuk Cheong*[1]; [1]Alcoa Inc

Al-Li alloys have been studied for aerospace applications. Compared to the incumbent non Li containing 2xxx alloys, the Li containing alloys provides attractive density savings in comparable strength and damage tolerance. The present paper discusses the effect of crystallographic texture on the mechanical properties in Al-Cu-Li alloys. Brass texture has been known as a deformation texture component in Al-Cu-Li alloys. The present work introduces Brass texture in a recrystallized microstructure, which presents a better strength and fracture toughness combination compared to Goss textured material. The work also discusses the recrystallization and texture evolution occurring during thermal mechanical processes in Al-Cu-Li alloys for sheet applications.

3:00 PM
Bending Performance of Al-Mg-Si Alloy after Interrupted and Delayed Quench: *Cyrille Bezencon*[1]; Jean-Francois Despois[1]; Juergen Timm[1]; Alok Gupta[2]; Corrado Bassi[1]; [1]Novelis Switzerland SA; [2]Novelis Global Technology Centre, Kingston

The hemming performance of 6xxx alloys is a key requirement for automotive panels' application. This mechanical behaviour can be improved by optimizing the alloy composition and the heat-treatment procedure applied during the sheet rolling process. In this paper, the influence of cooling conditions after partial solutionizing heat-treatment on the bending performance of industrial Al-Mg-Si alloys is assessed. Quenching is performed through a water spray system, allowing to control the temperature at which water quench start or end. Three points bending tests and SEM microstructure analysis have been performed for a range of final quenching temperature (interrupted or step quench) and various waiting time before quenching (delayed quench). It is shown that the bendability is highly dependant on the quenching procedure over a critical temperature range and that bending performance can be correlated to grain boundary precipitation.

3:20 PM
The Effect of Stress Triaxiality and Lode Angle on Failure Strain of 5083-H116 Plate: *Matthew Hayden*[1]; Charles Roe[1]; Xioasheng Gao[2]; [1]Naval Surface Warfare Center; [2]The University of Akron

Increased performance demands on next-generation vehicles are driving the use of structural aluminum alloys. In addition to strength, designers must consider the limited ductility of these complex structural aluminum alloys. Recent literature suggests that stress triaxiality alone does not fully characterize material failure strain. This study presents the experimental measurements of mechanical deformation of aluminum alloy 5083-H116 plate at multiple stress triaxialities and Lode angles. From these experimental observations Johnson-Cook strength and fracture parameters are calibrated with the goal of developing a more comprehensive failure criterion. Shear components represented by the Lode angle exhibited significant effects on failure strain not previously reported.

3:40 PM
The Effect of Microstructure on the Surface Finish of Extruded 6262 Aluminum Alloy Billet: *Qingyou Han*[1]; [1]Purdue University

6262 aluminum alloy is essentially nominal 6061 alloy with additions of lead and bismuth for improved machinability. However, the hot extruded 6262 alloy products suffer a poor surface quality, which varies from ingot to ingot. The intent of this study focuses on the effect of microstructure on the surface quality of the extruded products. The microstructure of the extruded samples is characterized and the microstructure of a sample with a good surface is compared with those of poor surface quality. To our surprise, the extruded samples contain a large number of Mg3Bi2 particles rather than lead-bismuth particles. The microstructure of a sample with a good surface is compared with those of poor surface quality. The volume fraction, size, and size distribution of the Mg3Bi2 are measured. Initial results suggest that the larger the Mg3Bi2 particles, the more negatively the surface quality of the extruded parts are affected.

4:00 PM Break

4:15 PM
Deformation Textures and Plastic Anisotropy of AA6xxx at Warm Temperatures: Manojit Ghosh[1]; *Alexis Miroux*[1]; Jurij Sidor[1]; Leo Kestens[2]; [1]M2i; [2]Delft University of Technology

Tensile and plane strain compression tests as well as deep drawing tests have been used to investigate the forming behaviour of Al-Mg-Si alloys from room temperature to 250ºC. In addition to the expected reduction of yield strength with increasing temperature, it is found that temperature also significantly influences the plastic anisotropy of the sheets. The earing profile of drawn cups show a four-fold symmetry after drawing at room temperature and the r-value is minimum along a direction at 45° from RD. At higher temperature the earing profile presents a 2-fold symmetry and the r-value is minimum along RD. The analysis of the deformed microstructures shows that other slip systems than {111}<110> can be activated at higher temperature. Crystal plasticity calculations reveal that for an adequate combination of {hkl}<110> slip systems a good correspondence between the experimental and calculated r-value, yield locus and textures at different temperatures is obtained.

4:35 PM
Formation of the {111}<110> and {111}<112> Shear Bands and the <111> Fiber Texture during Moderate and Heavy Wire Drawing of 5056 Al-Mg Alloys: *Mohammad Shamsuzzoha*[1]; Fingling Liu[1]; [1]University of Alabama

Metallography, conventional transmission electron microscopy and x-ray diffraction Techniques have been applied to study the microstructure of moderately and heavily wire drawn 5056 Al-Mg alloy. Samples drawn moderately (~70% of the original value) have been found to be comprised of columnar grains with no evidence of any recrytallization and contain deformation bands. Deformation bands are made of closely spaced parallel slip bands, which lie on {111} and extends along a <110> and contribute to the development of a moderate <111> fiber texture. These samples also possess a high residual stress and show material decohesion normal to fiber axis. The matrix of the sample drawn heavily (> 45% of the original value) showed fibrous microstructure with very little evidence of recrystallization, but exhibit a strong <111> texture. And a high residual stress. Deformation bands in these samples have been found to lie on {111} planes but extend along <112>.

4:55 PM
Micromechanics of Ductile Fracture of Aluminum 5083 as a Function of Material Stress State: *Marc Zupan*[1]; Christopher Cheng[1]; Matthew Hayden[2]; Charles Roe[2]; [1]UMBC- University of Maryland, Baltimore County; [2]Naval Surface Warfare Center

The formation of voids, their growth, subsequent ductile rupture, and strain at failure are strongly affected by material element triaxiality. Specifically, investigation of Aluminum 5083 demonstrates promise for insertion into complex weight efficient structural components of vehicles resulting in loading conditions of varied triaxiality. This work will present a ductile fracture failure surface fractography database for Aluminum 5083 loaded monotonically in tension at quasi-static and dynamic strain rates with triaxialities ranging between 0 and 3. The affect of the stress matrix shear components is also evaluated resulting in a failure surface for this alloy. Failure mechanisms including void entourage shearing, cupping, and shear linking are identified. Surface topology measurements are used to evaluate local material strain within the material at the cascade failure event. Failure mechanism maps for the micromechanical failure of this material will be presented.

5:15 PM
Pole Figure Characteristics of Annealed Aluminum Alloy 6061 in Different Magnetic Fields up to 30 Tesla: *Samuel Adedokun*[1]; [1]FAMU-FSU College of Engineering

This work presents the changes in the pole figure characteristics of an aluminum alloy 6061 given 85% deformation by cold rolling and later heat treated at 400C in different magnetic fields of up to 30 Tesla for different periods of time. Pieces of samples from the rolled specimen were heat treated in a resistive magnet of 30 Tesla strength with 50 mm bore. The texture changes in the samples were quantified by carrying out texture measurements through an x-ray diffractometer equipped with a texture goniometer. Changes in the texture with the use of the inverse and complete pole figures indicate that the strength of the magnetic field had no effect on the strength of the texture of the material.

5:35 PM
Comparison of Textures and Microstructures of AA3XXX Hot Bands from Two Different Casting Processes: *Xiyu Wen*[1]; Yansheng Liu[2]; Zhengdong Long[1]; Shridas Ningileri[3]; Tongguang Zhai[1]; Zhong Li[4]; Subodh Das[5]; [1]University of Kentucky; [2]Secat Inc. ; [3]Secat Inc.; [4]Aleris International Inc.; [5]Phinix LLC

Measurements of textures of AA3xxx hotbands made from two different casting routes (twin belt casting vs. the proprietary pellet cast process) by use of the orientation distribution function (ODF) method are carried out, respectively. Their microstructures are observed by use of optical and scanning electronic microscopes. The difference in textures and microstructures resulting from the two processing is studied and presented.

Biological Materials Science: Biological Materials I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Ryan Roeder, University of Notre Dame; John Nychka, University of Alberta; Paul Calvert, University of Massachusetts Dartmouth; Marc Meyers, University of California

Tuesday PM — Room: 3014
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

2:00 PM Keynote
Biological Materials: A New Frontier in MSE: *Marc Meyers*[1]; [1]University of California, San Diego

Biological Materials Science is part of the evolution of MSE from synthetic inorganic materials to organic materials, and finally to biology. It is not a passing fad, but represents a new direction in MSE encompassing three distinct areas: Biological (or natural) materials; Biomaterials (functional and structural); Bioinspired synthesis and processing (biomimetics). MSE brings to bear its unique approach rooted in the structure-property connection. Biological materials are being investigated using a methodology and characterization/testing methods developed by MSE for synthetic materials. This approach is yielding surprisingly rich results and is elucidating the complex hierarchical structures found in nature. We illustrate this approach for shells, crab exoskeletons, bird beaks and feathers, teeth, and bones. Attachment devices used in nature that are inspiring researchers are also reviewed as is current research on biomaterials. Research funding: National Science Foundation Biomaterials Program (DMR).

2:40 PM
Structure and Functional Morphology in Parasitic Wasps: *John Nychka*[1]; C. Andrew Boring[2]; Michael J. Sharkey[2]; [1]University of Alberta; [2]University of Kentucky

The insects have evolved intricate structures regarding many solutions to challenges of their daily life: locomotion, predation, and species propagation to name a few. Interpretation of the morphology of insect structures has long been mysterious. Nonetheless, many engineering designs have been generated from insect design (e.g., anti-reflection coatings on solar panels based on fly eye geometry, and serrated hypodermic needles mimicking the mosquito's proboscis to reduce pain). On the most basic level, the insect cuticle is a mastery of materials design, exhibiting variable and gradient properties via genetic control. This paper will describe the functional morphology of two systems in parasitic wasps, Homolobus truncator (Hymenoptera: Ichneumonoidea: Braconidae), namely the ovipositor (for egg deposition in hosts), and the hamuli-retinaculum (the hook and rail system of the wings). These systems have a multitude of fascinating structures when analyzed with regard to materials science and engineering principles, especially with regard to energy minimization, surface roughness, adhesion, and specific strength.

3:00 PM
Investigation on the Compressive Behavior of Turtle's Shell: Experiment, Modeling, and Simulation: Hongjoo Rhee[1]; Youngkeun Hwang[1]; *Seong Jin Park*[1]; Mark Horstemeyer[1]; [1]Mississippi State University

Turtle shell is a possible candidate as armor material. We investigated the microstructure, chemical composition, and compressive behavior of the turtle shell through the nano-indentor tests, uniaxial compression testing, and a three point bending test. The obtained experimental data were analyzed and modeled using elastic and viscoelastic theory such as Prony series for a similar bone material. The geometry of turtle shell was digitized and converted into mesh for finite element analysis to simulate three-dimensional deformation of turtle shell under compressive condition based on the developed model.

3:20 PM
Biological Composites: Mechanical and Structural Functions of Bird Beaks: *Yasuaki Seki*[1]; Sara Bodde[1]; Marc Meyers[1]; [1]University Of California, San Diego

The mechanical response and sandwich structure of Toco Toucan and Hornbill beaks were investigated. The rhamphotheca is composed of multiple layers of biological composite of keratin tiles. The orientation of intermediate filaments in keratin matrix was revealed by Transmission Electron Microscope (TEM). The diameter of the keratin filament is ~4 nm. The internal foam consists of closed-cell face of trabeculae is closed by lipid face. The mechanical properties of rhamphotheca were evaluated by tensile testing and indentation techniques. Computed Tomography (CT) was employed for characterizing macrostructure of the network of trabeculae. Visualization Toolkit (VTK) was used for creating three dimensional structure of foam. The created model was used for Finite Element, which were compared with experimental results. We have used Dawson and Gibson model in order to evaluate the optimization and stability of bird beaks.

3:40 PM
Sharp Biological Materials: *Yen-Shan Lin*[1]; Eugene Olevsky[2]; Marc Meyers[1]; [1]University Of California, San Diego; [2]san Diego State University

Teeth represent an important natural mineral tissue composed of collagen fibrils and carbonate apatite mineral. Structure and mechanical properties of teeth of a broad range of species including shark, piranha, alligator and hippo are investigated. Hardness test results are compared and show the similar hardness values in different living species. The hardness of the enamel ranges from1.2 to 1.7GPa and the hardness of the dentin is about 0.2 to 0.5GPa. Serrations are observed through SEM analysis for piranha and great white shark teeth with serration sizes of 25μm and 300μm, respectively. The conducted analysis indicates that serrations are used to optimize the biting mechanism. The compressive strength of the teeth was also investigated under longitudinal and transverse loading. The mechanical property of teeth are highly anisotropic due to collagen fibril. Human molar dentins are demineralized and deproteinized through chemical treatment to evaluate the microstructure and test them under compression.

4:00 PM Break

4:10 PM
Effects of Moisturizers on the Biomechanics of Human Skin: *Kemal Levi*[1]; Ka Yiu Alice Kwan[2]; Sumil Thapa[1]; Reinhold Dauskardt[1]; [1]Stanford University; [2]Wellesley College

Moisturizers are widely used in the treatment of skin disorders and their biophysical effects have received extensive attention. However, there remains a significant lack of understanding of how such treatments affect the biomechanical function and responses of human skin. Using a combination of thin-film substrate curvature and bulge techniques, we characterize the stress state of the outermost layer of human skin, stratum corneum (SC), after exposure to well known moisturizers and molecular components of moisturizing treatments. Different classes of moisturizing molecules showed distinctive stress profiles during drying directly demonstrating the efficacy of the treatment. Within the existing classification of moisturizers into humectants and occlusives, occlusives are shown to reduce residual drying stress in SC more effectively than humectants. Finally, the role of the molecules on the SC components including intercellular lipids and corneocyte proteins and their resulting effect on SC stress is examined.

4:30 PM
Structure-Property Relationships in Bovine Meniscus Attachments: *Animish Dande*[1]; Diego Villegas[1]; Tammy Haut Donahue[1]; Reza Shahbazian-Yassar[1]; [1]Michigan Technological University

The meniscus tissue plays a critical role in normal functioning of the knee, and various meniscal replacements have been designed for partial or total replacement of the tissue. The proper attachment of these replacements to the tibial plateau is critical and requires fundamental understanding of the nanomechanical properties of meniscal attachments. The insertion sites of the meniscal horn attachments typically contain four zones: subchondral bone, calcified fibrocartilage, uncalcified fibrocartilage and ligamentous zone. This study aimed to correlate mechanical properties of the various zones with their calcium contents. Bovine meniscus attachments were cut into blocks containing all four zones and characterized using quantitative backscattered electron (qBSE) imaging under the scanning electron microscope (SEM). The nanomechanical characterization was carried out by the atomic force microscopy to determine Young's modulus and Poisson's ratio along with topographical studies. A one-to-one correlation between the calcium content and nanomechanical data was made using the above approach.

4:50 PM

Evolution of Load Partitioning during Creep of Bone Measured by High-Energy X-Ray Diffraction: *Anjali Singhal*[1]; Jonathan Almer[2]; Stuart Stock[1]; Dean Haeffner[2]; David Dunand[1]; [1]Northwestern University; [2]Advanced Photon Source

Bone is a biological composite composed of two intimately mixed solid phases – an organic (protein) matrix, and a discontinuously-distributed reinforcing phase of calcium hydroxyapatite (HaP). An externally applied stress is partitioned by load-transfer between these stiff inorganic particles and the soft organic matrix. By using high-energy X-ray scattering, we determined the bulk in-situ elastic strains in each phase under an applied stress. Compressive creep tests were performed on a bovine femur bone, at different temperatures and stresses. The resulting wide- and small-angle diffraction patterns were used to determine the average phase strains for the two phases. With increasing creep time and stress, the high initial load partitioning between the HaP and protein phases decays. This load-shedding mechanism is discussed in terms of the interplay of viscoelastic deformation of the protein matrix and interfacial damage.

5:10 PM

Synergistic Effect between the Biomineral and Biopolymer Phases in Bone: *Po-Yu Chen*[1]; Damon Toroian[1]; Fred Sheppard[1]; Yu Fu[1]; Paul Price[1]; Joanna McKittrick[1]; [1]University of California

Bone is a composite of two main components: a biopolymer, collagen, and a mineral phase, carbonated hydroxyapatite. The collagen fibrils alternate orientation in the concentric rings that surround the main blood vessels (osteons), and the minerals lie primarily within the collagen fibrils. The purpose of this work was to investigate the structural and mechanical properties of demineralized and deproteinated compact and cancellous bone and to compare to untreated bone. Optical microscope, SEM and TEM observations were made and CT scans were used to reconstruct the 3D structure of both demineralzied and deproteinated samples. We found the concentric ring structure of the osteons to be undisturbed after demineralization. Compression tests on the compact bone showed that the sum of the stress-strain curves for demineralized and deproteinated bone was far lower than that of the untreated bone, indicating a strong molecular interaction between the two phases. (Support: National Science Foundation DMR 0510138).

Bulk Metallic Glasses VI: Structures and Mechanical Properties 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, The University of Tennessee

Tuesday PM   Room: 3007
February 17, 2009   Location: Moscone West Convention Center

Session Chairs: A Greer, University of Cambridge; Katharine Flores, Ohio State University

2:00 PM Keynote

Plastic Deformation of Bulk Metallic Glasses: *A. Greer*[1]; [1]University of Cambridge

Some recent results on the room-temperature plastic deformation of bulk metallic glasses will be reviewed. Particular attention will be paid to the mechanisms of shear banding and to structural changes caused by deformation. There will also be consideration of the relationship of plastic deformation to the elastic properties of the glasses as revealed by resonant ultrasound spectroscopy (RUS).

2:25 PM

Mechanical Properties of a $Zr_{57.4}Cu_{17.9}Ni_{13.4}Al_{10.3}Nb_1$ Bulk Metallic Glass at 300-4.2 K: *Elena Tabachnikova*[1]; Aleksey Podolskiy[1]; Sergey Smirnov[1]; Vladimir Bengus[1]; Peter Liaw[2]; Hongqi Li[2]; [1]B.Verkin Institute for Low Temperature Physics and Engineering of the National Academy of Sciences of Ukraine; [2]University of Tennessee

The mechanical behaviour of the alloy has been studied in a uniaxial compression at temperatures 300, 170, 145, 77 and 4.2 K with strain rate $2 \cdot 10^{-3}$ s^{-1}. Temperature dependences of the yield stress ($\sigma_{0.2}$), ultimate strength (σ_u) and plastic deformation till failure (ε_f) have been measured. Monotonous increase of $\sigma_{0.2}$, σ_u and ε_f have been registered in 300-77 K temperature interval. Thus, value of strength (σ_u) changes from 1640 MPa (at 300 K) to 1970 MPa (at 77 K), and ε_f increases from 0.15% (at 300 K) to 3% (at 77 K). At temperature 4.2 K value of strength σ_u reaches 2010 MPa, but macroscopic plastic deformation have not been observed. At whole investigated temperature interval (down to 4.2 K) the failure of the specimens in the two pieces by sliding-off along planes inclined at 45º relative to the compression axis.

2:35 PM Invited

Characterization of Flow and Fracture in Bulk Metallic Glasses: *Katharine Flores*[1]; Y.C. Jean[2]; Wolfgang Windl[1]; [1]Ohio State University; [2]University of Missouri, Kansas City

In order for bulk metallic glasses to realize their potential as structural materials, mechanisms of plastic deformation and fracture must be understood and controlled. Prior positron lifetime studies have identified three types of open volume sites in several metallic glass families: inherent interstitial sites, flow defects, and sub-nanometer scale voids. These results have been related to topological models for glass structure, as well as the relative fracture toughnesses of the alloys. In the present work, the crack tip damage zone in a Zr-based bulk metallic glass is examined in an effort to better characterize the relationship between shear band formation and crack growth. These experimental observations are discussed in light of computational studies of flow defects in simulated glass structures under tensile, compressive, and shear loading. It has been observed that flow localization in these simulations requires a pre-existing inhomogeneity in the glass structure, such as a void.

2:50 PM

Microstructural Characterization of a 200 nm Thick Glass-Forming Metallic Film for Fatigue-Property Enhancements: Rong Huang[1]; Zhe Zhi Liang[1]; *Jinn P. Chu*[2]; Fengxiao Liu[3]; Peter K. Liaw[3]; [1]National Taiwan Ocean University; [2]National Taiwan University of Science and Technology; [3]University of Tennessee

A 200 nm thick glass-forming metal film, $Cu_{31}Zr_{47}Al_{13}Ni_9$, deposited on the 316L stainless steel substrate using magnetron sputtering, has been investigated by using high resolution transmission electron microscopy (HRTEM) coupled with nanobeam energy x-ray dispersive spectroscopy. The fatigue life of the coated stainless steel is considerably improved by ~3,200%. In addition, the application of the sputtered film yields an increase of the fatigue limit by 30%. The HRTEM analyses are further used to establish the relationship between fatigue property improvement and microstructure. Persistent slip bands in the steel, when arriving the surface, can create a surface offset, which becomes the potential fatigue-crack-initiation site. Our coating film can prevent the surface offset because of its good adhesion, high ductility and strength, thus yielding an improved fatigue property. Consequently, the fatigue-crack initiation and propagation behavior in the nanocrystalline-containing amorphous thin film could be understood.

3:00 PM Invited

Characterization of Shear Transformation Zones for Plastic Flow of Bulk Metallic Glasses: *Mingwei Chen*[1]; [1]Tohoku University

The basic units of plastic flow of BMGs, in a form of atomic clusters known as shear transformation zones (STZs), are the key to establish a fundamental model of deformation of BMGs at low temperatures. However, despite of extensive theoretical predictions and MD simulations, a direct experimental portrayal of STZ volumes in BMGs is still missing due to their small length scales and diminutive time scales. Here we report an experimental characterization of STZ sizes by proposing an experimental approach based on a newly-developed cooperative shearing theory. The measured STZ volumes of a variety of BMGs are fairly consistent with those predicted by MD simulations. This study offers compelling evidence that the plastic flow of BMGs occurs through cooperative shearing of unstable atomic clusters activated by shear stresses, and provides a new way to gain a quantitative insight into the atomic-scale mechanisms of BMG mechanical behavior.

3:15 PM Invited
Structure – Mechanical Property Relationship in Metallic Glasses: *Evan Ma*[1]; [1]Johns Hopkins University

For monolithic BMGs with an internal structure that is completely and invariably amorphous, a clear understanding of how the amorphous structures influence mechanical properties (e.g., strength and ductility) remains elusive. Here we investigate typical BMG-forming systems such as Zr-Cu, and Zr-Cu-Al, which are the basis of many important multi-component BMGs. The relationship between the local structure, dynamics and plastic flow is uncovered. We report the structural disordering processes responsible for the initiation of plastic flow at room temperature. Fertile and resistant sites for carrying shear transformations, as well as their effects on shear banding, have been identified. The composition-dependent local order is monitored in a quantitative manner, in lieUniversity of qualitative arguments from the standpoint of free volume. This structural perspective offers a new explanation to the observation of BMGs with large plasticity reported before.

3:30 PM Break

3:40 PM
Structural Characterization of a Bulk Metallic Glass under a Tensile Stress via In-Situ High Energy X-Ray Diffraction: *ChihPin Chuang*[1]; W. Dmowski[1]; Peter K. Liaw[1]; J. H. Huang[2]; G. P. Yu[2]; [1]University of Tennessee, Department of Materials Science and Engineering; [2]National Tsing-Hua University

In the present work, the microstructural response of a Zr-based metallic glass (BMG) (Zr52.5Cu17.9Ni14.6Al10Ti5) to a uniaxial tension stress was investigated using the in-situ high-energy x-ray scattering technique. The atomic-scale elastic strain was recorded accurately by the high-energy synchrotron radiation through different stress levels up to 80% of the yield stress at room temperature. The strains extracted directly from the normalized scattering spectrum and from pair-correlation functions were carefully compared. The corresponding mechanical parameters, such as Young's modulus and Poisson's ratio, were calculated from the strain tensor and were in good agreement with the results deduced from macroscopic measurements.

3:50 PM Invited
Studies of Shear Band Propagation in the Absence of Macroscopic Stress Concentrations During Compression of a Bulk Metallic Glass: *Wendelin Wright*[1]; Marcus Samale[1]; Mary LeBlanc[2]; Jeffrey Florando[2]; [1]Santa Clara University [2]Lawrence Livermore National Laboratory

Quasistatic uniaxial compression was performed on dog-bone shaped bulk metallic glass samples in order to minimize stress concentrations at the sample ends and constrain shear band propagation to the gage section. Spatially and temporally resolved measurements of strain were made using strain gages. Digital image correlation was used to map the full field displacements in three dimensions. The image correlation technique has a strain resolution of 100 microstrain and a spatial resolution of approximately 100 microns. Combined with scanning electron microscopy of deformed specimens, the results reveal the strain state prior to shear band formation and propagation and provide insight into the role of stress concentrations and sample bending on the mechanical behavior. The implications of this data for shear band initiation and propagation in bulk metallic glasses will be discussed.

4:05 PM
Short-Range Order of Cu100-xZrxAl5 Bulk Metallic Glasses: *Norbert Mattern*[1]; Hermann Franz[2]; Juergen Eckert[1]; [1]Leibniz Institute IFW Dresden; [2]DESY Hamburg

The short-range order of rapidly quenched and copper mold cast Cu100-xZrxAl5 glasses was investigated by means of Synchrotron high energy X-ray diffraction and Extended X-ray Absorption Fine Structure measurements. The total atomic pair correlation function were determined as a function of Cu content (x= 30-65 at%). Atomic structure models were developed by the reverse Monte Carlo method. The influence of Al on the atomic structure will be discussed in comparison with corresponding data of binary Cu-Zr glasses. The the composition dependence of the mechanical properties of the Cu100-xZrxAl5 glasses were analyzed and compared with their structural behavior.

4:15 PM Invited
Sample Size Dependent Mechanical Behaviour of BMGs: *Yi Li*[1]; [1]National University of Singapore

In general, a smaller sample size with a corresponding higher cooling rate will induce more free volume and a larger degree of structural disordering in the as-quenched amorphous alloys. As a consequence, smaller samples should have lower strength and a higher malleability. On the other hand, as a brittle material, statistically, the strength of BMGs will decrease when the sample size increases. In this work, with low temperature annealing to minimize the free volume differences among different sized BMG samples, we discovered a sample size-dependent "malleable-to-brittle" transition in a Zr-based BMG. We attribute this transition mainly to the geometrical size effect rather than the structural effect. Accompanied with this transition, the strength of BMG also exhibited a sample size dependence, which was discussed by both the flaw sensitivity3 and free volume viewpoints. Our results point out the needs to identify the critical sample size for the "malleable-to-brittle" transition in BMGs, especially under engineering consideration.

4:30 PM Invited
Indentation Deformation of a Zr50Cu37Al10Pd3 Bulk Metallic Glass: Effect of the Shear Banding Zone: *Fuqian Yang*[1]; Hongmei Dang[1]; Gongyao Wang[2]; Peter Liaw[2]; Yoshihiko Yokoyama[3]; Akihisa Inoue[3]; [1]University of Kentucky; [2]University of Tennessee; [3]Tohoku University

The dependence of plastic deformation of bulk metallic glasses on the deformation history has not been well studied. In this contribution, a fatigue test was first performed on a Zr50Cu37Al10Pd3 bulk metallic glass, which created a shear banding zone near the crack surface in the direction parallel to the crack propagation. Nanoindentation then was used to characterize the indentation deformation of the Zr50Cu37Al10Pd3 BMG in both the shear banding zone and the area far away from the shear banding zone. The material in the shear banding zone had different indentation hardness from that away from the shear banding zone, suggesting that the plastic deformation of bulk metallic glasses depends on the deformation history. The plastic energy dissipated in an indentation cycle was calculated as a function of the indentation load. Implications of this study in characterizing mechanical behavior of metallic glasses are discussed.

4:45 PM
Microscale Measurement of Residual Stress by the Slit Method in Zr-Based Bulk Metallic Glass (Zr50Cu40Al10) Subjected to Severe-Plastic Deformation: *Bartlomiej Winiarski*[1]; R.M. Langford[1]; Jiawan Tian[2]; Yoshihiko Yokoyama[3]; Philip Withers[4]; Peter Liaw[2]; [1]University of Manchester; [2]University of Tennessee; [3]Tohoku University; [4] University of Manchester

A surface-treatment process, which was used to generate severe-plastic deformation in the near-surface layer in crystalline materials, is applied on the Zr50Cu40Al10 bulk metallic glasses (BMGs). The slit method is used to determine the local residual stress in the plastically-deformed BMG component. The method is based on the measurement of the displacements field arose when a slit is milled into the material under investigation. The displacement field is determined by digital image correlation (DIC) analysis of scanning electron microscope (SEM) images. The slit is milled using focused ion beam (FIB-SEM) workstation. The surface under investigation is decorate with Yttria-stabilized zirconium (YSZ) equiaxial particles of size 20-30 nm precipitated from ethanol suspension. It is found that the average compressive residual stress is equal to -300±100 MPa. Microstructures and the micro-hardness profile of the deformed samples are also discussed.

4:55 PM Invited
Stability, Mobility and Sinks of Point Defects in Bulk Metallic Glasses: *Yuri Petrusenko*[1]; Alexander Bakai[1]; Ivan Neklyudov[1]; Igor Mikhailovskij[1]; Peter Liaw[2]; Lu Huang[2]; Tao Zhang[3]; [1]National Science Center - Kharkov Institute of Physics and Technology; [2]Department of Materials Science and Engineering, The University of Tennessee; [3]Department of Materials Science and Engineering, Beijing University of Aeronautics and Astronautics

Point defects, dislocations and interfaces determine the mechanical properties, transport phenomena, and radiation resistance of solids. Due to the existence of ideal crystalline structures, the defects of crystals can be properly determined. The attempts to determine an ideal glass structure make no real sense because glasses are non-equilibrium, non-ergodic and unstable. Field-emission microscopy was used to investigate compositional and structural heterogeneities of Zr-based bulk metallic glasses (BMGs). It is revealed that the BMG consists of nanoclusters of ~10 nm size. Stable intercluster boundaries are identified as well. Electron irradiation of BMGs of different composites at low temperatures and electrical resistance-recovery experiments allow us to identify the existence of stable and mobile defects, such as vacancies. The temperature range of the defect mobility is found. Ordering and disordering processes under electron irradiation are observed. Evidently, the intercluster boundaries are strongest sinks of point defects in BMGs.

5:10 PM
Size Effect on the Mechanical Behavior of Amorphous Alloys: *F. X. Liu*[1]; Y. F. Gao[1]; W. R. Chiang[2]; P. K. Liaw[1]; [1]The University of Tennessee; [2]Metal Industries Research & Development Center

At low temperatures and high strain rates, the plastic deformation of amorphous alloys usually occurs in narrow shear bands. This highly-localized inhomogeneous deformation results in premature fracture and poor ductility under unconfined conditions. When the size of the amorphous alloys ranges from millimeter to micrometer, the introduction of exterior and interior constraints will effectively confines the excessive propagation of individual shear bands and promotes multiple shear-band formation, resulting in enhanced plasticity. More recently, it has been reported that the amorphous alloys could undergo homogeneous deformation without shear-band formation when the deformation volume or the sample size is down to the nanometer scale. This obvious size effect on the mechanical behavior of amorphous alloys will be summarized and reviewed in this paper. The underlying deformation mechanism will be discussed and validated with micro-compression studies. The application potentials of amorphous alloys implicating from the size effect will be pointed out.

5:20 PM
Crystallization Behavior of a Zr-Based Bulk Metallic Glass during Rapid Heating and Cooling: *Hongqing Sun*[1]; Katharine Flores[1]; [1]Ohio State University

Laser deposition is a useful technique to create metallic glasses and other non-equilibrium microstructures. In this work, $Zr_{58.5}Cu_{15.6}Ni_{12.8}Al_{10.3}Nb_{2.8}$ powder was deposited onto glassy substrates of the same nominal composition. Amorphous melt zones surrounded by crystalline heat-affected zones (HAZ) consisting of numerous crystal morphologies are observed via SEM and TEM and characterized as functions of the heat input. The thermal history of the HAZ was analyzed using a three-dimensional finite element model. Numerical simulation results indicate that crystallization occurs in regions where the peak temperature exceeds 900 K, while the heating and cooling rates are on the order of 10^3 K/s. This rapid heating appears to suppress nucleation, resulting in a critical crystallization temperature ~150 K higher than that observed during DSC experiments. The short heating time (~ 10^{-2} s) associated with the observed large crystal size (~10 μm) also suggests that crystallization in the HAZ is dominated by growth.

Cast Shop for Aluminum Production: Characterization and Furnace Operation

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Pierre Le Brun, Alcan CRV; Hussain Alali, Aluminium Bahrain

Tuesday PM — Room: 2005
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Shridas Ningileri, Secat Inc

2:00 PM Introductory Comments

2:05 PM
A Review of Inclusion Detection Methods in Molten Aluminium: *Steve Poynton*[1]; Milan Brandt[1]; John Grandfield[2]; [1]CAST CRC and Swinburne University; [2]Grandfield Technology and CAST CRC

Management of inclusions is an important part of quality control within the aluminium cast house. Inclusions including oxide films and particles, spinel, refractory particles and silica particles have a detrimental effect on many aluminium cast products. The ability to reliably detect inclusions in a timely fashion is an essential part of this process. There are a number of tools available for inclusion measurement based on different chemical, physical and electrical principles. This document reviews existing techniques for inclusion detection such as K-Mold, Podfa, Lais, Prefil, LiMCA and Ultrasound, and also examines new techniques which may have potential to offer improved inclusion detection.

2:25 PM
A New Methodology for Performance Evaluation of Melt Refinement Processes in the Aluminum Industry: *Bernd Prillhofer*[1]; Holm Böttcher[2]; Helmut Antrekowitsch[1]; [1]University of Leoben; [2]AMAG Casting GmbH

To produce high quality alloys with very low impurity contents, melt quality must be improved from one process step to another. Accordingly, casthouses must analyze and optimize their production processes from the beginning to the end. There are several methods available for process evaluation. Concerning to the strong fluctuating initial inclusion content, the testing methods are not suitable for a single process step improvement at any step in the production chain, within the commercial accomplishment. Only the PreFil®-Footprinter has the potential and the flexibility for inclusion measurement at any place of the process chain, but there is a leak of methodologies for a clear efficiency assessment of metal cleaning steps. This paper presents a new proceeding for quick efficiency evaluation of metal cleaning by using the PreFil®-Footprinter for instance on some refinement steps of the standard cast-house processing for the alloy AA 7075.

2:45 PM
A Multiphase Model to Describe the Behaviour of Inclusions in LiMCA Systems: *Xiaodong Wang*[1]; Mihaiela Isac[1]; Roderick Guthrie[1]; [1]McGill University

LiMCA (Liquid Metal Cleanliness Analyzer) is a technique for the in-situ detection of inclusions in liquid metals that is widely used in the aluminum industry. It relies on the Electric Sensing Zone (ESZ) principle. A multiphase flow numerical model is now proposed for describing the motion of inclusions passing through variously-shaped ESZ orifices. The predicted motions of these entrained second phase particles take into account the various forces acting on them, including standard drag forces, added mass, fluid acceleration, buoyancy, and, most significantly, electromagnetic forces. The implications of using parabolic, fluted, and cylindrical orifices are considered, for various metals. The influence of conditioning current operations, electric conductivities, plus the density and size of the inclusions, on their trajectories, is investigated. The numerical results are compared with recent industrial results.

3:05 PM
Hydrogen Measurement Practices in Liquid Aluminium at Low Hydrogen Levels: *Mark Badowski*[1]; Werner Droste[1]; [1]Hydro Aluminium Deutschland GmbH

Today's most common measurement systems for hydrogen content in aluminium melt use the principal of a carrier gas and heat flux sensitivity (thermal conductivity cell). Current publications indicate, that the ambient humidity might influence hydrogen readings due to diffusion of water into the measurement loop. New systems – using electrochemical sensors with a solid reference material of known hydrogen content– enter the market. A comparison of these newer and older ways to determine hydrogen levels in liquid aluminium in a low and normal hydrogen regime will be presented.

3:25 PM
Accurate Measurement of Hydrogen in Molten Aluminium Using Current Reversal Mode: *Matt Hills*[1]; Chris Thompson[1]; Mark Henson[1]; Andy Moores[2]; Carsten Schwandt[3]; Vasant Kumar[3]; [1]EMC Limited; [2]Foseco; [3]University of Cambridge

Indium doped calcium zirconate is a high temperature perovskite proton conductor suitable for application as the solid electrolyte in a hydrogen concentration cell in molten aluminium. However, prolonged exposure to the melt can result in the prevalence of oxide ion conduction leading to loss of accuracy. Current Reversal Mode (CRM) is an amperometric technique which involves measuring currents through an electrochemical cell at two different states of polarisation. It is capable of accurately determining the cell voltage of a solid electrolyte sensor, as well as providing diagnostic information in the form of sensor resistance. In this study, CRM is applied to the ALSPEK H sensor for the measurement of hydrogen in molten aluminium. The CRM parameters of frequency and bias voltage for making accurate measurements are identified. Results are presented that demonstrate the application of CRM as a reliable diagnostic tool to ensure that the sensor is measuring accurately.

3:45 PM Break

4:05 PM
Increasing the Surface Emissivity of Aluminum Shapes to Improve Radiant Heat Transfer: *Richard Chandler*[1]; *P. Shull*[2]; [1]Pyrotek Inc; [2]Transmet Corporation

It is common in the aluminum industry to utilize radiant heat during all or part of a melt cycle to transform solid aluminum shapes such as ingots, sows and T-bars to the molten state. The rate of heat transferred into or out of an object by thermal radiation will be governed by, among other things, the surface emissivity of the object, which is equal to the absorption coefficient. Aluminum has a very low absorption coefficient, resulting in inefficient radiant heat transfer during melting. However, it is possible to easily and significantly improve the absorption coefficient of aluminum shapes, thereby increasing melt rates, reducing energy consumption, and reducing oxidation. This paper summarizes the heat transfer theory involved in this process, describes how the absorption coefficient of aluminum shapes can be increased significantly, and presents laboratory and field trials which demonstrate the effects of increased surface emissivity on the melting process.

4:25 PM
Waste Heat Recovery in an Aluminium Cast House: *Tom Schmidt*[1]; Jan Migchielsen[1]; [1]Thermcon Ovens B.V.

The current fuel price development gives new impulses to further development of waste heat recovery in industries like aluminium cast houses. Fuel is becoming the major cost in melting and casting aluminium. The recovery of waste heat in this industry becomes more and more interesting. This paper will address the various sources of waste heat and the potentials of re-using these sources in and around the aluminium cast house. Eventually an economic evaluation is given for predicting coming developments in the field of heat recovery.

4:45 PM
Reducing Metal Loss in Side Well Charged Melters with Invisiflame Burner Technology: *James Feese*[1]; Felix Lisin[1]; [1]Hauck Manufacturing

An aluminum side well melting furnace was equipped with state of the art Ultra Low NOx (Nitrogen Oxide) burners resulting in not only reduced emissions but extremely low level of dross formation. Detailed studies of the furnace were carried out to quantify melt efficiency, dross formation and losses, and NOx emissions. Experimental data clearly demonstrates dross losses from the main melt chamber to be very low and additional Fluent Computational Fluid Dynamic (CFD) theoretical modeling of the melt chamber complete with burners further validates the experimental findings. The ultra low NOx burner technology applied results in very low levels of oxygen on the bath surface as well as low and uniform bath surface temperature distribution supressing dross formation and NOx emissions. Dross losses were quantified taking into account typical operating practices resulting in savings of several hundred thousand dollars per year.

5:05 PM
Structure and Features of Slag from the Aluminum and Aluminum Alloys Melting and the Consequences: *Sergey Novichkov*[1]; Anatoliy Zholnin[1]; [1]Mosoblprommontazh

The influence of the flux structure upon the consistence of hot slag was investigated on basis of the earlier published model of aluminum slag structure. The aim of the investigation was to receive an experimentally proved conclusion which ensued from the described model. A practical consequence of the realized investigation was the detection of additives minimizing the flux consumption required to achieve the pre-determined consistence of hot slag. Two possible ways for decreasing of flux consumption ensue from the suggested model: use of additives which decrease the surface tension of melted flux; use of fluoride additives which reduce to fragments the oxide scales as principal thickener of melted flux. The investigations were realized with slag from salt-free remelting of aluminum scrap. As basic flux the equipoised mixture of sodium chloride and potassium chloride was used. The possibility of the substantial decreasing of flux consumption was obtained.

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

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee, TMS/ASM: Composite Materials Committee
Program Organizers: Toru Okabe, University of Tokyo; Ann Hagni, Geoscience Consultant; Sergio Monteiro, UENF

Tuesday PM — Room: 3009
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Ann Hagni, Geoscience Consultant; Takashi Nagai, University of Tokyo

2:00 PM
Characterization Of Shear Deformation In Iron: *Ellen Cerreta*[1]; Amy Ross[1]; Mike Lopez[1]; George Gray[1]; John Bingert[1]; [1]Los Alamos National Laboratory

Through the utilization of a "tophat" shaped specimen, the influence of specimen geometry on forced shear testing has been examined. Seven different geometries have been quasi-statically loaded and sectioned for post mortem analysis. Traditional techniques for this analysis include: optical microscopy, scanning electron microscopy, electron back scattered diffraction, and transmission electron microscopy. However, even with such multi-scale investigations, quantifying the characteristics of shear deformation can be difficult. Here, we present a method that utilizes grain aspect ratio to determine the thickness of the shear affected zone and then utilize this method to quantitatively investigate the influence of geometry on shear deformation as well as provide data for model validation.

2:15 PM
Characterization Of The Phase Equilibria And Transformation Behavior Of TiNiPt High Temperature Shape Memory Alloys: *Grant Hudish*[1]; D. Diercks[2]; A. Garg[3]; R. Noebe[3]; Michael Kaufman[1]; O. Rios[4]; [1]Department of Metallurgical and Materials Engineering, Colorado School of Mines; [2]Department of Materials Science and Engineering, University of North Texas; [3]NASA Glen Research Center; [4]University of Flordia

One factor limiting more widespread use of conventional NiTi shape memory alloys (SMAs) is their near room-temperature transformation temperatures. Increasing the transformation temperature of these alloys would allow for their use in higher temperature applications in such industries as aerospace, automotive, and power-generation. While Pt is known to cause the highest increases in transformation temperatures for NiTi-based alloys, its effects on microstructure, phase equilibria, mechanical properties, work output etc. are unknown. We will report our results on several TiNiPt alloys with content ratios of Ti/(Ni+Pt)>1, Ti/(Ni+Pt)<1, and Ti/(Ni+Pt)=1 obtained using SEM, EBSD, and TEM techniques. We will show that several phases not present on published Ti-Ni and Ti-Pt phase diagrams are observed allowing us to establish portions of the Ti-Ni-Pt phase diagram. DSC and resistivity methods were also employed to confirm the assumed phase equilibria. Finally, the effects and phases of interstitial elements, namely C and O, were also investigated.

2:30 PM
Object Based Quantitative Analysis Of Complex Microstructures In Steel: *Martin Fischer*[1]; Florian Gerdemann[1]; Wolfgang Bleck[1]; [1]RWTH Aachen University

A new method for the quantitative image analysis of complex microstructures in steel is presented. Microstructures in modern steel grades show growing complexity and ever smaller constituents. It is therefore increasingly difficult or even impossible to access reliable quantitative information on ratios, shapes and spatial distributions of individual phases and constituents via traditional metallographic methods. The new analysis procedure works object-based. Its basic processing units are so-called "image objects" which reflect the pictured structures by merged groups of likely related pixels. Thus information on shape, surface texture and spatial distribution of the microstructural constituents becomes processible. With these data available, the method is able to quantitatively analyse complex microstructures. This is demonstrated by its application to SEM images of different multiphase steel grades. As the analysis routine is fully automated, the analysis of large amounts of image data is possible and thereby the investigation of statistically relevant areas of microstructures.

2:45 PM
Grain Boundary Engineering in Alloy 800H: *Daniel Drabble*[1]; Milo Kral[1]; [1]University of Canterbury

Grain boundary engineering has recently shown promise in its ability to improve the mechanical properties of metals by modification of the grain boundary network. The aim of this work was to investigate the feasibility of using grain boundary engineering to improve the high-temperature properties (especially creep) of a common superalloy, alloy 800H/HT. Samples of 800H plate were thermo-mechanically processed using a range of conditions and subsequently analysed using Electron Backscatter Diffraction (EBSD). From this analysis, results such as grain size and grain boundary character were obtained. Grain boundary connectivity was also compared using a novel technique involving grain boundary pixel maps.The microstructural analysis has then been correlated with secondary creep rate, measured in uniaxial tension at high temperate, low-stress conditions to replicate common service parameters.

3:00 PM
Microstructural and Mechanical Characterization of Copper Microsamples after Cold Drawing: *Christopher Cheng*[1]; Marc Zupan[1]; S. Banovic[2]; [1]University of Maryland Baltimore County; [2]National Institute of Standards and Technology

Copper jackets used in ballistic applications are manufactured via a multi-step drawing process. Cold drawing process causes non-uniform deformation along the longitudinal direction of the jacket, resulting in microstructural and mechanical property anisotropy. This may lead to variation in the performance of the jacket during flight, therefore a need to quantitatively understand the types and scale of deviation in the material. Optical microscopy and X-ray diffraction techniques, in concert with nanoindentation and novel micro-tensile testing, were used to examine the structure-properties relationship of the deformed material. Metallographical analysis revealed high levels of cold working at all locations of the jacket. Deformation level was non-uniform and increased from the jacket tip to tail. This was evidenced by the changes in Young's modulus, yield strength, and ultimate tensile strength, as well as the evolving crystallographic texture, as a function of location. The drawing-crystallographic texture-mechanical properties relationship will be discussed in detail.

3:15 PM
Plastic Localization Phenomena In A Mn-Alloyed Austenitic Steel: Giorgio Scavino[1]; Fabio D'aiuto[2]; Paolo Matteis[1]; Pasquale Russo Spena[1]; *Donato Firrao*[1]; [1]Politecnico Di Torino; [2]Fiat Group Automobiles s.p.a.

A 0.5 wt.% C, 22 wt.% Mn austenitic steel, recently proposed for fabricating automotive body structures by cold sheet forming, due to favorable overall strength and ductility, exhibits plastic localizations during uniaxial tensile tests, but not during biaxial Erichsen tests. Full-thickness tensile and Erichsen specimens, cut from as-produced sheets, were polished and tested at different strain rates. During the tensile tests, the plastic localization phenomena consist first of macroscopic deformation bands traveling along the tensile axis, and then of series of stationary deformation bands, each adjacent to the preceding ones; both types of bands involve the full specimen width and yield macroscopically observable surface relief. No comparable surface relief was observed during the standard Erichsen tests.The stress state being known to influence plastic localization phenomena, reduced-width Erichsen tests were performed with 10 to 50 mm width polished specimens, in order to explore the transition from biaxial to uniaxial loading.

3:30 PM Break

3:50 PM
Characterization Laser Shock Peened IN718 Superalloy: *Amrinder Singh Gill*[1]; S.R Mannava[1]; Vijay Vasudevan[1]; [1]University of Cincinnati

LSP enhances service lifetimes of critical metal parts like aircraft engine fans and compressor blades.LSP dramatically improves fatigue strength, life and crack propagation resistance with shock wave-induced generation of deep compressive residual stress and microstructural changes. This study aims to understand effects of LSP parameters on residual stress distributions and microstructural changes in an important aero-engine material, IN718. Coupons of alloy with and without sacrificial layer were LSP-treated with varying energy densities using the GENIV system at GE Aircraft Engines. Depth-resolved characterization of macro residual strains and stresses and degree of cold work in peening direction and transverse to it was achieved using high-energy synchrotron x-ray diffraction at the Advanced Photon Source. Property changes were also studied using EBSD in SEM and TEM. Local property changes were examined using micro and nano-indentation measurements. Results show dominant effects of sacrificial layer and energy density on residual stress distributions and microstructure.

4:05 PM
Effects of Long Term Aging on Creep Properties of HP Reformer Tubes: *Karl Buchanan*[1]; Milo Kral[1]; [1]Canterbury University

The centrifugally cast HP series has become the dominant reformer tube material for the petrochemical industry. HP alloys with small additions of Nb and Ti are reported to have superior creep properties over standard HP alloys in accelerated testing. However, tubes removed after 3-5 years service exhibit lower remaining life than expected. The present work studies the effects of long-term laboratory aging, as well as exposure to laboratory simulated service conditions, on the creep performance of these alloys. HP-Nb and HP-Micro alloys were aged at 900-1100°C for 500-10,000 hours. Aged samples were then creep tested. Both post creep and post aging samples were characterized in detail using high resolution SEM and high resolution TEM, with special attention paid to the size and distribution to intragranular carbides and the intergranular carbide networks.

4:20 PM
Microstructure and Residual Stress Distribution in Laser Shock Peening Processed Ti-6Al-4V Alloy: *Yixiang Zhao*[1]; Seetha Mannava[1]; Ulrich Lienert[2]; Jon Almer[2]; Yang Ren[2]; David Lahrman[3]; Vijay Vasudevan[1]; [1]University of Cincinnati; [2]Argonne National Laboratory; [3]LSP Technologies

Laser shock peening (LSP) is a novel surface process that generates deep compressive residual stresses and microstructural changes and thereby dramatically improves fatigue strength of critical metal aircraft engine parts. The present study was undertaken to develop a basic understanding of the effects of LSP parameters on the residual stress distributions and microstructural changes in Ti-6Al-4V. Coupons of the alloy with and without a sacrificial/ablative layer were LSP-treated at GE Infrastructure Aviation and LSP Technologies. Depth-resolved characterization of the macro residual strains and stresses and was achieved using high-energy synchrotron x-ray diffraction and conventional x-ray diffraction. The near-surface and through-the-depth changes in strain, texture and microstructure were studied using EBSD/OIM and by TEM of thin foils fabricated from specific locations. Local property changes were examined using microhardness and nanoindentation measurements. The results showing the relationship between LSP processing parameters, microstructure, residual stress distributions and hardness are presented and discussed.

4:35 PM
Interfacial Microstructure and Evolution of Magnetic Pulse Welded AA6061-T6 and Cu101 Plates: *Yuan Zhang*[1]; Suresh Babu[1]; Glenn Daehn[1]; Michael Miller[2]; Kaye Russell[2]; [1]The Ohio State University; [2]Oak Ridge National Laboratory

Magnetic Pulse Welding was applied to lap joining AA6061-T6 and Cu101 plates. The microsecond-duration process introduced high-strain rate deformation into the plates. Tensile tests indicated that failure occurred in the base metal. Scanning electron microscopy indicated that the interface had a wavy morphology with discontinuous pockets of an intermetallic phase. Transmission electron microscopy revealed elongated subgrains, lamellar microbands, micro-twining, dislocation cells, submicron and nano-crystal grains, and articulation teeth-like grains. The high dislocation density and large misorientation of grain boundaries accommodated the deformation. Atom probe tomography suggested that a rod-shaped eutectic phase formed on the intermetallic regions. These results suggest that the refined grain structure with large misorientation and eutectic phase are the origin of high bonding strength of the weld.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.

4:50 PM
Development Of New Ultra-High Strength Nb-Containing Q-P-T Steel: *Xiaodong Wang*[1]; Ning Zhong[1]; Yonghua Rong[1]; Zuyao Xu[1]; [1]Shanghai Jiao Tong University

A new heat treatment process was developed and named quenching – partitioning – tempering (Q-P-T) based on the previous quenching & partitioning (Q&P) process which is proposed by J.G. Speer et al. And an ultra-high strength steel containing the combination of Nb and less than 0.2%C was obtained using Q-P-T process. The results show that the above Q-P-T steel possesses excellent mechanical properties: the tensile strength of over 1400MPa and the elongation of over 10%. In addition, the volume fractions of martensite and retained austenite were determined and the microstructures were characterized to discover the correlation between microstructures and mechanical properties in the Q-P-T steel.

5:05 PM
Toughness Characterization of Recycled Polyethylene Composites Reinforced with Post-Used Jute Fabric: *Sergio Monteiro*[1]; Amanda Lima[1]; Luis Augusto Terrones[1]; Leandro Marques[1]; [1]State University of the Northern Rio de Janeiro - UENF

Jute fabric obtained from discarded sackcloth is a promising low cost and post-used material being considered as reinforcement of polymeric matrix composites. In particular, the combination of this fabric with a recycled plastic constitutes an environmentally correct composite with both economical and environmental advantages. The present work investigates the toughness of recycled polyethylene composites reinforced with up to 40 wt% of post-used jute fabric extracted from discarded sackcloth. Standard Izod notched specimens were impact tested and the results showed an increase in toughness with the amount of jute fabric. Observation of the composite fracture by SEM revealed that the fabric weave, in spite of damages caused by the previous use of the sackcloth, act as an effective obstacle to crack propagation.

5:20 PM
Characterization of the Toughness of Piassava Fiber Reinforced Epoxy Matrix by Izod Impact Test: *Sergio Monteiro*[1]; Denise Cristina Nascimento[1]; Ludy Motta[1]; [1]State University of the Northern Rio de Janeiro - UENF

Natural lignocellulosic fibers are increasingly being used as polymeric matrix composite reinforcement owing to economical and environmental advantages as renewable and biodegradable materials. Among these, the piassava fiber extracted from a palm tree native of South America stands as one of the most rigid with a potential to be used as composite reinforcement. Therefore, the present work investigates the notch toughness behavior, by Izod impact tests, of epoxy composites reinforced with up to 40 % in volume of continuous and aligned piassava fibers. It was found that the incorporation of piassava fibers results in significant increase in the impact energy of the composite. Scanning electron microscopy analysis showed that the nature of the piassava fiber interface with the epoxy matrix is the major responsible for the superior toughness of the composite.

5:35 PM
Pullout Tests of Curaua Fibers in Epoxy Matrix for Evaluation of Interfacial Strength: *Sergio Monteiro*[1]; Ailton Ferreira[1]; Felipe Lopes[1]; [1]State University of the Northern Rio de Janeiro - UENF

The interface between the matrix and the reinforcing fiber plays an important role in the efficiency by which an applied load is transmitted through the composite structure. The shear stress at the fiber/matrix interface can be associated with this load transference and,consequently,affects the composite strength. In the present work, pullout tests were used to evaluate the interfacial shear stress of curaua fiber in epoxy matrix composites. A small critical length was found for the curaua fiber embedded in epoxy, which corresponds to a relatively weak fiber/matrix bond and lower interfacial strength.

Computational Thermodynamics and Kinetics: Defects

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, ASM Materials Science Critical Technology Sector, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Long Qing Chen, Pennsylvania State University; Yunzhi Wang, Ohio State University; Pascal Bellon, University of Illinois at Urbana-Champaign; Yongmei Jin, Texas A&M University

Tuesday PM	Room: 3002
February 17, 2009	Location: Moscone West Convention Center

Session Chair: Yunzhi Wang, Ohio State University

2:00 PM Introductory Comments

2:05 PM Invited
Coupling Crystalline Defects in Microstructure Modeling: *Chen Shen*[1]; Ju Li[2]; Yunzhi Wang[3]; [1]GE Global Research; [2]University of Pennsylvania; [3]Ohio State University

Microstructure formation in solids typically involves mutual interactions among different types of crystalline defects, such as precipitates, grain boundaries, and dislocations. These interactions can include both chemical (e.g., through diffusion) and mechanical (e.g., through long-range elastic strain field) interactions. A capability of treating them in a common computational framework helps understanding their synergetics in the microstructure formation. We will discuss this attempt in the framework of phase field method, where various defects are treated with different types of phase fields that are coupled in energetics and kinetics. We will show that the marriage of phase field method with other computational techniques, such as ab initio energetic calculations and nudged elastic band method, can expand the capability of the framework to new territories, with examples given on precipitation at defects, dislocation network formation, and interactions between dislocations and precipitate microstructure.

2:35 PM
Coupling Phase Field Models with Continuum Crystal Plasticity: Anaïs Gaubert[1]; *Yann Le Bouar*[2]; Alphonse Finel[3]; [1]DMSM, ONERA; [2]LEM, CNRS; [3]LEM, ONERA

The aim of this study is to propose a phase field model to describe microstructural evolutions in nickel-base superalloys during a creep loading. The misfit stresses, the elastic anisotropy, as well as the inhomogeneity in elastic constants between matrix and precipitates are taken into account. Plasticity in the gamma phase is introduced by the means of a viscoplastic law coupled with the phase field model. More precisely, a continuum crystal plasticity formulation is used to efficiently take into account the mechanical behavior of the gamma phase as well as its anisotropy arising from the underlying dislocation slip systems. Our model has been applied to the microstructural evolution in monocrystalline AM1 superalloys. Several experimental tests have been performed to fully determine the coefficients of the viscoplastic law. Finally, we will show that our model is able to describe in detail the rafting of the microstructure during a creep loading.

2:55 PM
Dislocation and Grain Boundary Melting: A Phase Field Crystal Study: *Joel Berry*[1]; K. R. Elder[2]; Martin Grant[1]; [1]McGill University; [2]Oakland University

Dislocation and grain boundary melting are studied in three dimensions using the phase field crystal method. Isolated dislocations are found to melt radially outward from their core as the localized excess elastic energy drives a power law divergence in the melt radius. Dislocations within low angle and intermediate angle grain boundaries melt similarly until an angle-dependent first order wetting transition occurs when neighboring melted regions coalesce. High angle boundaries are treated within a screening approximation, and issues related to ensembles, metastability, and grain size are discussed.

3:15 PM
Grain Boundary Roughening Temperature for Ni is Predicted to Range from below 800K to above 1400K: *David Olmsted*[1]; Stephen Foiles[1]; Elizabeth Holm[1]; [1]Sandia National Laboratories

Two dimensional interfaces change from being smooth at low temperatures because of energy considerations to being rough at high temperature because of entropic effects. While this is true of grain boundaries, not much attention has been paid to the fact, except for faceted boundaries. We used molecular dynamics to study grain boundary mobility in fcc Ni as a function of temperature and driving force for 388 crystallographically distinct boundaries. In many of these boundaries a roughening transition can be observed, with the roughening temperature varying from below 800K to above 1400K, i.e. from 0.51 to 0.89 of the melting temperature for the EAM potential used. The grain boundary mobility was found to be much larger above the roughening temperature than below. Thus we expect roughening to affect microstructural evolution substantially during metallurgical processing.

3:35 PM
Influence of Stress Evolution on Nanotwin Formation in Copper by First Principles Calculations: *Di Xu*[1]; Vinay Sriram[1]; Vidvuds Ozolins[1]; Jenn-Ming Yang[1]; K. N. Tu[1]; Gery Stafford[2]; Carlos Beauchamp[2]; [1]University Of California, Los Angeles; [2]National Institute of Standards and Technology

High density nanotwins in Cu have been shown to improve the mechanical strength and maintain good ductility and electrical conductivity. The formation of nanotwin is believed to relate to stress evolution during deposition and has been studied using first principles calculations of the total crystal binding energy. Under biaxial stress, the total energy of strained Cu can be larger than that of strain-relaxed periodic nano-twinned Cu. We propose that, during the deposition, highly strained Cu can undergo recrystallization and grain growth to relax stress and form strain-relaxed nanotwins. In-situ stress measurements were performed and showed periodic change of stress and stress relaxation in high frequency pulse electrodeposition. First principles calculations were used to predict nanotwin spacing that can be formed with the measured stress followed by a complete stress relaxation. The calculation results are in good agreement with experimental data.

3:55 PM Break

4:10 PM Invited
Palladium-Hydrogen Interaction in Dislocations: Trapping and Diffusion: *Dallas Trinkle*[1]; [1]University of Illinois, Urbana-Champaign

Pd has a high H 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.

4:40 PM
Atomistic Simulation of Diffusion along Dislocation Cores in Aluminum: *Ganga Purja Pun*[1]; Yuri Mishin[1]; [1]George Mason University

Kinetics of many materials processes are controlled by dislocation core diffusion. Its experimental measurements are very difficult and atomistic calculations are rare and nontrivial. We have performed molecular dynamics simulations of self-diffusion along screw and edge dislocations, both isolated and assembled in low-angle grain boundaries, in aluminum using an embedded-atom potential. While vacancy migration is confirmed to be the most important diffusion mechanism, the interesting and unexpected finding is that diffusion can occur even without pre-existing point defects in the core. This "intrinsic" diffusion mechanism is mediated by dynamic vacancy-interstitial pairs (Frenkel defects) that constantly form and recombine in the core due to thermal fluctuations. The Frenkel-pair formation can be assisted by the formation and motion of thermal jogs, making the intrinsic mechanism the dominant one in screw dislocations. The dislocation interactions in low-angle boundaries result in acceleration of dislocation diffusion.

5:00 PM
Phase Field Modelling of Stacking Fault Shear in Ni-Base Superalloys: *Vassili Vorontsov*[1]; Roman Voskoboynikov[1]; Catherine Rae[1]; [1]University of Cambridge

The "Phase-Field Microelasticity Theory" has been used to simulate dislocation propagation in nickel-base superalloys. In particular, the cutting of gamma-prime precipitates by matrix dislocations has been comprehensively studied with the aim of verifying the governing mechanisms of primary creep. Formation of nodes and networks has also been examined. The model demonstrates various dislocation reactions, such as recombination, reordering and annihilation. The formation and evolution of superlattice and complex stacking faults and anti-phase boundaries is achieved by incorporating fault energy data from Molecular Dynamics simulations. For the first time, a double-plane gamma-surface is used to incorporate extrinsic faults. The observed likelihood of stacking fault formation is found to exhibit strong stress dependence. The simulated stacking faults form less readily as the applied stress approaches the superalloy's theoretical yield. This demonstrates why numerous stacking faults are observed in primary creep specimens and not in those subjected to a simple tensile test.

5:20 PM
Interaction of He-Vacancy Clusters with Cr in FeCr Alloys from First Principles: *Enrique Martinez*[1]; Chu Chun Fu[1]; Frederic Soisson[1]; Maylise Nastar[1]; [1]CEA-Saclay

FeCr alloys are nowadays the strongest candidates as structural materials for fusion applications. As it is well known experimentally, irradiation modifies the response of such an alloy reducing considerably its working lifetime. During irradiation with fast neutrons He is produced by transmutation. How this He affects the properties of the base material is not well understood. We have studied the interaction of He, vacancy and small He-vacancy complexes of very high binding energy, and their possible diffusion paths as a function of Cr concentration and Cr-cluster spacing within the Density Functional Theory. The accuracy from various pseudo-potential and basis sets approximations are discussed in detail. Formation, binding and migration energies have been calculated systematically. The resulting energetics will be used in Monte Carlo simulations to try to understand how He bubbles nucleate in the FeCr matrix as well as how α' precipitates may be formed in the presence of He.

5:40 PM
Interfaces and the Behavior of Nanocomposites under Irradiation: *Michael Demkowicz*[1]; [1]Los Alamos National Laboratory

Nanocomposites—both hetero- and homophase—contain a high volume fraction of interfaces that can heal radiation damage by absorbing and recombining radiation-induced Frenkel pairs. But are all interfaces equally effective at reducing radiation damage? This question is addressed by a diffusion-reaction analysis that models interfaces as surfaces where point defect properties such as formation energies, migration energies, and vacancy-interstitial recombination rates differ from those of the surrounding crystalline medium. The dependence of an interface's response to irradiation with varying interface properties and irradiation conditions is investigated and opportunities for designing radiation-tolerant nancomposites for nuclear energy applications are discussed. We are grateful for support from the LANL LDRD and Director's Postdoctoral Fellowship programs as well as from DOE-OBES.

CO_2 Reduction Metallurgy 2009: Ferrous and Titanium Metallurgy

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

Tuesday PM — Room: 2012
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Jean-Pierre Birat, Arcelor Mittal; Malti Goel, Former Advisor and Senior Scientist, Ministry of Science and Technology

2:00 PM Introductory Comments

2:05 PM
Reduction of CO2 Emissions In Steel Industry Based on LCA Methodology: Ana-Maria Iosif[1]; Jean-Pierre Birat[1]; *Olivier Mirgaux*[2]; Denis Ablitzer[2]; [1]ArcelorMittal R&D; [2]LSG2M-Nancy

Integrating environmental considerations into the product traditional process design is now the major challenge for steel industry. Life Cycle Assessment (LCA) is nowadays considered as an appropriate method for assessing environmental impact and selecting new technologies to reduce CO2 emissions for steel industry. In this paper we propose a new methodological concept which combines LCA thinking with process simulation software in order to carry out the life cycle inventory of classical steelmaking process. Using Aspen PlusTM software, a physicochemical model has been developed for the integrated steelmaking route. This model gives the possibility to carry out life cycle inventories for different operational practices in order to optimise the use of energy, to calculate CO2 and other emissions and to control the mass and the heat balances of processes.It is also shown that such approach can be used to design and assess new technologies for steelmaking, without large industrial application.

2:25 PM
Electrolytic Reduction Of Ferric Oxide To Yield Iron And Oxygen: Antony Cox[1]; *Derek Fray*[1]; [1]University of Cambridge

Hematite pellets were electrolytically reduced to iron in molten sodium hydroxide at 530C to produce iron sponge, free of carbon and sulfur, with about 10 wt% oxygen. The cell was operated at 1.7V with a hematite cathode and an inert nickel anode. Control of the activity of sodium oxide in the melt allowed the cell to be operated below the decomposition potential of the electrolyte with the overall reaction being the ionisation of oxygen from the hematite, its subsequent transport to the anode and discharge, leaving iron at the cathode. A current density of 0.5 A/cm2 was attained and the energy consumption was 2.8 kWh/kg of iron.

2:45 PM
"Enhanced Energy Efficiency And Emission Reduction Through Oxy-Fuel Technology In The Metals Industry": *Norman Bell*[1]; [1]Jupiter Oxygen Corporation

Jupiter Oxygen Corporation has an over ten year history of utilizing Oxy-Fuel technology to process aluminum scrap with melt rates in the range of 700-950 BTGU/pound. Recent events have led to consideration of the Jupiter patented process in steel mill furnaces also. A walking beam billet furnace at a major steel company is currently under consideration for conversion to this process to reduce fuel costs and increase production. In addition the firing alumina kilns with Oxy-Fuel is being considered. Jupiter is testing the use of their Oxy-Fuel system for boilers in the power industry with very good results. The process reduces carbon emissions and virtually eliminates NOx from the flue gas stream leaving a much smaller volume of gas for sequestration or reuse.

3:05 PM
Suspension Ironmaking Technology with Greatly Reduced CO_2 Emission and Energy Requirement: Hong Yong Sohn[1]; *Moo Eob Choi*[1]; Yao Zhang[1]; Joshua Ramos[1]; [1]University of Utah

A new technology for ironmaking based on direct gaseous reduction of iron ore concentrate is under development. This technology would drastically lower CO_2 emission and reduce energy consumption by nearly 38% of the blast furnace requirements. Experiments were performed using iron oxide concentrate at 1150°C in a bench-scale facility, which was the highest temperature that could be reached in the facility. The reduction extent was determined at residence times of 3.5 - 5.5 seconds in which the reduction extent approached 43% with 0% excess H_2 and 95% with 860% excess H_2. Separate kinetics measurements showed that the rate is much faster at 1200 - 1400°C. Experiments were also carried out using syngas (mixtures of H_2 + CO). About 90% reduction in 3.5 seconds at 860% excess hydrogen contained in the syngas demonstrated sufficiently fast reduction for a suspension process and the feasibility of using syngas instead of pure hydrogen.

3:25 PM Break

3:40 PM
Investigation of Carbonic Anhydrase Assisted Carbon Dioxide Sequestration Using Steelmaking Slag: *Charles Rawlins*[1]; Simon Lekakh[2]; Kent Peaslee[2]; Von Richards[2]; [1]Montana Tech; [2]Missouri S&T

Batch aqueous leaching and carbonation tests were conducted using industrial steelmaking slags to determine the effect of carbonic anhydrase enzyme as a catalyst. Calcium leaching is a strong function of particle surface area, and the extent can be expressed as a function of time and particle size. Carbonic anhydrase did not affect the calcium-leaching rate, however, it did catalyze calcium carbonate formation to achieve a neutralization time near the theoretical rate. Additionally, carbonic anhydrase modified the precipitate morphology due to accelerated particle nucleation. Time controlled tests in which the pH dropped to ~6 decreased the amount of carbonate produced, and this effect was exaggerated by carbonic anhydrase, while pH controlled tests (>8.5) exhibited the highest rate of carbonation. Because the leaching rate was ~50% faster than the carbonation rate, a further increase in the amount of carbonation may be realized by using carbonic anhydrase however pH must be >10.3.

4:00 PM
Novel Alkali Roasting Of Titaniferous Minerals And Leaching For The Production Of Synthetic Rutile: *Animesh Jha*[1]; Abhishek Lahiri[1]; [1]University of Leeds

We present a novel route for the production of high purity synthetic rutile (>95 wt% TiO2) via a two-step chemical process. In the first step, the titaniferous minerals are roasted with alkali in air below 900C. After roasting in air the quenched reaction product is leached in water for the separation of water-soluble alkali phases from the insoluble alkali titanate. After aqueous leaching the alkali titanate is further leached in an organic acid medium in N2 atmosphere from which the synthetic rutile is derived. Alkali phase was recovered from the leach solution. The synthetic rutile derived from roasting and leaching was contained 95-97 wt% of TiO2 and 3-5 wt% oxide/hydroxide impurities of Fe/Na/Al/Ca. The paper will present the physical chemistry of phase separation process in detail including recycling of CO2. We also discuss the suitability of this process for potential mineral beneficiation with virtually zero-process waste.

4:20 PM
Accelerated Electro-Reduction of TiO2 to Metallic Ti in CaCl2 Bath Using Inert Anode: Xiaobing Yang[1]; Abhishek Lahiri[1]; *Animesh Jha*[1]; [1]University of Leeds

Discovery of the FFC Cambridge process for the electro-reduction of metal oxides to metals using carbon anode has been thought to be novel means to produce reactive metals of high purity. In this paper we will discuss the mechanistic aspects of electro-reduction of TiO2 in the presence of a CaCl2 bath and an inert anode of alloy material. A remarkable reduction in time from the reported 24-96 hours to less than 10 hours has been achieved by accelerated dissociation of perovskite phase in the presence of alkali modifiers in the TiO2 pellet. We also discuss the stability of anodes in chloride bath and means to enhance the longivity of such anode materials by monitoring reduction.The paper will discuss the thermodynamics and kinetics aspects of reactions and the steps that leads to rapid conversion of TiO2 to Ti metal.

4:40 PM
The Theoretical Calculation and Validation of Burden Trajectory in Blast Furnace of Bell-less Top: *Yu Yaowei*[1]; Bai Chenguang[1]; Zhang Zhengrong[1]; Wang Feng[1]; Lv Daguang[1]; [1]Chongqing University

Blast furnace ironmaking is a main method by which iron is efficiently reduced from iron-bearing materials with CO. Charging is one of primary systems for blast furnace control. The trajectory of materials is an important parameter in determining impact point where falling materials intersect with the stockline profile in the charging. In order to clarify the trajectory of materials and validate theoretical calculated trajectory, a 1/15 scale cold model of an actual 2500m3 shaft and bell-less top charging system has been built. The results indicate that the measured trajectory is consistent with the theoretical one.

Diffusion in Materials for Energy Technologies: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Nuclear Materials Committee, TMS: Solidification Committee, ASM-MSCTS: Atomic Transport Committee
Program Organizers: Jeffrey LaCombe, University of Nevada, Reno; Yongho Sohn, University of Central Florida; Carelyn Campbell, National Institute of Standards and Technology; Afina Lupulescu, GE; Ji-Cheng Zhao, Ohio State University

Tuesday PM Room: 3006
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Yongho Sohn, University of Central Florida; Maria Okuniewski, University of Illinois at Urbana-Champaign

2:00 PM Invited
Rapid Diffsuion-limited Pathways to CuInxGa1-xSe2 Thin Film Synthesis: *Timothy Anderson*[1]; Carelyn Campbell[2]; [1]University of Florida; [2]National Institute of Standards and Technology

A systematic search for rapid reaction pathways for the formation of CuInxGa1-xSe2 thin films was performed using in-situ high temperature X-ray diffraction. Time-resolved high temperature X-ray diffraction data were collected using a position sensitive detector while the precursor film temperature was held constant or ramped. Reaction pathways under both inert and Se overpressure were examined for a variety of elemental and bilayer precursor film structures. The observed pathways are compared to those suggested by diffusion limited transport with equilibrium conditions at the interfaces. Analysis of the diffraction data was supported by high resolution structural and compositional measurements of synthesized absorbers. The results indicate absorber synthesis is a robust process with different precursor structures and operating conditions leading to CuInxGa1-xSe2 formation, often through formation of intermediate compounds. Other results also suggest that MoSe2 forms after complete formation of the absorber when using Mo/Glass substrates.

2:35 PM
Diffusion and Reaction Kinetics in the Al_2O_3-TiO_2 System under Electric Field Application: *Dat Quach*[1]; Joanna Groza[1]; [1]University of California, Davis

Materials processing under external electric field / electrical current applications is of great interests due to its high heating rates and possibly enhanced mass transport. Dense Al_2TiO_5 ceramic is obtained from a powder mixture of Al_2O_3 and TiO_2 in a few minutes using the novel field-assisted sintering technique (FAST). Under field application the reaction kinetics and diffusion in this ceramic system are studied via powder reaction and diffusion couple experiments at temperatures from 1370—1500°C. Results from FAST show enhanced nucleation and a different activation energy for reaction compared with conventional heating.

3:00 PM
Silver Diffusion in Silicon Carbide: *Erich Friedland*[1]; Nic Van der Berg[1]; Johan Malherbe[1]; Thulani Hlatshwayo[1]; [1]University of Pretoria

This study aims to obtain information on volume and grain boundary diffusion as well as on the influence of radiation damage. For this purpose 360 keV 109-Ag was implanted with a fluence of 2xE16 cm-2 in poly and single crystalline SiC at temperatures ranging from room temperature to 900 K. Diffusion coefficients were obtained from implantation profile broadening after isochronal and isothermal annealing up to 1900 K, using RBS analysis combined with alpha-particle channeling spectrometry. Structural information was obtained by scanning and transmission electron microscopy. As the surface region of the room temperature implants was completely disordered, the initial broadening was used to study diffusion in amorphous silicon carbide. Comparison of results for annealed single and poly crystalline samples yielded information on the relative importance of volume and grain boundary diffusion. The influence of radiation damage was extracted by comparing results for implants done at room and elevated temperatures.

3:25 PM
Microstructure and Annealed Effect of Sn-Sb-(Ni) Thin Films on the Charge-Discharge Capacity Characteristics: *Wu Chao-Han*[1]; Fei-Yi Hung[2]; Lui Truan-Sheng[1]; Chen Li-Hui[1]; [1]Department of Materials Science and Engineering, Center for Micro/Nano Science and Technology, National Cheng Kung University; [2]Institute of Nanotechnology and Microsystems Engineering, Center for Micro/Nano Science and Technology, National Cheng Kung University

In this study, radio frequency magnetron sputtering was adopted to prepare Sn-Sb-(Ni) film anodes. The effects of the thickness of the film and its index of crystalline (IOC) on the charge-discharge capacity characteristics are discussed. Increasing the thickness of the film anode from 500nm to 2000nm, not only raised the IOC, but also improved the migration of lithium ions and electrons because of the lower resistivity. So, the cyclability of the as-adopted film was enhanced with increasing the film thickness. After recrystallization, the IOC rose and the resistivity fell. However, cracks on the film induced by thermal strain increased the area of the passive film, resulting in reduced cyclability. In addition, added micro-Ni into Sn-Sb matrix was able to enhance the charge-discharge capacities and the high temperature cyclability.

3:50 PM Break

4:05 PM Invited
High-Pressure Hydrogen Permeation and Diffusion in Iron and Steels: *Zhili Feng*[1]; Lawrence Anovitz[1]; Timothy Armstrong[1]; [1]Oak Ridge National Laboratory

Hydrogen induced mechanical property degradation is a primary concern for the safe operation of hydrogen delivery and storage systems made of metallic engineering materials such as ferritic steels. The degree of degradation is directly related to the amount of hydrogen in the metal. In this work, we investigated the hydrogen permeation and diffusion processes in pure iron and ferritic steels by means of high-pressure gaseous hydrogen permeation experiment (up to 2,000psi). The dependency of hydrogen diffusion on hydrogen charging pressure and temperature are obtained and will be discussed. In addition, the observed surface effects will be discussed in relation to the high-pressure hydrogen permeation test and their implications in controlling hydrogen into metal. Issues related to hydrogen transport under high-pressure hydrogen environment to ensure the long-term reliability of the hydrogen delivery infrastructure will be highlighted.

4:40 PM
Hydrogen Permeation in Steels *via* Fractional Diffusion: *Alonso Jaques*[1]; Jeffrey LaCombe[1]; [1]University of Nevada

Frequently, hydrogen diffusion is analyzed using electrochemical permeation per methods per ASTM G148-97, where the experimental setup (Devanathan-Stachurski) is established, but the analysis methods are still largely based on "traditional" diffusion theory. Ample experimental evidence published in the literature calls this analytical approach into question, as much of the reported data in the literature shows deviation from theoretical predictions. For example, discrepancies observed in hydrogen diffusion in steel show signs of so-called "anomalous diffusion", where the characteristic length scale for diffusion is not proportional to $t^{1/2}$, but rather to $t^{1/\alpha}$, where ($1 \leq \alpha \leq 2$). The formal description of anomalous diffusion is expressed using fractional calculus. Preliminary analysis of published "problematic" data from the literature shows improved agreement with fractional diffusion when compared to analyses using the traditional square-root scaling. The fractional calculus approach to diffusion will be discussed here, including implementation of the method and discussion of the phenomenological foundations.

5:05 PM
Magnetic and Electric Field Effects on Hydrogen Absorption and Mass Transfer at the Metal/Electrolyte Interface: *John Roubidoux*[1]; Brajendra Mishra[1]; Joshua Jackson[1]; David Olson[1]; [1]Colorado School of Mines

The superposition of a uniform magnetic field during laboratory-scale electrochemical hydrogen charging of pipeline steels (X52, X70, X80, X100) indicates an increase in the measured hydrogen content compared to unmagnetized charging. Increased hydrogen absorption may be associated with the disturbance of the Gouy-Chapman Layer (GCL) and the Helmholtz Double Layer (HDL).

The disturbance of the GCL and HDL may be due to the interaction of the large magnetic and electric fields, which results in an altering of the kinetics of the system. The rate of mass transfer to the working electrode (steel sample) is also know to increase when a magnetic field is superimposed on the experimental system. The objectives of this research were to determine the mechanism by which hydrogen absorption occurs at the metal/electrolyte interface and determine what influence combined magnetic and electric fields have on the rates of mass transfer to the working electrode.

5:30 PM
Aspen Plus Modeling of a Diffusion-Limited Three-Reaction Hydrogen Producing CuCl Thermochemical Cycle: Alexandra Lupulescu[1]; *John Prindle*[1]; Victor Law[1]; [1]Tulane University

As the world continues to grow at a steadfast pace, an even bigger strain is placed on already limited energy resources. Consequently, the fossil supply must be replaced by new methods of producing energy. A promising field is hydrogen. In the current work, the Copper-Chloride thermochemical cycle has been studied due to its low temperature requirements and easy implementation as a result of minimal solids transfer. A three-reaction scheme has been proposed: $2CuCl + 2HCl \cdot 2CuCl_2 + H_2$ (electrochemical) 100°C $2CuCl_2 + H_2O \cdot Cu_2OCl_2 + 2HCl$ (vacuum) 375°C $Cu_2OCl_2 \cdot 2CuCl + 0.5O_2$ 550°C For the first time, to our knowledge, the electrolyzer has been modeled in Aspen by a calculator block rather than a stoichiometric reactor. This was done in order to accurately depict the diffusion across the ionic membrane of the electrolyzer, which in turn determines the kinetic rate of the hydrogenproducing reaction.

Dislocations: 75 Years of Deformation Mechanisms: Nanostructured and Temperature Effects on Dislocations

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: David Bahr, Washington State University Erica Lilleodden, GKSS Research Center; Judy Schneider, Mississippi State University; Neville Moody, Sandia National Laboratories

Tuesday PM — Room: 3022
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

2:00 PM Invited
Dislocation Dynamics And Storage In Nanocrystalline Materials: *Scott Mao*[1]; Zhiwei Shan[1]; [1]University of Pittsburgh

This talk focuses on dislocation dynamics and storage in nc materials through in-situ TEM and in-situ synchrotron tests. It is believed that the dynamics of dislocation processes during the deformation of nanocrystalline materials can only be visualized by computational simulations. Here we demonstrate that observations of dislocation processes during the deformation of nanocrystalline Ni with grain sizes as small as 10nm can be achieved by using a combination of in situ tensile straining and high-resolution transmission electron microscopy. In collaboration with Dr.Yang in Argonne National Laboratory, 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. The stored dislocations are reversible after unloading. In course grained sample, stored dislocations are not reversible.

2:30 PM
Mechanical Behavior Of Trimetallic Nanocomposites Under Various Loading Conditions: *Ioannis Mastorakos*[1]; Hussein Zbib[1]; David Bahr[1]; Firas Akasheh[2]; [1]Washington State University; [2]Taskegee University

Nano-Metallic Material (NMM) composites represent a novel class of advanced engineering materials whose scientific significance and technological potential as high performance materials is just beginning to be explored. Presently, NMM composites are made of bimetallic systems and are typically classified into coherent (the two metals having the same crystal structure and a small lattice parameter mismatch) and incoherent systems (the two metals having different crystal structure and a large lattice parameter mismatch). While coherent systems are more ductile, incoherent systems are generally stronger.The purpose of this work is to expand our understanding on the behavior of NMM by performing atomistic simulations on trimetallic systems. The simulated composite material is consisted of alternating layers of Ni/Cu/Nb, thus creating a combination of coherent/incoherent interfaces. The deformation behavior as well as the damage mechanisms of the triaxial systems are investigated under uniaxial, biaxial and fatigue loading.

2:50 PM
Stress and Dislocation Core Controlled Plasticity of Graphene Based Nanostructures: *Shuo Chen*[1]; Elif Ertekin[2]; Daryl Chrzan[1]; [1]Department of Materials Science and Engineering, University of California, Berkeley; [2]Berkeley Nanoscience and Nanoengineering Institute, University of California, Berkeley

Graphene based nanostructures are expected to be plastically deformed under certain loading conditions mediated by defects best described as two-dimensional dislocations. Here we explore a novel mechanism of plasticity unique to these systems. Specifically, stress relaxation is studied via kinetic Monte Carlo simulations (both at T=0 K and finite temperatures) based on the empirical Tersoff-Brenner potential. In contrast to the usual glide response in bulk materials, the stress relaxation is initially achieved by generating an array of dislocation dipoles (Stone-Wales defects). At lower stresses, the plastic deformation mechanism switches to conventional dislocation glide. The kinetic pathway is further analyzed within linear elasticity theory. It is revealed that the nature of the plasticity is closely related to the core structure of the two-dimensional dislocations which, in turn, is strongly affected by the local curvature and the local stress and strain fields.

3:10 PM
Dislocation-Interface Interaction in Nanoscale Metallic Multilayers: *Sergey Medyanik*[1]; Shuai Shao[1]; [1]Washington State University

Nanoscale multilayered metallic materials often exhibit very high strength levels, close to the theoretical strength limits. This strengthening phenomenon has been usually attributed to the presence of interfaces between dissimilar materials that serve as barriers to the gliding dislocations. In this work, we present results of atomistic simulations that demonstrate some of the mechanisms of dislocation interaction with interfaces. We employ nanoindentation model to generate dislocations at and near the surface and focus on investigating bi- and tri- metallic systems composed of Cu, Ni, and Nb. Interaction of propagating dislocations with three types of interfaces (coherent, semi-coherent, and incoherent) is analyzed. Specific mechanisms that cause strengthening in nanoscale multilayered metallic composites are investigated in detail.

3:30 PM
Dislocation Dynamics (DD) Analysis of Strength in Heterogeneous Nanoscale Tri-Metallic Multilayered Composites: *Firas Akasheh*[1]; Hussein Zbib[2]; Cory Overman[2]; Sreekanth Akarapu[2]; David Bahr[2]; [1]Tuskegee University; [2]Washington State University

In this work, multiscale DD-continuum analysis of plasticity in heterogeneous multilayered structure made of 3 different metals is studied. Typical DD analysis does not account for Koheler image forces due to elastic properties mismatch. Such forces become increasingly significant in the case of NMM composites, affecting the strength and dislocation interaction among themselves and with the interfaces. A methodology based on the concept of eigenstrain and superposition was implemented and validated to account for such effects. The channeling strength of layer-confined glide dislocations in different FCC-BCC material systems is estimated and Koheler forces are quantified as a function of the layer thicknesses and layering scheme.

3:50 PM Break

4:10 PM Invited
The Importance of Reordering During the High Temperature Deformation of Ni-Base Superalloys: Libor Kovarik[1]; Raymond Unocic[1]; Yunzhi Wang[1]; Ju Li[2]; *Michael Mills*[1]; [1]Ohio State University [2]University of Pennsylvania

In order to improve the capabilities of polycrystalline gamma-prime ($L1_2$ structure) strengthened Ni-based superalloys for turbine disk applications, the rate-controlling deformation mechanisms must be fully understood, and robust theory/models developed that connect the microstructure to creep and fatigue properties. Dramatically different deformation mechanism can occur depending upon temperature, applied stress and initial precipitate structure in disk alloys. Most remarkably, in the range of 600-800°F extended faulting through precipitates and matrix, isolated shearing of gamma-prime precipitates and microtwinning are observed. While these mechanisms are distinct, we argue that they are all connected and controlled by the same thermally-activated process of chemical reordering in the ordered precipitates after shearing by Shockley partial dislocations. The evaluation of key activation processes, suggested from direct experimental observations of the deformation mechanisms, is being conducted using a novel combination of atomic-scale and phase field dislocation modeling.

4:40 PM
Introducing Dislocation Climb by Bulk Diffusion in Discrete Dislocation Dynamics Simulations: *Dan Mordehai*[1]; Emmanuel Clouet[2]; Marc Fivel[3]; Marc Verdier[3]; [1]SRMP, CEA-Saclay, (currently at Department of Materials Engineering, Technion-Israel Institute of Technology); [2]SRMP, CEA-Saclay, (currently at LMPGM, Universite Lille); [3]SIMaP, Grenoble INP

One of the computational tools to study dislocation microstructure and plasticity at the mesoscopic scale is Discreet Dislocation Dynamics (DDD) simulations, in which dislocations are treated as elastic entities. In this talk, we present a method to incorporate dislocation climb by bulk diffusion in Dislocation Dynamics simulations, by coupling this simulation technique with the diffusion theory of vacancies. We adapt the method to a 3-dimensional DDD simulation, in which dislocations are represented by pure edge and screw segments. The calculation is demonstrated by simulating the activation of a Bardeen-Herring climb source upon the application of an external stress or under vacancy supersaturation, as well as isolated dislocation prismatic loops shrinkage and expansion. The model is shown to reproduce the coarsening of dislocation loops in annealed bulk, where large dislocation loops expand on the expense of smaller ones. The processes observed in our simulations agree with experimental observations in fcc metals

5:00 PM
Characterization of Dislocations and Modeling of Creep Mechanisms in Zirconium Alloys: *Benjamin Morrow*[1]; Robert Kozar[2]; Ken Anderson[2]; Michael Mills[1]; [1]Ohio State University [2]Bechtel Bettis Inc

Zirconium alloys are used commonly for applications in nuclear reactors. Accurately predicting creep deformation of zirconium alloys throughout the lifecycle of a reactor depends on reliable deformation models. The Modified Jogged-Screw Model asserts that the motion of tall jogs in screw dislocations act as the rate controlling mechanism during creep in certain regimes. Previous studies have demonstrated the applicability of the Modified Jogged-Screw model to the thermal creep behavior of hcp metals. Scanning transmission electron microscopy (STEM) was used to directly observe and characterize the dislocation structure of creep tested Zircaloy-4 and quantify model parameters such as jog height, jog spacing, and dislocation density. Attempts to correlate dislocation density measurements using X-ray diffraction and STEM techniques will be reported. Thorough characterization will provide a better understanding of crept dislocations structures in zirconium alloys, which will ultimately result in more robust creep deformation predictions.

5:20 PM
Shock Induced Deformation Substructures in a Copper Bicrystal: *Fang Cao*[1]; Irene Beyerlein[1]; Bulent Sencer[2]; Ellen Cerreta[1]; George Gray[1]; [1]Los Alamos National Laboratory; [2]Idaho National Laboratory

Controlled shock recovery experiments have been conducted to assess the role of shock pressure and orientation dependence on the substructure evolution of a [100]/[01-1] copper bicrystal. Electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) were utilized to characterize orientation variation and substructure evolution of the post-shock specimens. Well defined dislocation cell structures were displayed in both grains and the average cell size decreases with increasing shock pressure. Twinning has been occasionally observed in the 5 GPa shocked [100] grain and becomes the dominant substructure at higher shock pressure. The stress and directional dependence of twinning in the bicrystal is analyzed in considering the energetically favorable dissociation of dislocations into Shockley partials and the stress-orientation effect on the partial width. Moreover, a critical 'tear apart' stress is proposed and the calculated value is in good agreement with the experimental observations.

5:40 PM
Static Recovery Of Pure Copper Near Room Temperature: *Chen-ming Kuo*[1]; Chih-Sheng Lin[1]; [1]I-Shou University

Static recovery experiments of pure copper near room temperature have been conducted via TEM, DSC, hardness and extensometer to explore the time and temperature dependent relationships. By using different strain rates, dislocation density is generated differently. The recovery phenomenon is more significantly as time and temperature increase. Activation energy at initial static recovery is 48 kJ/mol, which is the energy for dislocation annihilation by glide or cross-slip, and varies linearly with static recovered strain. Once dislocation annihilation processes are exhausted, more energy is required for subgrains to form and then grow. The recovered strain is slowed down and eventually is saturated.

Electrode Technology for Aluminum Production: Joint Reduction and Electrode Technology Session: Coping with Changes in Coke Quality

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Barry Sadler, Net Carbon Consulting Pty Ltd; John Johnson, RUSAL Engineering and Technological Center LLC

Tuesday PM — Room: 2003
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Barry Sadler, Net Carbon Consulting Pty Ltd

2:00 PM Introductory Comments

2:05 PM Invited
Calcined Coke Quality in 2009 and Beyond – Adapting for the Future: *Les Edwards*[1]; [1]Rain CII Carbon LLC

Numerous papers have been written over the last 20 years forecasting a deterioration in the quality of calcined petroleum coke for the aluminum industry. Until recently, average calcined coke quality has not changed significantly and the industry has had no problem sourcing low sulfur (<3%), low vanadium (<300ppm) cokes with good bulk densities. This situation is changing however, and 2008 represented a turning point. Demand for coke has increased rapidly with the growth in aluminum production and the trend towards processing heavier, higher sulfur crudes by the refining industry is having a significant impact on coke quality and availability. This paper presents an overview of the changes that have occurred and will continue into the future. The industry is adapting but the rate of adaptation will need to increase in the future towards the use of cokes with higher impurity levels, higher sulfur, lower bulk densities and more isotropic structures.

2:30 PM Keynote
Inert Anodes - The Status of the Materials Science, the Opportunities They Present and the Challenges That Need Resolving before Commercial Implementation: *Barry Welch*[1]; [1]Welbank Consulting Ltd.

Arguments in favor of developing smelting technology with inert anodes include; capital saving by eliminating the need for anode fabrication, baking and rodding plant; potential capital saving in the smelter by enabling higher intensity production per unit volume of cells; elimination of a significant ongoing material cost; reduced greenhouse gas emissions; and even lowering the cell voltage. With an increasing proportion of electricity being generated from natural gas or coal, the environmental advantage is substantially diminished and that appeal is more confined to areas with substantial nuclear or hydropower. As the materials science for producing satisfactory electrodes has progressed two paths emerged -ceramic or cermet conducting electrodes which has been championed by Alcoa, and the so-called "metal anodes" developed by Moltech and protected by more than 80 patents. In reality these two types of the electrodes become extremely similar at the active interface with the electrolyte and ultimately the choice will probably be favoured by engineering considerations. Both groups can claim they appear to have conquered the materials science with respect to the primary criteria, and both have reported larger scale trials retrofitting conventional cell design. These trials have thrown up new challenges, especially in design that will enable high intensity production and withstand the new operating conditions. Retrofitting may not be the preferred path The materials science aspects will be summarized together with the engineering challenges and my more likely development paths from this point on.

3:00 PM Invited
Use of Under-Calcined Coke for the Production of Low Reactivity Anodes: *Jérémie Lhuissier*[1]; Laïlah Bezamanifary[1]; Magali Gendre[1]; Marie-Josée Chollier[1]; [1]Rio Tinto Alcan

The quality of petroleum coke used for the production of anodes for the aluminium industry is declining, affecting both the density and the purity of the cokes. Anodes produced from these cokes will have higher reactivity resulting in higher carbon consumption in the smelter. Under-calcined coke can be used as a response to the higher anode reactivity. The coke has a similar structure to the binder phase (a mix of ultra fines and coal tar pitch), resulting in a more homogenous reactivity of all anode components following baking. The preferential consumption of the anode binder phase by the side reactions with oxygen or carbon dioxide in the cell is reduced. In this paper, the theory behind the performance of under-calcined coke will be reviewed. This will be followed by results from laboratory experiments and industrial tests. Examples of the benefits obtained by plants using under-calcined coke will be provided.

3:25 PM Invited
Use of Shot Coke as an Anode Raw Material: *Les Edwards*[1]; Franz Vogt[1]; Ric Love[2]; Tony Ross[2]; William Morgan[3]; Marilou McClung[2]; R.J. Roush[2]; Mike Robinette[1]; [1]Rain CII Carbon LLC; [2]Century Aluminium of West Virginia; [3]Century Aluminum of Kentucky

With the aluminum industry's rapidly growing demand for anode grade petroleum coke, supplies have become very tight and the industry has started using cokes not considered suitable as little as 5 years ago. Shot coke is available in large volumes and is currently used for fuel and TiO2 applications. Rain CII Carbon and Century Aluminum started a project in 2004 to explore the use of shot coke and other isotropic cokes for anode production. The project was intended to address anode grade coke shortages being felt by the industry today. The paper summarizes laboratory and plant test work completed and makes a strong case for routine incorporation of up to 30% of these cokes into anode blends. Depending on how it is used, shot coke can improve properties such as anode density and it offers a lower cost raw material in today's world of escalating raw material costs.

3:50 PM Break

4:00 PM Invited
Maintaining Consistent Anode Density Using Varying Carbon Raw Materials: *Siegfried Wilkening*[1]; [1]VAW Aluminum Technology

At first, fundamental structural properties such as real density, apparent density and porosity of the solid carbon raw materials as well as of the green and baked anode blocks will be discussed. In this context practical examples and correlations will be given, and a method for the important determination of the apparent density and total porosity of coke particles will be presented. A method and facility will further be described how to react in the control room of the paste plant to density changes of the particle fractions in the preparation plant. The up-to-date vibro-compactor is key equipment, which allows the immediate adjustment of the compaction and thus the forming density of the green anode. A major problem is currently to bake high-density anodes without cracks. A future high-temperature mixing and forming process will be outlined to overcome some quality deficiencies of the carbon raw materials.

4:25 PM Invited
Combating Anode Quality Trends in Potrooms: *Mark Taylor*[1]; [1]University of Auckland

Three factors are conspiring to create greater challenges for anodes in smelters. Firstly the quality of raw materials is deteriorating – especially coke quality. Secondly the amperage on each cell technology is still increasing year on year, with substantial impacts on heat generation in anodes and electrolyte. And thirdly the impact of greenhouse policy is driving ever lower carbon consumption targets. This presentation describes practical measures which smelters can take to counteract the first two factors above, in order to achieve the third. Specifically the issue of anode temperature needs to be addressed in order to counteract coke reactivity trends. Similarly, anode/assembly induced stresses in the carbon must be tackled both from an assembly design viewpoint and from an anode cover and bath processing design perspective if systematic anode cracking is to be avoided. Lastly the impact of operating practice with anode cover and dressing is pervasive.

4:50 PM Invited
The Origin and Abatement of SO_2 Emissions from Primary Aluminium Smelters: *Stephan Broek*[1]; Brian Rogers[1]; [1]Hatch Ltd

Modern aluminium smelters use dry scrubbing technologies to clean the ventilation gases from electrolytic cells before they are emitted into the atmosphere. Particulate and fluoride emissions are typically very low but while this is an excellent achievement, focus is slowly shifting towards the one emission that remain untouched, which is SO_2 that originates from the sulphur in the coke. In this paper is discussed the sulphur balances of modern aluminium smelters to understand where and how sulfur in the form of SO_2 is emitted. It will provide insight in what the best strategy is to address the issue of reducing sulphur emissions. Technologies are available to provide effective emissions reductions and over recent years many have been reviewed for their effectiveness. The paper will show some of the readily available technologies as well as taking a peek at emerging technology that may help the smelters to further mitigate their emissions.

5:15 PM Invited
The Downstream Consequences of Rising Ni and V Concentrations in Smelter Grade Metal and Potential Control Strategies: *John Grandfield*[1]; J.A. Taylor[2]; [1]Grandfield Technology Pty Ltd; [2]University of Queensland

Technology for controlling smelter metal impurities post reduction has steadily improved. For example control of sodium has seen the reduction and in some plants elimination of chlorine gas. However, changes in the purity of cell feed materials such as anodes are giving rise to new challenges in impurity control; vanadium and nickel levels are for example an emerging problem. This paper reviews the important impurities and their effects on downstream casting, forming and final application properties. Methods of controlling these impurities are also discussed and areas where new technology is needed also highlighted. In some cases it is not know where the tolerable limits of impurities are. There are a plethora of metal refining techniques used in the extraction of other metals which can be investigated for control of impurities in smelter grade aluminium.

Emerging Applications of Neutron Scattering in Materials Science and Engineering: Phase Transformation

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Xun-li Wang, Oak Ridge National Laboratory; Brent Fultz, California Institute of Technology; Hahn Choo, University of Tennessee

Tuesday PM — Room: 3012
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Brent Fultz, California Institute of Technology; Kenneth Kelton, Washington University

2:00 PM Invited
Ordering in Liquids and the Influence on Phase Transitions: *Kenneth Kelton*[1]; [1]Washington University

Recent developments in novel levitation methods have led to a renewed interest in the structures of equilibrium and supercooled liquids. Such investigations are also of practical note, allowing regions of the alloy phase diagram to be quickly and accurately determined. Employing the technique of electrostatic levitation, we have measured metallic and semiconductor liquid x-ray diffraction patterns as a function of supercooling. The liquid order was characterized in terms of a dominant local cluster and by Reverse Monte Carlo (RMC) fits to the diffraction data. The topologies of the RMC structures are expressed in terms of their bond orientational order parameters and Honeycutt-Andersen indices. Case studies are presented and discussed, focusing on the influence of liquid ordering on thermophysical properties and phase transitions. Evidence for icosahedral short-range ordering in transition metal liquids and its role in crystallization and the glass transition are discussed.

2:30 PM
Structural Studies of Amorphous Materials: *Joerg Neuefeind*[1]; [1]Oak RIdge National Laboratory

Many structural materials are amorphous materials. While this name may suggest that these materials are structureless, they in fact do possess structure but no long-range periodicity and their properties are intimately liked to that structure. Neutron and X-ray scattering provide complementary information to understand the structure and ultimately the properties of amorphous systems. Neutrons in particular offer high sensitivity to light elements, high penetration power, isotope substitution experiments and sensitivity to magnetic moments. The Nanoscale Ordered Materials Diffractometer (NOMAD) under construction at the Spallation Neutron Source in Oak Ridge and scheduled to be completed in 2010 will allow neutron scattering experiments with unprecedented speed. Recent examples of structural studies of amorphous materials including deformation studies and time-resolved experiments to access metastable structures will be given.

2:50 PM
Neutron Diffraction Study Of Amorphous Zr-Cu Alloys Using Isotopic Substitution: *A.D. Stoica*[1]; Dong Ma[1]; X.-L. Wang[1]; M. Kramer[2]; [1]Oak Ridge National Laboratory; [2]Ames National Laboratory

The recent discovery of bulk metallic glasses in the binary Zr-Cu system sparks renewed interest in determining atomic structure of amorphous alloys. In this study, a series of amorphous Zr-Cu alloys have been investigated by time-of-flight neutron diffraction using isotopic substitutions with 63Cu, 65Cu and natural Cu, respectively. Partial structure factors and partial pair correlation functions were determined in terms of both Faber-Ziman and Bhatia Thornton formalisms. By linking the resolved structures factors with packing of atomic clusters, this study sheds light on the topological and chemical orders in amorphous structure.

3:10 PM Invited
Dynamic Pair-Density Function Method for Neutron Scattering: *Takeshi Egami*[1]; Wojciech Dmowski[1]; [1]University of Tennessee

In crystals the atomic vibrations are described by phonons, of which dispersion can be determined by inelastic scattering of neutrons and x-rays. In glasses, liquids and strongly disordered crystals, however, phonons are damped, scattered, and localized. The conventional triple-axis spectrometer is not suited to catching these local modes. The dynamic pair-density function (DPDF) method was invented to catch these local modes of lattice and spin dynamics [1]. The dynamic structure factor S(Q, E) is Fourier-transformed to obtain the DPDF, g(r, E), that describes the atomic vibration at a distance r, with the frequency E/η. In crystalline solids this method captures only the semi-localized modes near the Van Hove singularities. We demonstrate how the DPDF describes local vibrations of Pb in relaxor ferroelectric Pb(Mg1/3Nb2/3)O3, and gives insight into the dynamic origin of the relaxor behavior. [1] W. Dmowski, et al., Phys. Rev. Lett. 100, 137602 (2008).

3:40 PM Break

4:00 PM Invited
Lattice Dynamics and Structural Phase Transitions: *Stephen Shapiro*[1]; [1]Brookhaven National Laboratory

Inelastic neutron scattering is an ideal probe to study the lattice dynamics and their modifications due to external perturbations such as temperature, pressure, electric or magnetic field, etc. Of particular interest is the anomalous behavior of the lattice dynamics of advanced materials undergoing phase transitions such as martensitic transformations. The soft mode theory of structural phase transitions was developed nearly 50 years ago and successfully predicted that the energy of a specific lattice mode would go to zero as the transition is approached and the material would spontaneously transform to a different structure. In this talk I shall review how neutron studies of single crystals show that specific modes exhibit strong anomalies that are precursors to the transitions. These observations, coupled with first principles calculations of the interaction between the electrons and the lattice can explain the driving mechanism of the phase changes in several metallic systems undergoing martensitic transformations.

4:30 PM Invited
Inelastic Neutron Scattering Studies of Material Dynamics and Thermodynamics: *Brent Fultz*[1]; [1]California Institute of Technology

When neutron wavelengths match interatomic distances, the neutrons have energies typical of room temperature. This is lucky — with inelastic neutron scattering, thermal vibrations of atoms and electron spins can be studied with enough detail to reveal both their energies and their wavelengths. Because atom motions are where heat is stored in solids, vibrations generate entropy. Differences in vibrational entropy are now known to be big enough to alter the phase stabilities of different alloys. More recently, we have been studying how vibrational frequencies change with temperature. There are numerous exceptions to the textbook story of how thermal expansion causes vibrational frequencies to decrease in a non-parabolic potential. In this talk I will show why phonon-phonon interactions and electron-phonon interactions, which change the textbook story, are big enough to alter the phase diagrams of materials at elevated temperatures, and how they are studied by inelastic neutron scattering.

5:00 PM
Thermodynamics from Elementary Excitations: Combined Studies with Inelastic Neutron Scattering and First-Principles Simulations: *Oliver Delaire*[1]; Matthew Lucas[1]; Max Kresch[2]; Jiao Lin[2]; Brent Fultz[2]; [1]Oak Ridge National Laboratory; [2]Caltech

Inelastic neutron scattering is the preferred experimental technique to measure phonons and magnons, but it has long been limited by the low flux available at neutron sources, which limited the range of practical experiments. The high neutron flux of the Spallation Neutron Source, combined with the large detector banks of new time-of-flight spectrometers, allows for a qualitative change in feasible inelastic scattering measurements. However, a difficulty in analyzing the neutron scattering results resides in the complexity of the experimental datasets. First-principles simulations of the phonon dynamics have now reached a level of reliability such that the full phonon S(Q,E) predicted from quantum mechanics can directly be compared to experimental data. I will present examples of phonon investigations in transition metal alloys, that have benefited from combined studies with inelastic neutron scattering and first-principles (density functional theory) calculations.

5:20 PM Invited
Neutrons Probe Dynamics in Metals and Polymers: *Kenneth Herwig*[1]; [1]Oak Ridge National Laboratory

With the recent construction of new facilities and major upgrades of existing sources, world-wide access to advanced neutron scattering instrumentation has never been better. Techniques which have been historically limited by low neutron fluxes will benefit greatly from the increased power of the new sources including the Spallation Neutron Source (SNS) at the Oak Ridge National Laboratory. This is particularly true for inelastic neutron scattering where a neutron exchanges energy with atoms in a sample conveying information on the motion of atoms. Neutrons are particularly adept at identifying the diffusive motions of hydrogen atoms. Examples include identifying the various states of water during the curing of cement pastes and the high temperature diffusion of hydrogen atoms in metals. This talk will briefly introduce inelastic and quasielastic neutron scattering, continue with applications, and conclude with a summary of the instrumentation available at the SNS for these types of measurements.

Fatigue: Mechanisms, Theory, Experiments and Industry Practice: Fatigue in Engineering Components

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

Program Organizers: Koenraad Janssens, Paul Scherrer Institute; Corbett Battaile, Sandia National Laboratories; Brad Boyce, Sandia National Laboratories; Luke Brewer, Sandia National Laboratories

Tuesday PM Room: 3008
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Ryuichiro Ebara, Hiroshima Institute of Technology; Koenraad Janssens, Paul Scherrer Institute

2:00 PM Invited

Residual Stress Profiles for Mitigating Fretting Fatigue in Gas Turbine Engine Disks: *Kwai Chan*[1]; Michael Enright[1]; Patrick Golden[2]; Ramesh Chandra[3]; Alan Pentz[3]; [1]Southwest Research Institute [2]Air Force Research Laboratory; [3]NAVAIR

The driving force for fretting fatigue in engine disks is the contact stresses generated by fretting of the blade and the disk surfaces in the attachment region. In this paper, we examine the use of different residual profiles to counteract the undesirable effects of contact stresses and to mitigate fretting fatigue. A global finite-element analysis of the disk blade assembly is first performed. The contact pressure and shear traction at the attachment region are extracted from the FEM results and used to compute the contact stress distribution. The contact stresses are then combined with the residual stresses and the bulk stresses. The overall stress distribution is then utilized in a probabilistic crack growth model to predict the risk of disk failure for a military engine under simulated loading conditions. The results are used to identify the minimum residual stress profile for mitigating fretting fatigue in engine disks.

2:30 PM

Fatigue Life Improvement of C-130 Airplane Propeller Blade Roots by Cold Surface Rolling: *Behzad Majidi*[1]; [1]Amirkabir University of Technology

In turboprop engines of C-130 aircrafts, the propeller assembly has an important contribution to the safety of the aircraft and its failure leads to catastrophic aircraft crash. Service life of propeller blades is increased by conducting cold surface rolling to generate near surface compressive stresses. In this study service loads of the propeller has been simulated and maximum service stresses was determined. Compressive stress gradients were calculated from finite element modeling of rolling process using ABAQUS/Explicit. Fatigue lives of blades with and without rolling process determined by Basquin method. Results showed that the surface rolling has a considerable effect on fatigue life of the blades and results in 20 times more fatigue life.

2:50 PM

Effect of Varying Elasticity of Helical Gear Teeth on Loading and Stressing: *Ahmed Elkholy*[1]; [1]Kuwait University

The study presents an analytical model for the calculation of loads and stresses of helical gears due to machining and assembly errors. The model accommodates, tooth elastic deformation caused by contact loads and the varying meshing stiffness of engaged teeth due to material properties. Load sharing among meshing teeth is also determined throughout the contact from the elastic deformation and gear material properties. To check the validity of the model, the changes in maximum root stresses throughout the contact regions have been compared with strain gauge measurement and finite element results. All results compared very well and the maximum deviation did not exceed 11%. The model, therefore, provides accurate load capacity rating of helical gear drives, and reduces computation time when compared with similar models available from literature.

3:10 PM

Structural Prognosis During an EA-6B Outer Wing Panel Fatigue Test: *John Papazian*[1]; Elias Anagnostou[1]; Stephen Engel[1]; Daniel Fridline[1]; John Madsen[1]; Jerrell Nardiello[1]; Robert Silberstein[1]; [1]Northrop Grumman

A full-scale fatigue test of a retired EA-6B outer wing panel was conducted to validate the functioning of the Northrop Grumman/DARPA Structural Integrity Prognsosis System (SIPS). The panel had been retired from active service with a Fatigue Life Expended index of 185. Laboratory fatigue testing of the entire panel was performed to determine its remaining life, evaluate several sensor systems, evaluate the SIPS fatigue models, and validate the SIPS reasoning and prediction system. Phased array ultrasonics was used as a non-destructive inspection system to establish the starting state, and pitch-catch ultrasonics, eddy current and electrochemical fatigue sensors were used to monitor fasteners on rib 1 during the test. The entire system performed admirably, and the accuracy and precision of the remaining life predictions improved continuously during the test.*This work sponsored by the Defense Advanced Research Projects Agency under contract HR0011-04-C-0003. Dr. Leo Christodoulou is the DARPA Program Manger

3:30 PM

Rotating Bending Fatigue Tests at High Speed (150 Hz) on AISI-SAE 1018 Steel, Close to Elastic Limit: *Gonzalo Dominguez*[1]; Mauricio Guzmán[1]; Lourdes Mondragón[2]; Edgar Tapia[1]; [1]University of Michoacan; [2]Technologic Institute of Morelia

This work deals with rotating bending fatigue tests at high speed (150 Hz), carried out on AISI-SAE 1018 steel for witch the highest experimental stress inside the specimen is close to the elastic limit of material. Simulation results are obtained by Visual Nastran software in order to determine the numerical stress and strain distributions in the specimen; afterwards, this information is used for the experimental set up. A general description of experimental test machine and experimental conditions are developed too, then, experimental results are presented and discussed according the observed failure origin related to the high stress zones. Finally, a simple model is proposed to predict the fatigue life of this steel under high speed rotating bending fatigue tests based on the geometrical properties of fatigue failure origin and the micro-plastic deformation zones close to crack initiation.

3:50 PM Break

4:10 PM

The Examination of Failure of Crankshaft Used in Tractors: *Huseyin Adanir*[1]; [1]New Mexico Tech.

Failure of crankshaft samples that used in tractors were examined. The samples had been taken from real life and failure during service. Metallurgical factors, design and manufacturing process steps were investigated for crankshaft samples. Materials characterization such as chemical composition analysis of the samples, metallography, and hardness were investigated and SEM was used to examine fracture surfaces. As known that from design and manufacturing process of crankshaft, micro-alloyed steel used as crankshaft material and shaped by forging method. After forging process, normalization as a heat treatment and surface hardening by induction method were done. In the micrographs examination, due to Mn and S content in the steel chemical composition, occurring of MnS forms are discussed. Micro hardness testing with hardness vs depth scrutiny was investigated. Materials selection as a design criteria and manufacturing procedure of crankshaft were discussed and finally, some advices were given to prevent future crankshaft failures.

4:30 PM

Fatigue Life Estimation for Tank Vehicle Structures Subjected to Liquid Sloshing: *Liming Dai*[1]; [1]University of Regina

Strength analysis of heavy vehicles carrying liquid cargo becomes complex because of the dynamic liquid behavior due to external excitations and operation maneuvers. The research work discussed in this article is on an investigation of the fatigue life estimation of the subframe of a B-Train tank vehicle. The effect of liquid sloshing inside the tank on the structure of the vehicle is determined through a newly developed approach. The stress and strain histories of the subframe subjected to the liquid slosh are quantified with the employment of numerical analyses. Various operating conditions of the tank truck, such as sway, turning, lane change, sudden acceleration and braking, are considered in the investigation. The research results generated will benefit the heavy vehicle industry in designing a sturdy structure and will decrease the time and cost in the design stage of such vehicles.

4:50 PM
Fatigue Susceptibility of Silicon Thin Films in Harsh Environments With and Without Nanometer-Scale ALD Alumina Coatings: *Michael Budnitzki*[1]; Olivier Pierron[1]; [1]Georgia Institute of Technology

The present study investigates the fatigue degradation of 2-micron-thick polycrystalline silicon notched cantilever beam structures in a high-temperature (80C), high-humidity (90%RH) environment. In addition, some structures are coated with ~20nm of atomic-layer-deposited (ALD) alumina (Al2O3). The specimens are subjected to fully reversed sinusoidal loading at resonance (~40 kHz) with stress amplitudes ranging from 2.1 to 2.3 GPa, resulting in life-spans between 10^6 and 10^9 cycles. The degree and rate of degradation are assessed by monitoring the change in resonant frequency resulting from a change in compliance of the structure. It is found that the damage accumulation rate in the 80C, 90%RH environment exceeds the reference (30C and 50%RH) by two orders of magnitude and tends to increase towards failure, as opposed to decreasing rates at 30C, 50%RH. Preliminary data on ALD coated devices suggest a considerably decreased susceptibility to fatigue degradation.

5:10 PM
Effect of Load Ratio and Hydrogen Concentration on Crack Growth Rate in Zr-2.5Nb Tubes: *Young Suk Kim*[1]; Vidas Makarevicius[1]; [1]Korea Atomic Energy Research Institute

Crack growth rates (CGRs) were determined under sustained and cyclic loads using 17 mm compact tension and cantilever beam specimens taken from the Zr-2.5Nb tubes charged to 6 to 100 ppm H. The cyclic load effect on the CGR was investigated at 250o^C where load ratios, R changed from 0.13 to 1 with constant Kmax. Under sustained loads, the CGR of the Zr-2.5Nb tube increased with increasing supersaturation of hydrogen, deltaC and leveled off above 20 to 25 ppm H of the deltaC. Under cyclic loads with 1 cycle/min, the CGR at 250^oC decreased with decreasing R: 3.2x10^-8 m/s at R=1 and 4.8x10^-9 m/s at R=0.13. The striation spacing, corresponding to the critical hydride length, decreased with decreasing R, indicating easier cracking of the hydrides under cyclic loads. The decreased CGR under cyclic loads and its dependence on the deltaC are discussed using Kim's delayed hydride cracking model.

5:30 PM
Effect of Conductivity and Dissolved Hydrogen on Environmental Fatigue Behaviors of Type 316LN Stainless Steel in Pressurized Water Reactor Environment: *Hun Jang*[1]; Pyoung Cho[1]; Changheui Jang[1]; Hyunchul Cho[2]; [1]KAIST; [2]Corporate R&D Institute Doosan Heavy Industries and Construction Co., Ltd.

Low cycle fatigue damage was known as one of the main degradation mechanisms of structural materials in nuclear power plant. To investigate the environmental fatigue behaviors of type 316LN stainless steel, low cycle fatigue tests in a simulated pressurized water reactor environment at 310^{o}C has been performed. Among many parameters affecting the fatigue resistance in such condition, the effects of conductivity and dissolved hydrogen are subject of interest in this paper. Conductivity and dissolved hydrogen may act as environmental factors in role of environmental assisted cracking. In this regard, we are performing low cycle fatigue tests of type 316LN stainless steel in pressurized water reactor environment. And then, effects of conductivity and dissolved hydrogen on environmental fatigue behaviors of type 316LN stainless steel will be investigated from fractography analysis and by comparing current results with previous ones obtained in 310^{o}C deoxygenated water environment.

Friction Stir Welding and Processing-V: Session IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee
Program Organizers: Rajiv Mishra, Missouri University of Science and Technology; Thomas Lienert, Los Alamos National Laboratory; Murray Mahoney, formerly with Rockwell Scientific

Tuesday PM — Room: 2014
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Dwight Burford, Wichita State University

2:00 PM Invited
Friction Stir Spot Welding of Advanced High Strength Steels: *Yuri Hovanski*[1]; Michael Santella[2]; Glenn Grant[1]; [1]PNNL; [2]Oak Ridge National Laboratory

Friction stir spot welding was used to join two advanced high-strength steels using polycrystalline cubic boron nitride tooling. Numerous tool designs were employed to study the influence of tool geometry on weld joints produced in both DP780 and a hot-stamp boron steel. Tool designs included conventional, concave shouldered pin tools with several pin configurations; a number of shoulderless designs; and a convex, scrolled shoulder tool. Weld quality was assessed based on lap shear strength, microstructure, microhardness, and bonded area. Mechanical properties were functionally related to bonded area and joint microstructure, demonstrating the necessity to characterize processing windows based on tool geometry.

2:20 PM
Evaluation of Friction Stir Spot Welded Advanced High Strength Steel: *Jeff Rodelas*[1]; Raiv Mishra[1]; Greg Hilmas[1]; Wei Yuan[1]; [1]Missouri University of Science and Technology

Friction stir spot welding (FSSW) is a solid-state alternative to resistive spot welding (RSW) of advanced high strength steels (AHSS), such as dual phase (DP) and fully martensitic steels. By avoiding bulk melting, FSSW offers the benefit of avoiding weld solidification defects that tend to be problematic in AHSS. Steel sheets were joined using plunge-type FSSW with a cemented carbide tool. Lap shear strengths for DP 590 steel 2 to 3.5 times greater than existing RSW design guidelines were obtained using FSSW process parameters which maximized the bonded region. Additionally, the relationship between failure mode and lap shear strength was interpreted in the context of bonded area. Microstructures of the resulting welds and tool wear mechanisms will also be discussed.

2:40 PM
Metallurgical Characterization of Friction Stir Spot Welded Mg Alloy: *Qi Yang*[1]; Sergey Mironov[2]; Yutaka Sato[2]; Kazutaka Okamoto[3]; [1]Hitachi American Ltd; [2]Tohoku University; [3]Hitachi, Ltd

Hook and metallurgical features such as microstructure and texture are directly related to the thermo-mechanical interaction between the material and welding tool, and they could significantly affect strength of a friction stir spot weld. In the present work, friction stir spot welding was carried out on AZ31 Mg sheets in lap configuration at various rotation speeds and plunge depths. Copper foil, pre-placed between the overlapped metal sheets, was used as a tracer to apparently monitor material flow in the vicinity of the tool during welding. Furthermore, electron backscatter diffraction was conducted to obtain the microstructures and preferred crystallographic orientations in the stir zone and the hook region, respectively. Using these two characterization approaches, an understanding of hook formation and microscopic features, especially texture, in Mg spot welds was attained. Finally, the effect of tool rotation speed on the strength of a friction stir spot weld was discussed.

3:00 PM
Study of Failure Modes in Friction Stir Swing Welded Lap Shear Specimens: *Harsha Badarinarayan*[1]; Qi Yang[1]; [1]Hitachi America Ltd

Swing (Stitch) friction stir spot welding is an extension of the conventional friction stir spot welding. This process produces an elongated spot (hence, the presence of advancing and retreating sides). The main advantage of this process is that it gives appreciably higher strength. In this research, an attempt is made to understand the failure mechanism of stitch welds made in lap shear configuration. There are 4 possible ways (orientations) in which coupons can be welded to produce a lap shear specimen – 2 in transverse direction and 2 in longitudinal. The static strength of welds made with these orientations was found to be different. A fracture mechanics and finite element analysis approach is used to explain this variation in strength. It was seen that failure occurred quicker when spot welds were loaded on the retreating side or when the loading condition caused the stress concentration around the keyhole.

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Low Z-Force Friction Stir Spot Welding: *Tze Jian Lam*[1]; Christian Widener[1]; Jeremy Brown[1]; Dwight Burford[1]; [1]Wichita State University

Low Z-force FSSW can be achieved by studying the relationships between pin tool features, geometries, processing parameters and the resultant strength of FSSW. The objective of this research paper is to study the effects of geometrical changes to the pin tool, such as shoulder and pin diameter without compromising the mechanical properties of the spot weld. A variety of pin tool features are included in this study in an effort to reduce the required Z-force. Two concave shoulder diameters and three probe designs were investigated. The probe features included flutes, threads, Trivex™ and flats. The process parameters included in the research are spindle speed, plunge depth, plunge load and plunge rate. Static ultimate tensile strength (UTS) and metallographic analysis are correlated with process parameters, process forces, static UTS and pin tool designs.

3:40 PM
Joining PPSU Thermoplastic to 6061 Aluminum Using the Refill Friction Stir Spot Welding Process: *Adam Gladen*[1]; William Arbegast[1]; Michael West[1]; Bharat Jasthi[1]; [1]South Dakota School of Mines and Technology

This paper presents the results of a preliminary study to investigate the joining of polyphenylsulfone (PPSU) thermoplastic sheets to 6061 aluminum sheets in lap joint configurations using the Refill Friction Stir Spot Welding (RFSSW) process. Both the "pin first" and "shoulder first" RFSSW methods were investigated. The RFSSW process on these materials results in a "button" of extruded aluminum being formed within the PPSU sheet which contributes to a strength which exceeds 300 pounds per spot. Samples were prepared with and without a supersonic cold sprayed (SCS) copper (Cu) interlayer between the sheets to enhance the bonding to the PPSU surfaces. The effects of process parameters and interface coatings on the joint quality and strength are discussed. Lap shear test results per AMS-W-6858 are presented along with metallurgical examinations of the interface. Overall, these findings show that the RFFSW can result in mechanical joints between these two radically different materials.

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Friction Stir Form Welding of Aluminum Tubes: *K. Gupta*[1]; Rajiv Mishra[1]; Y. Chen[2]; X. Gayden[2]; [1]Missouri University of Science and Technology; [2]GM R&D Center

This paper summarizes the results of different methods of joining 6063 rectangular section aluminum tubes by friction stir welding (FSW). FSW was evaluated for three tube configurations: unsupported, plug supported and unsupported with top sheet. In these runs, fixturing and process variables were kept fixed and variability was assessed only with respect to the joint configuration. Two different tool designs were used for making these runs. Metallurgical and mechanical properties evaluations were done for three weld configurations. Comparison of properties was done to demonstrate the best joining method for tubes. Unsupported welds exhibited best mechanical properties, with 65% weld efficiency. This work was performed under the NSF-IUCRC for Friction Stir Processing and the additional support of NSF and GM for the Missouri S&T site is acknowledged.

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Friction Stir Spot Welding of Aluminum Alloy 6016: *Wei Yuan*[1]; Rajiv Mishra[1]; Yen Lung Chen[2]; Xiaohong Gayden[2]; Glenn Grant[3]; [1]Center for Friction Stir Processing, Department of Materials Science and Engineering,; [2]General Motors R&D Center; [3]Pacific Northwest National Laboratory

Friction stir spot welds of 6016-T4 aluminum alloy were made using conventional pin (CP) tool and off-center feature (OC) tool. Different tool rotation speeds were employed for maximizing bonded region: 1500 rpm for CP tool and 2500 rpm for OC tool. Effects of penetration depth and plunge speed on cross-tension failure load were investigated. Maximum failure load of about 1.8 kN was obtained. Results indicated cross-tension failure load did not change after paint-bake cycle for both tools. Two different failure modes were observed: debonding and pull-out under cross-tension loading condition. Based on the experimental observation of failure path, load-displacement curve and microhardness profile, the effect of paint-bake cycle on cross-tension strength and the failure mechanisms are discussed.

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Refill Friction Stir Spot Weld Process Optimization for 2024, 6061, and 7075 Aluminum Lap Joints: *Clark Oberembt*[1]; William Arbegast[1]; Dana Medlin[1]; Michael West[2]; [1]SDSM&T; [2]South Dakota School of Mines and Technology

This paper describes the optimization procedures and results for the refill FSSW of 2024, 6061, and 7075 Aluminum alloys in the 0.040", 0.060", 0.080", & 0.125" sheet thicknesses. Both the "pin first" and "shoulder first" process methods were evaluated using test coupons consistent with AMS-W-6858. Welds were conducted on the MTS ISTIR 10 FSW system with custom designed FSSW adapters. These results of several designed experiments which varied processing parameters and pin tool designs are presented along with the results of static strength testing and metallurgical evaluations. The effect of spot size on strength and failure mode is presented along with the compression test results of integrally stiffened compression test panels fabricated using the refill FSSW process.

5:10 PM
Evaluation of Swept Friction Stir Spot Welding in 2219-T62: Dwight Burford[1]; Christian Widener[1]; Jeremy Brown[1]; Ken Poston[2]; Gary Moore[2]; [1]Wichita State University; [2]Bombardier Aerospace Short Brothers, PLC

The purpose of this investigation was to evaluate the effects of swept Friction Stir Spot Welding (FSSW) on tensile strength and fatigue life in 2219-T62 material with a faying surface gasket compound. The sheets were 2.5 mm (0.100-in.) thick. The top sheet was chromic acid anodized while the bottom sheet was sulfuric acid anodized. A poly-urethane based non-setting and non-hardening gasket compound was placed at the faying surface. The first round of testing involved exploratory bounding of the process windows for three tools. The bounding spots were evaluated through macroscopic inspection of spot cross sections. One tool was eliminated in the first round; however, the remaining two tools were evaluated for coupon tensile strength. The coupons were pulled to failure in a single spot, unguided lap shear configuration. Weld parameters for each tool varied per a Box-Behnken design of experiment (DOE). Coupons were also produced for limited fatigue testing from the 3 best weld parameters for each tool. The coupons were made in the 100% load transfer configuration from the NASM 1312-21 specification. A single tool was then chosen to continue based on the previous tensile and fatigue results. Another set of DOEs were performed to evaluated tensile strength and fatigue life. These DOEs again used the NASM 1312-21 100% load transfer coupons. Select FSSW coupons were then compared to riveted coupons at equal fatigue load levels. The rivets used in this experiment were MS20426E5-7 flush countersink. The FSSW coupons were able to outperform the riveted coupons in regards to tensile strength and for fatigue life at high load levels. At lower load levels FSSW coupon results were comparable to riveted coupons.

5:30 PM
Energy Generation during Friction Stir Spot Welding (FSSW) of Al 6061-T6 Plates: *Mokhtar Awang*[1]; Victor Mucino[2]; [1]Universiti Teknologi Petronas; [2]West Virginia University

Effective and reliable computational models would greatly enhance the study of energy dissipation during friction stir spot welding (FSSW) process. Approaches for the computational modeling of the FSSW process, however, are still under development and much work is still needed, particularly the application of explicit finite element codes for a verifiable simulation. The objectives of this work are to develop a finite element modeling of FSSW of 6061-T6 aluminum alloy and analyze energy generation during the welding process. In this work, a three dimensional (3-D) finite element (FE) coupled thermal-stress model of FSSW process has been developed in Abaqus/Explicit code. Rate dependent Johnson-Cook material model is used for elastic plastic work deformations. Temperature profile and energy dissipation history of the FE model have been analyzed. The peak temperature at the tip of the pin and frictional dissipation energy are in close agreement with an experimental work.

5:50 PM
Refill Friction Stir Spot Weld Process Optimization for Magnesium AZ31B-H24 Lap Joints: *Clark Oberembt*[1]; William Arbegast[1]; Dana Medlin[1]; Michael West[1]; [1]South Dakota School of Mines and Technology

This paper describes the optimization procedures and results for the Refill FSSW of AZ31B-H24 Magnesium sheet in lap joint configurations. Both the "pin first" and "shoulder first" process methods were evaluated using test coupons consistent with AMS-W-6858. Samples were prepared on the MTS ISTIR 10 FSW system with custom designed RFSSW adapters. The unguided lap shear, cross tension, T-peel, and fatigue test results are presented. Metallurgical and SEM analysis and microhardness testing results are presented with correlations to the effects of processing parameters and defect formations. High strength and high quality RFSSW spot welds were produced with the results compared to conventional resistance spot welds in this magnesium alloy.

Frontiers in Solidification Science III: Prediction and Control of Solidification Behavior and Cast Microstructures

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Phase Transformations Committee, TMS: Solidification Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Ralph Napolitano, Iowa State University James Morris, Oak Ridge National Laboratory

Tuesday PM Room: 2018
February 17, 2009 Location: Moscone West Convention Center

Session Chair: To Be Announced

2:00 PM
Recent Advances in Phase-Field Modeling of Polycrystalline Solidification: *Laszlo Granasy*[1]; Gyorgy Tegze[1]; Gyula Toth[2]; Laszlo Kornyei[2]; Tamas Pusztai[2]; [1]Brunel Centre for Advanced Solidification Technology; [2]Research Institute for Solid State Physics and Optics

Various aspects of polycrystalline solidification will be addressed in the framework of conventional and atomistic phase field approaches. Parameter-free predictions for the nucleation barrier from Ginzburg-Landau free energy based phase-field models are compared to results from Monte Carlo simulations and other nucleation models in the hard-sphere system of recently revised interface properties [1]. A similar approach is applied for competing fcc-bcc nucleation in the Fe-Ni system. Solidification in a phase separating liquid will also be addressed. A phase-field model of polycrystalline solidification that relies on quaternion fields in representing crystallographic orientation is applied for describing complex 3D solidification structures including multi-particle dendritic morphologies, columnar to equiaxed transition, and various types of spherulites. Preliminary results will be shown for nucleation and polycrystalline solidification in phase field crystal models. [1] R.L. Davidchack et al., J. Chem. Phys. 125, 094710 (2006).

2:20 PM
Predicting the As-Cast Grain Structure with a Hybrid Stochastic-Deterministic Mathematical Model: Vinicius Biscuola[1]; *Marcelo Martorano*[1]; [1]University of São Paulo

A hybrid stochastic-deterministic model has been implemented to predict the as-cast grain macrostructure formed during solidification of binary alloys. The model is a combination of deterministic macroscopic equations, derived from mass, energy, and species conservation principles, and the cellular automaton technique to predict the nucleation and growth of grains. An isothermal and a unidirectional solidification system were simulated with the model. In the isothermal system, the grain macrostructure was obtained for either a log-normal or a normal distribution of nucleation undercoolings, allowing the calculation of the average grain size under different solidification parameters. In the unidirectional solidification system, the columnar-to-equiaxed transition was also obtained from the calculated macrostructure. The model results were compared with those from a pure deterministic and a pure stochastic model proposed in the literature.

2:40 PM
Comparative Study Of CET In Refined And Non Refined Al-Based Alloys Using In Situ And In Real Time X-Ray Synchrotron Imaging: *Hyejin Jung*[1]; N. Mangelinck-Noël[2]; H. Nguyen-Thi[2]; N. Bergeon[2]; B. Billia[2]; G. Reinhart[2]; A. Buffet[3]; J. Baruchel[3]; [1]NFRI; [2]IM2NP; [3]ESRF

The control of the Columnar to Equiaxed microstructure Transition (CET) is needed to determine final desired products. The main objective of the study presented is to better elucidate the CET phenomenon with carefully defined experiments, In-situ and real-time observation of the solid-liquid interface is a fruitful method to reveal the dynamics of the solidification phenomena. Such experiments have been conducted at the European Synchrotron Radiation Facility (ESRF) using Synchrotron X-ray Radiography and Topography. Firstly, we focus on Al-3.5wt%Ni alloy adding refining particles to study CET when it mainly comes from heterogeneous nucleation of equiaxed grains on particles followed by the blocking of the columnar front by the growing equiaxed grains. Secondly, Al-7.0wt%Si alloy without refiners is carefully considered to study fragmentation from pre-existing dendrites as another origin for equiaxed grains.Thanks to this comparative study, valuable information on CET phenomena are yielded and related to the characteristics of each alloy.

3:00 PM
Quantitative Simulation of Fe-rich Intermetallics in Al-Si-Cu-Fe Alloys during Solidification: *Junsheng Wang*[1]; Peter D. Lee[1]; [1]Imperial College

Fe-rich intermetallic phases (e.g. β-Al5FeSi) which form during the solidification of Al-Si-Cu-Fe alloys can have a detrimental effect on the mechanical properties of cast components. A thermodynamic database was combined with a numerical solution of the multicomponent diffusion solver to predict the formation of this phase. The growth kinetics and morphology was simulated using a combined Monte-Carlo algorithm to calculate the transformation frequency for each cell as a function of total free energy, with the faceted shapes replicated via a decentred needle/plate algorithm. The non-stoichiometric composition of both Fe and Si was simulated by changing the partition coefficient at the S/L interface according to local kinetic parameters such as growth velocity and effective diffusion. The model predicted phase fraction, size and morphology of Fe-rich intermetallic phases in type-319 alloys (Al-7.5wt.%Si-3.5wt.%Cu-0.2~0.8wt.%Fe) compared well to high resolution synchrotron tomography results.

3:20 PM
Size and Morphology of Gas Porosity in Castings-Computational Experiments Using a Cellular Automaton Model: *Roschen Sasikumar*[1]; Michael Walker[2]; S. Savithri[1]; [1]NIIST (CSIR); [2]General Motors Corporation

We use a cellular automaton simulation model as a virtual experimental set up to study growth of gas bubbles in solidifying Aluminum castings. The model assumes that gas porosity originates from pre-existing micro-bubbles that grow by diffusion of hydrogen from the growing solid into the bubbles. The finite time available for the diffusion of hydrogen as well as the space constraint imposed by the growing solid limit the growth of the bubbles. The cellular automaton model with rules specially adapted for bubble growth, track the solid-liquid and bubble-liquid interfaces explicitly on a fine grid. The numerical experiments are performed with a eutectic Al-Si alloy solidifying with different grain sizes and solidification rates. The size of the pre-existing bubble and the microstructural environment in which it finds itself are seen to be the critical factors that determine the final size and morphology of porosity

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High Density Nanocrystal Formation in Marginal Glass Forming Alloys: *Eren Kalay*[1]; Scott Chumbley[1]; Matthew Kramer[1]; Ralph Napolitano[1]; Iver Anderson[1]; [1]Ames Laboratory / Iowa State University

The rapid solidification products of Al-rich Al-RE and Al-TM-RE (TM: transition metals; RE: rare earth elements) marginal glass-forming alloys represent an interesting class of metallic glass due to their common observation of a very high density of nuclei embedded in the amorphous matrix. A mechanism to explain such high nanocrystal densities has not been identified. The Al-Sm system was chosen to investigate this nucleation phenomenon in an Al-RE alloy. Both liquid and rapidly solidified structures were analyzed using high energy X-ray diffraction (HEXRD). A medium range order (MRO) structure different from fcc-Al was resolved in the liquid and in as-quenched alloys within composition range where glass formation occurs. Results from a combined study of calorimetry, electron microscopy and HEXRD with RMC (Reverse Monte Carlo) simulations will be presented to discuss the effects of MRO structure on the formation of high nanocrystal density. Research supported by U.S. DOE under Contract No.DE-AC02-07CH11358.

4:20 PM
Microstructure Of Undercooled Al-Fe-Nd Alloy: *Walman Castro*[1]; Benedito Luciano[1]; [1]Universidade Federal de Campina Grande

Rapid Solidification Processing of metals and alloys, is establish by increasing of the undercooling applying high cooling rates (100 - 1000000 K/s) or by reduce nucleation sites using low cooling rates (1 K/s). Melt undercooling opens new solidification pathways for new non-equilibrium phases and unusual microstructures. Several techniques have been developed to reduce nucleation sites and produce increased undercooling in metals and alloys including the fluxing technique. In this work, an Al96Fe2Nd2 alloy was solidified, by fluxing technique, and its microstructure and microhardness investigated as a function of the undercooling level. The increasing undercooling level from 30 K to 109 K promotes change in morphology and on the microhardness of the Al96Fe2Nd2 alloy.

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Laser Aided Direct Metal Deposition of Inconel 625 Superalloy: Solidification Microstructure and Thermal Stability: *Guru Prasad Dinda*[1]; Ashish Dasgupta[2]; Jyoti Mazumder[1]; [1]University of Michigan; [2]Focus: HOPE

Inconel-625 has been widely used for over 50 years in the aerospace, chemical, and marine applications. Direct Metal Deposition is a laser aided rapid manufacturing process for fabricating metal parts directly from CAD models. In the present study, a high-power continuous wave CO2 laser beam was focused on the Inconel-625 substrate to create a melt pool into which the Inconel-625 powder was delivered through a special nozzle carried by an inert gas where the powder streams converge at the same point on the focused laser beam. The solidification microstructure of the laser deposited sample was investigated by optical microscopy, SEM and X-ray diffraction. The as-deposited microstructure is columnar dendrite in nature. However, the orientation of the primary dendrite varies with the direction of the deposition. This paper presents the important concepts necessary for any process control for epitaxial growth and the microstructural stability of the dendritic structure at high temperature.

5:00 PM
Phase Diagram Studies in γ-TiAl Turbine Blade Alloys By In Situ Diffraction Of Synchrotron Radiation: Olga Shuleshova[1]; Dirk Holland-Moritz[2]; *Wolfgang Löser*[1]; [1]IFW Dresden; [2]DLR Köln

Solidification processes and high-temperature phase transformations of Ti-Al-Nb turbine blade alloys with compositions Ti-45 to 54 at.% Al-5 to 10 at.% Nb have been studied in situ using high-energy x-ray diffraction on electromagnetically levitated droplets. The direct determination of the solidification modes in conventional microstructure analysis methods of Ti-Al alloys is normally impeded by the melt reactivity at elevated temperatures and various post-solidification transformations. This has led to discrepancies between existing Ti-Al-Nb phase diagrams. Here it was proved that by Nb addition the β-Ti primary solidification range extends to higher Al contents than in previous assessments. The experimental observations of solidification modes and phase transformation sequences for selected compositions match well with a recent CALPHAD calculation of the Ti-Al-Nb system.

5:20 PM
Near Net Shape Repair And Remanufacturing Of High Value Components Using DMD: *Bhaskar Dutta*[1]; Jyoti Mazumder[1]; Harshad Natu[1]; Guru Dinda[2]; [1]POM Group; [2]University of Michigan

Direct Metal Deposition (DMD) with patented close loop feedback system enables greater control of the deposition process as compared to other deposition processes. Its ability to deposit different material at different pixels with a given height directly from a CAD data opens up a new horizon in the materials processing and allows re-configuration of parts with better properties. This becomes particularly critical for repair and reconstruction of components that desire high quality. The current work focuses on application of DMD for high value components with a special emphasize on the defense applications. The presentation will review the DMD process, its material capabilities with particular emphasize on dissimilar material cladding process and properties of DMD materials. Properties of different engineering alloys under different processing condition as compared to wrought alloys will be presented . Finally, case studies involving reconstruction of expensive components made of Al-alloys and steels will be discussed.

General Abstracts: Light Metals Division: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee, TMS: Energy Committee, TMS: Magnesium Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Anne Kvithyld, SINTEF; Alan Luo, General Motors Corp

Tuesday PM Room: 2004
February 17, 2009 Location: Moscone West Convention Center

Session Chair: Alan Luo, General Motors Corp

2:00 PM
A Homogenization Treatment Study for Twin Roll Cast 3003 and 8006 Aluminium Alloys: *Beril Corlu*[1]; Aziz Dursun[1]; Canan Inel[1]; Murat Dündar[1]; [1]Assan Aluminium R S

High solidification rates in twin roll casting of aluminum alloys results in concentration gradients through the thickness having a supersaturated microstructure with very fine grains at the surface. Homogenization treatment is usually applied at higher gauges in order to remove those microstructural gradients. Current study is aimed at optimizing our homogenization treatment for TRC 3003 and 8006 aluminium alloys by applying a series of homogenization temperatures between 520 and 580°C at different holding times. Surfaces and through thickness microstructure of the specimens were investigated under optical and scanning electron microscope, and the mechanical properties have been discussed accordingly. It is found that required microstructural transformations can not be completed if temperature and time combination of homogenization treatment is not sufficient enough, even after following thermomechanical processes. This in turn results in anisotropy in mechanical properties which causes poor deep drawability and earing formation.

2:20 PM
Aluminum as Windings in Transformers: *Joel Liebesfeld*[1]; [1]James F. Valentine and Assoc Inc

The alloys of aluminum/aluminum wiring that have been used in the past, in certain transformer applications, seemingly have a high fault rate under certain conditions. Specifically, aluminum deployed as windings in step-down transformers that have been part of motive charger systems have faulted in ways that have caused fires. As an electrical investigator, that has examined these devices in fire situations, the faulting apparently results in the breakdown of aluminum with a rise in temperatures as it changes state and causes a sudden load increase on power sources. The explanation and clarification of the ambient/environmental conditions that cause such breakdown and result in catastrophic failures will be the subject of the paper's analysis.

2:40 PM
Carbon Compound as Anode Material Electrode in Super Lithium Ion Capacitor: Jie Li[1]; *Yang Juan*[1]; Yan-Qing Lai[1]; Zhi-An Zhang[1]; Xin Hao[1]; [1]Central South University

Series of carbon compounds as anode materials were prepared for super lithium ion capacitor using graphite and active carbon (AC) as raw materials. Their electrochemical properties were investigated by constant current charge-discharge test. The results showed that the compound anode had good capacitive performance as well as Li-ion battery performance. The potential of the capacitor could be as high as 3.5V vs Li/Li^+ when compared with 2.5V vs Li/Li^+ in the AC-AC capacitor, accordingly, the energy density increased from 21.7Wh/kg to 40.3Wh/kg. The compound anode also had excellent rate performance that as the current density increasing from 0.1A/g to 1A/g,the capacitance decreased only 1.3F/g, and good cycle performance that the capacitance holding remained 96.7% after 10 times cycles even at the highest potential of 3.5 V vs Li/Li^+.

3:00 PM
Comparative Analysis of the Effects of Acidified Vegetable Extracts on the Corrosion Behaviour of Al – Zn Alloy Systems: *Ekuma Chinedu Ekuma*[1]; Ndubuisi Idenyi[2]; Greg Avwiri[1]; Israel Owate[1]; [1]University of Port Harcourt; [2]Ebonyi State University, Abakaliki

The effects of acidified extracts of vernonia amegdalina, pipernigrum and telferia occidentalis on the corrosion behaviour of Al – (1.0%, 2.0% and 3.0%) Zn alloy in varying molarities of HCl has been investigated. Preweighed samples of the alloy were subjected to 0.5M and 1.0M HCl, each containing 50 cm3 of the extracts respectively. The set-ups were allowed to stand for 28 days with a set of samples withdrawn weekly for corrosion rate characterization. The results obtained showed the usual corrosion trend of initial steep rise in corrosion rate followed by a gradual decline over exposure time, characteristic of most passivating metals. Expectedly, the vegetable extracts showed inhibition traits with pronounced effects at lower acid molarities but the inhibition potentials diminished with increase in the reinforcing phase. Comparatively, it was noticed that telferia occidentalis showed the best inhibition potentials followed by pipernigrum while vernonia amegdalina was the worst.

3:20 PM
Homogenized Baking Quality: *Domenico DiLisa*[1]; Hans-Peter Mnikoleiski[1]; Detlef Maiwald[1]; [1]Innovatherm

The quality of a baked anode is defined mainly by the heat treatment. Each anode in a pit has to reach overall a specific temperature for a specific time. The heat transfer is given by the temperature versus time curve of the surrounding flues. Due to physical design of an open top ring furnace less energy is introduced to the outer flues. Further, the pitch burn starts and ends at a later point then at the inner flues. This lack of heat transfer will be compensated at a later stage by the new extended firing index module. In addition to this module a control strategy has been developed to modulate the burner temperature target in correlation to the preheat area temperature development. The result of the improved temperature target modulation is a homogenized baking quality

3:40 PM
Improving the Robotized GMAW Technique for Joining Light Aluminum Extrusions: *Michel Guillot*[1]; Isabelle Bouchard[1]; [1]Laval University

Gaz metal arc welding is commonly used for joining large assemblies. Even for light assemblies, the GMAW technique is increasingly preferred despite limitations. In this paper, several parameters of the GMAW process and robot motion are investigated on joining AL6061-T6 extrusions. For different thicknesses and joint types, are found sets of parameters providing good and repeatable joint quality with minimal distortion and good appearance at relatively high travelling speeds. Simple extruded flat stocks of three thicknesses are tested with parameters like typical GMAW settings, joint preparation geometries, wire gages and alloys, gun positions and angles, robot speeds and accuracy of welding path. The geometric and positioning errors of the components mounted in the fixture are also studied. The repeatability of weld quality is tested. Finally, the best conditions are applied for joining and testing a floor panel made of several extrusions.

4:00 PM
Mechanical Properties of Powder Metallurgy Titanium Alloys Dispersed with Carbon Nano Particles: *Katsuyoshi Kondoh*[1]; Thotsaphon Threrujirapapong[1]; Hisashi Imai[1]; Bunshi Fugetsu[2]; [1]Osaka University [2]Hokkaido University

Titanium powders are uniformly coated with carbon nanotubes (CNTs) and nano-scale carbon black (CB) particles via wet process in using the surfactant solutions, and mixed by ball milling. The titanium composite powder are compacted by spark plasma sintering (SPS) process, and consolidated by hot extrusion. TiC particles synthesized by the reaction of carbon nano particles and titanium powders are necessary to improve mechanical properties of the powder metallurgy (P/M) composite materials. In this study, SPS temperature is decided by high-temperature XRD analysis which detects the synthesis of TiC during heating. When using pure titanium powders and SPS temperature of 1073K, their composite material with 1% CNTs shows UTS of 810 MPa and 21% elongation, which are superior to the wrought material including no CNT with 585MPa UTS and 30% elongation. The uniform distribution of both of CNTs and TiC particles is remarkably effective for the improvement of their mechanical properties.

4:20 PM
The Effect of Trace Boron Addition on Grain Growth Kinetics of As-Cast Beta21S: *Balakrishna Cherukuri*[1]; Raghavan Srinivasan[1]; Sesh Tamarisakandala[2]; Daniel Miracle[3]; [1]Wright State University [2]FMW Composite Systems Inc; [3]US Air Force

Several primary ingot breakdown steps are typically carried out to reduce the as-cast grain size and thus improve processability / workability in titanium alloys. Obtaining finer grain size in as-cast condition would eliminate / reduce processing steps and thus result in cost savings. Addition of boron in trace amounts to Beta21S (Ti-15Mo-2.6Nb-3Al-0.2Si) resulted in significant grain refinement in the as-cast condition. The presence of TiB particles along the grain boundaries in the boron modified alloy restricts the grain boundary mobility at elevated temperatures, thus providing microstructural stability during hot working. The grain growth exponents and activation energies were calculated to study the effect of trace boron additions on the kinetics of grain growth. The effect of volume fraction, aspect ratio and orientation of the TiB particles with respect to the grain boundary are modeled using modified Zener pinnig criteria. Results show good correlation between the predictions and experimental results.

4:40 PM
Microstructural Uniformity in AZ31B and its Impact on Further Processing and Properties: *David Foley*[1]; Majid Al-Maharbi[1]; Ibrahim Karaman[1]; Laszlo Kecskes[2]; Suveen Mathaudhu[2]; Karl Hartwig[1]; [1]Texas A & M University [2]U S Army Research

Recently there has been building interest in improving the strength and formability of Mg alloys by severe plastic deformation (SPD) via equal channel angular extrusion. While improvements have been made to commercial alloys by this technique, our experience has shown that chemical and microstructural uniformity can play a key role in improving the material by SPD. This study examines AZ31B starting with cast and rolled forms. For a given low-temperature strain and route, we demonstrate the influence of initial heterogeneity on post-processing microstructure and mechanical properties. Issues such as mechanical property variability and shear banding are compared to material subjected to a prior homogenization.

5:00 PM
Processing Response of Boron Modified Ti-6Al-4V Alloy: Microstructure and Texture Evolution during (a+ß)-Working: *Shibayan Roy*[1]; Satyam Suwas[1]; S Tamirisakandala[2]; R Srinivasan[3]; D.B. Miracle[4]; [1]Indian Institute of Science; [2]FMW Composite Systems, Inc.; [3]Wright State University; [4]Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB

Titanium alloys e.g. Ti-6Al-4V are the backbone materials for aerospace, energy, and chemical industries. Hypoeutectic boron addition to Ti-6Al-4V alloy produces a reduction in as-cast grain size by roughly an order of magnitude resulting in the possibility of avoiding ingot breakdown step and thereby reducing the processing cost. In the present study, ISM processed as-cast boron added Ti-6Al-4V alloy showed identical, although refined microstructure and largely randomized/weak texture compared to normal Ti-6Al-4V alloy. When deformed in (a+ß)-phase field, a-lath bending and globularization seemed to be the dominating deformation mechanisms at lower and higher temperatures, respectively for both these materials. Wherein normal Ti-6Al-4V showed flow instabilities (cavitations, cracking etc.) at low temperatures, boron added materials possessed enhanced workability under all processing conditions. The texture evolved during deformation was, however, completely different in these two kinds of alloys, suggesting a difference in the operating deformation mode that produces the end stable orientations.

5:20 PM
Production of the Composite from 6082 Al Alloy Chips and Fly Ash Particles by Hot Pressing: Harun Mindivan[1]; Huseyin Cimenoglu[2]; *Eyup Kayali*[2]; [1]Ataturk University; [2]Istanbul Technical University

In the present experimental investigation, 6082 Al alloy machining chips as matrix material and up to 20 wt% of fly ash particulate composite was fabricated using hot pressing method. Characterization of the composites was made by structural examinations and mechanical tests (hardness and dry sliding wear tests). The results indicate that the wear resistance of the fly ash reinforced composite increased with increase in fly ash content and the composites reinforced with fly ash particle exhibited superior hardness and wear resistance compared to the unreinforced 6082 Al alloy.

5:40 PM
Selenium Treatment Technologies: *Karen Hagelstein*[1]; [1]TIMES Limited

This paper discusses recent selenium treatment technologies which have been applied to metal processing, mining, agricultural and other industrial wastewaters. Physical, chemical, and biological properties of selenium compounds are discussed as well as analytical procedures. The removal technologies include membrane processes, ion exchange, chemical precipitation, adsorption processes, and biologically-based technologies such as biological treatment plants, in-situ treatment, constructed wetlands, evaporation ponds, Biopass and other passive technologies. Some technologies require management of the residues from treatment, such as brines or wash solutions from membrane filtration and ion exchange, or sludges resulting from iron precipitation. Best management practices for seleniferous wastes including pilot testing, process chemistry, source control, and water management are discussed relative to industry case studies. The selenium treatment technologies which have been tested at a pilot-scale facility, implemented as treatment processes, are cost effective for large volumes of wastewater and can potentially meet regulatory treatment objectives are highlighted.

6:00 PM
Surface Chemical Analyses of the Anti-Wear Boundary Film on Oxygen-Diffusion Treated Titanium: *Jun Qu*[1]; Harry Meyer[1]; [1]Oak Ridge National Laboratory

Previous work has demonstrated that oxygen diffusion (OD) can dramatically improve the tribological characteristics for titanium alloys by enabling the formation of an anti-wear boundary film in a lubricated environment. In this study, XPS surface chemical analyses were conducted to reveal the film chemical compositions and compounds. The 50 nm thick boundary film detected on the worn OD-treated titanium (OD-Ti) surface contains significant amount of Ti, Zn, Ca, S, P, N, and O, while no such film exists on either the unworn OD-Ti surface or the worn, untreated titanium surface. Shifts of bonding energy spectrum peaks for elements extracted from the lubricant additives as well as the elements from the OD-Ti surface imply that the additive molecules reacted with the OD-Ti surface and formed new compounds during the wear process. Further analyses using an Auger parameter plot for Zn confirmed the existence of ZnS and ZnO in the boundary film.

Magnesium Technology 2009: Alloys II: Rare Earth (Cerium and Other)

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

Tuesday PM — Room: 2006
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Mark Easton, Monash University

2:00 PM Introductory Comments

2:05 PM
Microstructure and Mechanical Properties of an Mg-Rare Earth Based Alloy AM-SC1: *Mark Gibson*[1]; Colleen Bettles[2]; Suming Zhu[3]; Mark Easton[3]; Jain-Feng Nie[3]; [1]CSIRO; [2]ARC Centre of Excellence for Design in Light Metals; [3]CAST CRC

AM-SC1 is a high temperature Mg alloy that was originally developed as a sand casting alloy for automotive powertrain applications. In order to be truly competitive in critical automotive applications, magnesium alloys need to have properties that are similar to, or better than, aluminium alloy counterparts and they need to be readily manufacturable. AM-SC1 is suitable for both sand casting and permanent mould casting with properties that have been proven for both the AVL Genios LE and the USCAR lightweight magnesium engine projects. This paper describes the interrelationship between the microstructure and the mechanical properties of AM-SC1. The microstructural features contributing to the creep resistance are both inter- and intra-granular in nature and are on length scales from nanometres to micrometres. The creep behaviour at 150°C and 177°C is diffusion controlled, with any contribution from the grain boundaries being negligible.

2:25 PM
Interdependence between Cooling Rate, Microstructure and Porosity in Mg Alloy AE42: *Liang Wang*[1]; Sergio Felicelli[2]; Adrian Sabau[3]; John Berry[2]; [1]Mississippi State University ; [2]Mississippi State University; [3]Oak Ridge National Laboratory

Porosity is a major concern in the production of light metal parts. This work aims to identify some of the mechanisms of microporosity formation during the gravity-poured castings of magnesium alloy AE42. Two graphite plate molds and a ceramic cylindrical mold were selected to produce a wide range of cooling rates. Temperature data during cooling was acquired with type K thermocouples at 60 Hz in two locations of each casting. The microstructure of samples extracted from the regions of measured temperature was then characterized with x-ray computed tomography and optical metallography. The gathered data was analyzed to search for correlations between cooling rate, dendrite arm spacing, pore volume fraction and pore size. The experimental outcomes were compared with simulations performed with a finite element continuum model of dendritic solidification. The results of this study confirm some of the findings observed in similarly cast aluminum alloys.

2:45 PM
Application of Neutron Diffraction in In-Situ Studies of Stress Evolution in High Temperature Creep Testing of Creep Resistant Magnesium Alloys: *Dimitry Sediako*[1]; [1]National Research Council of Canada

In-Situ Studies of Stress Evolution in magnesium alloys developed for high temperature applications have been the major focus of this study. Several alloying groups have been analyzed representing Mg-Al-Rare Earth, Mg-Al-Sr, Mg-Al-Ca, and Mg-Zn-Rare Earth systems. The samples were cast in permanent mould and extruded, and then subjected to 200 hrs of creep under load of 50 MPa for duration of 200 hours. Two temperatures were used in the creep tests: 150°C and 175°C. Primary and secondary creep evolution was observed for the studied alloys. In-situ and creep-induced residual stresses were analyzed with application of neutron diffraction techniques at the Canadian Neutron Beam Centre in Chalk River, Ontario. The in-situ diffraction pattern clearly shows the consecutive responses of the crystallographic lattice to the sample heating, creep loading, unloading, and cooling. Correlation of the resultant elongation of the sample (creep) to residual stress has also been demonstrated for several crystallographic planes.

3:05 PM
Influence of Rare Earth Elements on the Microstructure and Texture Development during Rolling of Magnesium Alloy Sheets: *Jason Hadorn*[1]; Kerstin Hantzsche[2]; Joachim Wendt[3]; Karl Kainer[3]; Jan Bohlen[2]; Dietmar Letzig[2]; Sean Agnew[1]; [1]University of Virginia; [2]GKSS-Forschungszentrum; [3]Hamburg University of Technology

The addition of rare earth elements has been observed to weaken and alter the predominant basal texture which normally occurs in conventional rolled magnesium-zinc alloy sheets. This effect offers the possibility to alter the mechanical properties and, in particular, improve the formability. Preliminary results have suggested that the “rare earth effect” is due to changes in the recrystallization behaviour of the alloys. Results from laboratory rolling trials designed to examine the effects of solid solution alloying with zinc and rare earth elements on the microstructural development will be presented. X-ray diffraction is used to characterize the bulk texture evolution. Electron backscattered diffraction (EBSD) is used to reveal the unique nucleation and growth patterns during recrystallization. Transmission electron microscopy is used to examine the level of grain boundary solute segregation. Finally, the impact of the resulting microstructure and texture on mechanical properties will be shown and discussed.

3:25 PM
The Intergranular Microstructure of Magnesium Based Die-Cast AE Alloys: *Liu-Ying Wei*[1]; Kun Wei[1]; Richard Warren[1]; [1]Malmö University

A systematic investigation on microstructures of magnesium based die cast Mg-Al-RE alloys (AE alloys) has been performed by XRD, SEM and TEM. The alloys are with content of Al around 4wt% and various content of rare earth (RE). Samples of these alloys are in as-cast condition as well as in aged condition at 200°C and 250°C. The intergranular microstructure of the alloys has been studied in details. Three types' binary Al-RE phases were found intergranularly.

Al11RE3 is predominant intergranular phase in the as cast alloys. Al3RE particles and small amount of Al2RE phase were found in alloys with high RE content. Thermal stability of the Al-RE phases in AE alloys was suggested to decrease in sequence: Al2RE —> Al11RE3 —> Al3RE. The Al/RE ratio of the die cast alloys determined their phase constitutions. Promising AE alloys for creep resistance is suggested to have an Al/RE ratio not higher than 1.8.

3:45 PM Break

Magnesium Technology 2009: Alloys III: Rare Earth (Gadolinium, Neodymium)

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

Tuesday PM Room: 2006
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Liming Peng, Shanghai Jiaotong University; Karl Kainer, GKSS Research Center

4:00 PM Introductory Comments

4:05 PM
Characterization of Dynamic Strain Ageing in Mg-3.11wt.%Gd Alloy: Lei Gao[1]; *Rongshi Chen*[1]; Enhou Han[1]; [1]Institute of Metal Research Chinese Academy of Sciences

To elucidate the dynamic strain ageing behavior of Mg-Gd based alloys which were developed as high specific strength and good creep resistant magnesium alloys at elevated temperature, tensile tests were carried out for Mg-3.11wt.%Gd in the temperature range of 25-300°C and in the strain rate range from 1×10^{-4} to 1×10^{-2} s^{-1}. At given strains, stress relaxation (SR) experiments were performed. Serrated flow, negative strain rate sensitivity, and post-relaxation effect were observed in some cases. The post-relaxation effect was sensitive to testing temperature and the strain at which the stress relaxation was performed. The critical strain for the onset of serrated flow was observed to increase with increasing strain rate but decrease with increasing temperature. In addition, activation energy for serrated flow was calculated. The results were analysed in relation to dynamic strain ageing effect (DSA) due to interactions between dislocations and solute Gd atoms.

4:25 PM
Effect of Cold Roll on Microstructure and Mechanical Properties of Mg-8Gd-3Y-0.5Zr Alloy: *Li Dejiang*[1]; Zeng Xiaoqin[1]; Dong Jie[1]; Zhai Chuanquan[1]; [1]Shanghai Jiao Tong University

The simplest TMT process including cold roll with strain of 8%,15%,22% and subsequently aged at different temperatures to peak hardness were carried out to investigate the influence on microstructure and mechanical properties of heat resistant Mg-8Gd-3Y-0.5Zr alloy. The microstructure observation showed that basal plane dislocation sliding and twinnings (including double twinning) were the main deformation mechanisms during cold rolling, the amount of twins were increased with increasing deformation strain. The initial hardness of the alloy specimen was increased with the increasing of strain and the aging time to peak hardness was greatly shortened for the reason of work hardening and acceleration of precipitation from the supersaturated solid solution, respectively. However, the peak hardness value of the deformed and non-deformed alloy specimens remained almost the same. TEM investigation confirmed that the precipitation in the deformed microstructure preference for the equilibrium phase was attributed to lower age hardening response.

4:45 PM
Mechanical Properties and Creep Behavior of Mg-10Y-5Gd-0.5Zr Alloy: Yan Gao[1]; *Qudong Wang*[1]; Jinhai Gu[2]; Yang Zhao[1]; [1]Shanghai Jiaotong University; [2]Hitachi (China) Research & Development Corp., Shanghai Research Institute

We have investigated the microstructure, mechanical properties at room and elevated temperatures (250°C, 300°C), strengthening mechanisms, creep behavior and creep deformation mechanisms of Mg-10Y-5Gd-0.5Zr alloys of in the cast and T6 conditions. The results showed that the tensile properties of the cast-T6 specimen are much higher than that of the as-cast specimen and the creep resistance of the cast-T6 specimen is markedly better than that of the as-cast specimen at both conditions. The creep resistance of the Mg-10Y-5Gd-0.5Zr at T=250, σ=80MPa is markedly better than that at T=300, σ=50MPa. This means the temperature makes more effects on the creep resistance than the stress. Finally, the creep mechanism of the alloy at different condition is further analyzed.

5:05 PM
The Fracture Behavior of B2 Magnesium-Rare Earth Intermetallics: *Rupalee Mulay*[1]; James Wollmershauser[1]; Sean Agnew[1]; [1]University of Virginia

Intermetallic compounds have many attractive properties. The main issue, which limits the practical application of these compounds, is that most are brittle at room temperature. Recently, however, a family of binary intermetallic compounds has been discovered which exhibit appreciable polycrystalline ductility. These compounds are composed of a rare earth metal and a main group or transition metal and they have the B2 crystal structure. We are presently investigating the possibility that B2 compounds composed of Magnesium and a rare earth element may also exhibit the ductilizing effect. The compounds we are studying are MgY, MgCe, MgNd and MgDy. We have discovered that these compounds are brittle, and have undergone primarily cleavage fracture. We have used a combination of SEM-based stereology (to determine the facet normal) and EBSD (to determine the grain orientation) to, in turn, characterize the cleavage plane crystallography. Results indicate that MgY tends to cleave along {100} planes.

5:25 PM
On Microstructure and Texture Development of RE-Containing Magnesium Alloys: *Joachim Wendt*[1]; Karl Ulrich Kainer[1]; Gurutze Arruebarrena[2]; Kerstin Hantzsche[3]; Jan Bohlen[3]; Dietmar Letzig[3]; [1]Hamburg University of Technology; [2]Mondragon Goi Eskola Politeknikoa; [3]GKSS-Forschungszentrum GmbH

The usage of Magnesium sheets as light-weight structures is limited due to their poor formability - in particular at room temperature - resulting from the typical strong basal texture of commercial magnesium sheets. Recently, it was shown that the basal texture may be weakened by the addition of rare earth (RE) or other alloying elements. The responsible mechanisms, however, are not yet well understood. In the presented work a series of experiments has been performed to study in more detail the influence of RE-elements on the texture development during hot rolling. The experiments are performed in several rolling passes with subsequent heat treatment. The paper presents texture and microstructure development over the whole rolling process and shows the distinct influence of the rolling procedure and a subsequent heat treatment. The research provides basic knowledge for targeted modification of the alloy composition aiming at enhanced formability of wrought magnesium alloys.

5:45 PM
Effects of Solid Solution Heat Treatment on the Microstructures and Mechanical Properties of Mg96.82Zn1Gd2Zr0.18 Alloy with 14H-Type LPSO Structure: Y.J. Wu[1]; *X.Q. Zeng*[1]; D.L. Lin[1]; L. M. Peng[1]; W.J. Ding[1]; [1]Shanghai Jiao Tong University

In as-cast Mg96.82Zn1Gd2Zr0.18 alloy, the microstructure mainly consists of eutectic structure in which the second phase, ß-phase, is (Mg,Zn)3Gd having fcc structure, and α'-Mg solid solution and fine-lamellae consisting of 2H-Mg and 14H-type long period stacking ordered (LPSO) structure. The LPSO structure has been firstly observed in as-cast Mg96.82Zn1Gd2Zr0.18 alloys. During solid solution heat treatment at 773K for 0.5h to 234h, the microstructure evolution, especially the evolution of 14H-type LPSO structure were studied. It is concluded that ß-phase can transform into a novel lamellar X-phase with 14H-type LPSO structure. Furthermore, it is concluded that the alloy heat-treated at 773K for 10h exhibits higher tensile strength (246.21MPa) and larger elongation (11.79%) owing to the dissolution of the ß-phase and the much dispersion of X-phase with 14H-type LPSO structure and the lamellar 14H-type LPSO structure in matrix.

6:05 PM
Microstructure and Mechanical Properties of Hot Extruded Mg-3Nd-0.2Zn-0.4Zr (wt. %) Alloy: Penghuai Fu[1]; *Liming Peng*[1]; [1]National Engineering Research Center of Light Alloy Net Forming, School of Materials Science and Engineering, Shanghai Jiaotong University

The microstructure and mechanical properties of 350, 450 and 525°C hot extruded Mg-3Nd-0.2Zn-0.4Zr (NZ30K) (wt. %) alloys are investigated. The grains are significantly refined by hot extrusion and the extruded alloys show a bimodal grain distribution, in which the finer grains are less than 1µm and the coarser grains are several to ten micrometers in size. The lower extrusion temperature, the finer grains are got. The extruded NZ30K alloys have aging hardening ability. The higher extrusion temperature, the higher aging hardening effect ΔHV (HV peak-aged – HV as-extruded) is obtained. After aging treatment, both yield strength (YS) and ultimate tensile strength (UTS) are significantly improved. The 350°C extruded NZ30K alloy shows the best strength after 200°C peak-age treatment. The YS, UTS and elongation are 290MPa, 317MPa and 22%, respectively. The extruded alloys show dimple fracture pattern characterized by dimples covered all of fracture surfaces.

Materials for High Temperature Applications: Next Generation Superalloys and Beyond: Refractory Alloys II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS: Refractory Metals Committee
Program Organizers: Joseph Rigney, GE Aviation; Omer Dogan, National Energy Technology Laboratory; Donna Ballard, Air Force Research Laboratory; Shiela Woodard, Pratt & Whitney

Tuesday PM Room: 3010
February 17, 2009 Location: Moscone West Convention Center

Session Chair: Bernard Bewlay, GE Global Research (K1-MB271)

2:00 PM Invited
Microstructural Designs for High Temperature Mo-Si-B Alloys: *John Perepezko*[1]; Ridwan Sakidja[1]; Megan Jarosinski[1]; [1]University of Wisconsin

While existing high temperature alloys exhibit a remarkable performance, the prospects are limited for advances in high-temperature capability (>1400°C). Among the new systems that can enable a step change in performance, multiphase Mo-Si-B alloys are attractive in meeting the difficult challenges of high temperature performance. Systematic studies of the phase equilibria and diffusion have provided a foundation of understanding of the governing phase stability and microstructure evolution. The baseline microstructure with a Mo +Mo3Si + Mo5SiB2 (T2) phase mixture provides a good performance, but solidification processing is difficult. An alternate approach based upon alloy design is also available. Phase stability guidance allows the Mo3Si phase to be replaced by a Mo5Si3 (T1) structure and provides access to a monovariant three-phase BCC + T2 + T1 eutectic reaction. Similarly, other alloy designs yield a BCC + T2 +D88 phase. The microstructure designs provide new directions for processing and new performance levels.

2:25 PM
Deformation Behavior of Mo_5SiB_2: *Oleg Kontsevoi*[1]; Nadezhda Medvedeva[2]; Arthur Freeman[1]; John Perepezko[3]; [1]Northwestern University [2]Institute of Solid State Chemistry; [3]University of Wisconsin

Multiphase Mo-Si-B alloys attract increasing attention as very promising materials for applications at temperatures above 1200°C; poor ductility is one of the main drawbacks. To analyze the fracture and deformation mechanisms for Mo_5SiB_2, we performed ab initio calculations of generalized stacking fault energies for possible directions on the [001], {010}, {110} and {012} slip planes. A striking result was obtained that the three favorable systems, <100>(001), <110>(001) and [001]{010}, have almost equal unstable and stable stacking faults, and the preference among them cannot be established. This finding explains a large variety of experimental data on the observed slip systems. The dislocations associated with these slips may dissociate into partials joined with stacking faults and separated by the large splitting width of 5-6 nm, as estimated from elasticity theory. The absence of a strong preference for a certain slip system suggests a path for enhancing ductility through operation of multiple systems.

2:45 PM
Compressive Deformation Behavior of High Temperature Mo-Si-B Alloy: *Xingshuo Wen*[1]; Padam Jain[2]; Joachim Schneibel[3]; K.Sharvan Kumar[2]; Vijay Vasudevan[1]; [1]University of Cincinnati; [2]Brown University; [3]Oak Ridge National Laboratory

Alloys based on Mo-Si-B ternary system are of interest for very high temperature structural applications. The compression behavior at 1200, 1300 and 1400°C of a nominally Mo-20Si-10B (in wt.%) alloy that was processed such as to yield varying α-Mo volume fractions (from 5 to 46%), with the balance made up of Mo_3Si and T2-Mo_5SiB_2 phases, was studied. The results of constant strain rate compression tests showed that the stresses required to maintain a given strain rate increased with a decrease in temperature and α-Mo volume fraction. The values of the stress exponents determined from the data ranged from ~3-9, depending on temperature and volume fraction of α-Mo; the activation energy for creep was found to be in the range of ~200-600 kJ/mole depending on stress level and volume fraction of α-Mo. These results were correlated with SEM and TEM observations of the damage processes, deformation structures and deformation mechanisms.

3:05 PM
Fabrication and High Temperature Stability of a $(Mo,Cr)Si_2$-Based Composite: *Erik Ström*[1]; Mats Sundberg[1]; Yiming Yao[2]; Changhai Li[2]; Xinhai Li[3]; Karin Lindqvist[4]; [1]Kanthal; [2]Chalmers University of Technology; [3]Siemens Industrial Turbomachinery AB; [4]Swerea IVF

$MoSi_2$ is an interesting intermetallic compound to be used as a potential structural material, because of its high melting point, excellent oxidation resistance and low density. However, it is brittle at low temperatures and has relatively low creep resistance. It has been recognized that these problems can be alleviated by using $MoSi_2$ as a composite matrix. $MoSi_2$-ZrO_2 composites exhibited a monotonic increase in fracture toughness with increasing volume fraction of reinforcement. Especially, unstabilized ZrO_2 particles resulted in the highest room temperature fracture toughness, reaching a level three times that of pure $MoSi_2$. But, high volume percentage of reinforcement had a negative effect on oxidation resistance. To improve oxidation resistance of $MoSi_2$-composites while maintaining high toughness, Cr was added to the $MoSi_2$-matrix. After oxidation at 1400°C for 100 hours, a dense oxide layer was formed on the specimen surface. Hardness and toughness seemed relatively stable after high temperature exposure.

3:25 PM Invited
Structural Molybdenum Borosilicides: Processing as the Key for an Optimum Balance of Properties: *Martin Heilmaier*[1]; Manja Krüger[1]; Holger Saage[1]; Pascal Jehanno[2]; Mike Böning[2]; Heinrich Kestler[2]; Joachim Schneibel[3]; Easo George[3]; [1]Otto Von Guericke University [2]Plansee SE; [3]Oak Ridge National Laboratory

We review the current development status of molybdenum borosilicide (Mo-Si-B) alloys for ultra-high temperature applications in excess of 1100°C in air. The assessment of several ingot and powder metallurgy approaches revealed that (i) the presence of a continuous Mo solid solution matrix is crucial for adequate fracture toughness near ambient temperatures and (ii) wrought processing of such alloys at temperatures typical for refractory metals requires an ultrafine (sub-micron) microstructure. Both prerequisites could be fulfilled using mechanical alloying (MA) as the crucial processing step [1]. However, the ductile-to-brittle transition temperature (DBTT), 800°C, was high due to grain boundary embrittlement by Si segregation. First results on the effect of different microalloying additions (e.g. Zr) on reducing this segregation will be presented and discussed. A short outlook on current industrial activities closes the presentation. [1] M. Krüger et al., Intermetallics 16, 933 (2008).

3:50 PM Break

4:00 PM
CALPHAD Based Thermodynamic Modeling of the Mo-W-Si-C System: *Sujoy Kar*[1]; Swetha Ganeshan[1]; Don Lipkin[2]; Martin Morra[2]; [1]GE Global Research, Bangalore; [2]GE Global Research, Niskayuna

Composite refractory systems based on SiC with Mo and W silicides have been found suitable for high temperature applications in both oxidizing as well as partially reducing atmospheres. Understanding the phase stability in the Mo-W-Si-C system is critical for optimizing the composition and processing of these alloys for specific applications. A CALPHAD based technique has been employed to develop the Mo-W-Si-C quaternary thermodynamic database. Prior literature in this system does not reveal any ternary compounds in three of the four constituent ternary systems: Mo-W-Si, Mo-W-C, and W-Si-C. This indicates the sufficiency of the ideal solution assumptions for the respective ternary interaction parameters. However, it has been reported that the Mo-Si-C system has a ternary compound, namely the Nowotny phase ($Mo_{5-x}Si_3C_y$). Consequently, the Mo-Si-C ternary database has been assessed. Details of the database development and examples of phase stability in these alloy systems are described.

4:20 PM
Microstructural Engineering of Mo-Si-B Alloys Produced Using Nitride-Based Reactions: *Michael Middlemas*[1]; Joe Cochran[1]; Arun Gokhale[1]; [1]Georgia Institute of Technology

Mo-Si-B intermetallic composite alloys are of interest as next-generation, high-temperature materials. Three-phase alloys consisting of bcc-Mo and the intermetallic phases Mo_3Si and Mo_5SiB_2 have been investigated. The intermetallic phases enhance creep strength and oxidation resistance, but hinder fracture toughness due to crack propagation through the brittle intermetallics. For good mechanical properties, the intermetallic phases must be present as a fine dispersion in a continuous molybdenum matrix. This has been achieved using a powder metallurgy approach through the reaction of molybdenum, Si_3N_4 and BN powders. The effect of different boron nitride reactant powders on the dispersion of the intermetallic phases has been investigated. Electron backscatter diffraction imaging has been used to map the location of individual phases. Two-point correlation functions were used to quantify microstructural parameters in order to examine the effect of processing on the resulting microstructure.

4:40 PM
The High Temperature Oxidation Behavior of a $MoAl_xSi_{2-x}$ - Based Composite: *L. Ingemarsson*[1]; M. Halvarsson[2]; M. Sundberg[3]; L.-G. Johansson[1]; J.-E. Svensson[1]; Erik Ström; [1]High Temperature Corrosion Centre (HTC), Chalmers University of Technology; [2]Department of Applied Physics, Chalmers University of Technology; [3]Kanthal AB, Hallstahammar, Sweden

It is well known that the intermetallic compound $MoSi_2$ exhibits excellent oxidation properties at high temperatures (1000-1700°C) due to the formation of a protective SiO_2 scale, making it a promising material for high temperature applications. However, at low temperatures (400-600°C) $MoSi_2$ may suffer from accelerated oxidation and/or pesting. Also, in reducing environments the SiO_2 scale is decomposed and $MoSi_2$ is susceptible to high temperature corrosion. To avoid this problem it is possible to use a composite material $MoAl_xSi_{2-x}$ (Kanthal Super ER) which forms a protective alumina scale. This work focuses on the oxidation properties of the $MoAl_xSi_{2-x}$ composite in an oxidizing atmosphere at temperatures between 1100-1500°C. The oxidation has been studied using isothermal furnace exposures and the morphology and composition of the oxide scale were analyzed with scanning electron microscopy (SEM), energy dispersive x-ray analysis (EDX) and X-ray diffraction (XRD). The results are linked to the oxidation mechanisms.

5:00 PM
Rapid Synthesis and Consolidation of Nanostructured WSi2-SiC from Mechanically Activated Powders by Pulsed Current Activated Heating: In-Jin Shon[1]; *Jeong-Hwan Park*[2]; Kee-Do Woo[2]; Jin-Kook Yoon[3]; [1]Division of Advanced Materials Engineering, the Research Center of Industrial Technology, Chonbuk National University; [2]Division of Advanced Materials Engineering and the Research Center of Industrial Technology, Engineering College, Chonbuk National University; [3]Advanced Functional Materials Research Center, Korea Institute of Science and Technology

WSi2 has an attractive combination of properties, including high melting temperature, high modulus, high oxidation resistance in air, and a relatively low density. To improve on its mechanical properties, the approach commonly utilized has been the addition of a second phase to form composite and to make nanostructured materials. Dense nanostructured WSi2-SiC composite was synthesized by pulsed current activated heating within 2 minute in one step from mechanically activated powders of WC and 3Si. Highly dense WSi2-SiC with relative density of up to 99.9% was simultaneously synthesized and consolidated under simultaneous application of a 80 MPa pressure and the pulsed current. The average grain sizes of WSi2 and SiC were about 47 nm and 38 nm, respectively. The average hardness and fracture toughness values obtained were 1698 kg/mm2 and 4.8 MPa•m1/2, respectively. The present fracture toughness and hardness are higher than those(3.3 MPa·m1/2, 1375 Kg/mm2) of monolithic WSi2.

Materials in Clean Power Systems IV: Clean Coal-, Hydrogen Based-Technologies, and Fuel Cells: Hydrogen Storage Materials

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Harvesting and Storage Committee
Program Organizers: K. Scott Weil, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; Ayyakkannu Manivannan, US DOE; Z. Gary Yang, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University Zi-Kui Liu, Pennsylvania State University

Tuesday PM — Room: 3005
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Zhenguo "Gary" Yang, Pacific Northwest National Laboratory

2:00 PM Introductory Comments

2:05 PM Keynote
High Capacity Hydrogen Storage Based on Solid Amine Boranes: *Chris Aardahl*[1]; [1]PNNL

In the current vision of the hydrogen economy, fuel cell vehicles using some form hydrogen fuel will replace automobiles relying on gasoline powered internal combustion engines. A wide range of candidate on-board hydrogen storage methods are being evaluated including pressurized hydrogen gas tanks, liquefied hydrogen, and a host carriers from which hydrogen gas can be desorbed and regenerted. A portion of the current research within the US DOE Center of Excellence for Chemical Hydrogen Storage focuses on solid ammonia borane (AB). Ammonia borane and its derivatives are promising hydrogen storage materials because they contain large fractions of releasable hydrogen with reasonable kinetics. The results discussed in this presentation will cover release of hydrogen from these materials as well as the chemical regeneration required to recycle the fuel. Technical focus will be on release mechanism,how to increase kinetics, and on novel approaches for reducing H-depleted boron centers to enable fuel regeneration.

2:50 PM Invited
Enhancing The Hydrogen Storage Capacity Of Nanoporous Carbons: *Nidia Gallego*[1]; Cristian Contescu[1]; Vinay Bhat[1]; [1]Oak Ridge National Laboratory

Efficient storage of hydrogen for use in fuel cell-powered vehicles is a challenge that is being addressed in different ways, including adsorptive, compressive, and liquid storage approaches. In this presentation we report on adsorptive storage in Palladium-doped nanoporous carbon fibers. Nanoparticles of Pd, when dispersed in activated carbon fibers (ACF), enhance the hydrogen storage capacity of ACF. The adsorption capacity of Pd-ACF increases with increasing temperature below 0.4 bar, and the trend reverses when the pressure increases. To understand the cause for such behavior, hydrogen uptake properties of Pd with different degrees of Pd-carbon contact are compared with Pd-sponge using in situ XRD under various hydrogen partial pressures (<10 bar). The results support the spillover mechanism (dissociative adsorption of H2 followed by surface diffusion of atomic H). Research sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy under contract with UT-Battelle, LLC.

3:25 PM
Structural Phase Transitions Under Pressure in $Mg(BH_4)_2$: *Lyci George*[1]; Vadym Drozd[1]; Maximilian Fichtner[2]; Surendra Saxena[1]; [1]Florida International University [2]Forschungszentrum Karlsruhe GmbH

The structural stability of $Mg(BH_4)_2$, a promising hydrogen storage material, under pressure has been investigated up to 21 GPa with combined synchrotron X-ray diffraction and Raman spectroscopy. The analyses show a structural phase transition around ~ 3.5-5.4 GPa and again around ~14 GPa. At ambient conditions $Mg(BH_4)_2$ has a hexagonal structure (space group-P61, a=10.1647(6) Å, c= 36.902(8) Å and V= 3301.9(8) Å ^{3}), which agrees well with early reports. The high pressure phase is found to have different structure from theoretically determined structures; the structure also does not match with that of the high temperature phase. The high pressure phase is found to be stable on decompression similar to the case of high temperature phase.

TUESDAY PM

3:45 PM
An Investigation of Hydrogen Capacity of Magnesium Powder: *Hung-Yu Tien*[1]; Mahesh Tanniru[1]; Chang-Yu Wu[2]; Fereshteh Ebrahimi[1]; [1]Materials Science and Engineering, University of Florida; [2]Environmental Engineering Science, University of Florida

Ideally magnesium hydride has a total capacity of 7.6wt%. However, during hydrogenation of magnesium powders the saturation level achieved is lower than this maximum capacity. In this study the kinetics of hydrogenation of a commercial magnesium powder is investigated as functions of temperature, pressure and hydrogenation procedure. Ni was applied as catalyst using two different techniques. The nucleation and growth of magnesium hydride were characterized using cross-sectional electron microscopy. The results of this study revealed that the hydrogenation kinetics and the saturation level depend on the sequence of heating and pressurization. When the powder was heated after pressurization a lower saturation level was obtained. Furthermore, the dry method of applying Ni was found to render faster hydrogenation kinetics because of the additional change in the morphology of the powder. In this study mechanism of hydrogenation is discussed based on microstructural evaluation. The financial support by NSF (DMR-0605406) is greatly appreciated.

4:05 PM Break

4:10 PM Invited
Synthesis Of Alkali Amidoboranes For Hydrogen Production: *Xiong Zhitao*[1]; Wu Guotao[1]; Chen Ping[1]; [1]Dalian Institute of Chemical Physics

Holding a hydrogen capacity of 19.6wt%, ammonia borane (NH3BH3) shows potential to be a hydrogen storage material. Though considerable amount of hydrogen evolves from NH3BH3, its decomposition also generates borazine, a volatile product highly poisoning to PEM fuel cell. In addition, relatively high kinetic barrier in dehydrogenation of this chemical hinders the release of hydrogen at acceptably low temperatures. Our recent effort in modifying NH3BH3 by replacing one of its H with alkali metal successfully produced lithium amidoborane (LiNH2BH3) and NaNH2BH3. TPD and volumetric release showed amidoboranes evolved 2 equiv. H2 in two steps; a burst was observed around 90C and a broad TPD signal centralized at 150C. Borazine was undetectable. DSC indicated mild exothermic nature of that burst and the second dehydrogenation process was nearly thermal neutral. Therefore, isothermally heating LiNH2BH3 and NaNH2BH3 at 91 degree C for 19h resulted in the evolution of 10.7wt% and 7.4wt% hydrogen, respectively.

4:45 PM
Nano Approaches To Use Light Metals Magnesium And Aluminum In Hydrogen Storage: *Rajeev Ahuja*[1]; [1]Uppsala University

A fundamental understanding of the role of catalysts in dehydrogenation of MgH2 nanoclusters is provided by carrying out first principles calculations based on density functional theory. It is shown that the transition metal atoms Ti, V, Fe, and Ni not only lower desorption energies significantly but also continue to attract at least four hydrogen atoms even when the total hydrogen content of the cluster decreases. In particular, Fe is found to migrate from the surface sites to the interior sites during the dehydrogenation process, releasing more hydrogen as it diffuses. This diffusion mechanism may account for the fact that a small amount of catalysts is sufficient to improve the kinetics of MgH2, which is essential for the use of this material for hydrogen storage in fuel-cell applications. Further, we have also studied the role of catalysts in dehydrogenation of AlH3.

5:05 PM
Effect Of Al Addition On Dehydrogenation Characteristics Of MgH_2: *Mahesh Tanniru*[1]; Jacob Jones[1]; Darlene Slattery[2]; Fereshteh Ebrahimi[1]; [1]University of Florida; [2]Florida Solar Energy Center

Magnesium hydride, which has a theoretical hydrogen capacity of about 7.6wt%, is an attractive hydrogen storage material for fuel cell applications. The main shortcoming of employing MgH_2 is its high desorption temperature. Alloying has been shown to affect the stability of this hydride. In this study the effect of Al addition on the dehydrogenation characteristics of magnesium hydride was investigated. Mg-Al alloy powders were fabricated by an electrodeposition technique. Ni was added as a catalyst for promoting the hydrogenation of these alloy powders. Electron microscopy techniques were employed for microstructural and compositional analyses. Pressure-composition isotherms were developed at different temperatures to evaluate the enthalpy of formation/dissociation of MgH_2. The evolution of phases during the dehydrogenation was investigated using an in-situ high temperature x-ray diffractometer. In this presentation the effects of Al addition on the stability of MgH_2 will be discussed.The financial support by NSF (DMR-0605406) is greatly appreciated.

5:25 PM
Preparation of TiFeMn Hydrogen Storage Alloy by SOM Method: *Chonghe Li*[1]; Xiaosu Ye[1]; [1]Shanghai University

In this study, a new Solid Oxygen-ion Membrane (SOM) process is introduced to produce TiFeMn hydrogen storage alloys directly from metal oxides by electrolysis reduction. The short process flow decreases the alloy's cost significantly. Compared with FFC, SOM method possesses many advantages, including faster electrolysis speed, higher current efficiency, and no side reactions etc. If hydrogen fills into the anode, the product of anode will become water vapor, which means this method may be real environment friendly. In order to speed up the electrolysis process and diminish barrier of reduced metal for further electrolysis, which usually occurs for SOM, the preparation process of polar plate in secondary battery industry is borrowed. This cathode consists of foamed nickel employed to be the cathode matrix and oxides particle adhered to the matrix, the foamed nickel could enlarge the electrolysis area. This improved cathode structure makes the electrolysis process faster and more downright.

5:45 PM
The Hydrogen Storage Behaviours Of Nanocrystalline And Amorphous $Mg_{20-x}La_xNi_{10}$(x=0-6) Alloys Prepared By Melt-Spinning: *Huiping Ren*[1]; Baowei Li[1]; Zaiguang Pang[1]; Yanghuan Zhang[1]; [1]Inner Mongolia University of Science and Technology

The Mg_2Ni-type $Mg_{20-x}La_xNi_{10}$ (x= 0, 2, 4, 6) hydrogen storage alloys were prepared by melt-spinning technology. The microstructures and hydrogen storage performances as well as thermal stabilities of the alloys were studied in detail. The results shows that the no amorphous phase forms in the as-spun La-free alloy, but the as-spun alloys containing La hold a major amorphous phase, confirming that the substitution of La for Mg significantly heightens the glass forming ability of the Mg_2Ni-type alloy. Melt-spinning significantly improves the hydrogen storage behaviours of the alloys. When the spinning rate increases from 0 (As-cast was defined as spinning rate of 0 m/s) to 30 m/s, the hydrogen absorption capacity of the alloys (x = 2) at 200°C and 1.5 MPa in 10 min rises from 1.26 to 2.60 wt%, and its discharge capacity rises from 197.23 to 406.5 mAh/g at a current density of 20 mA/g.

Materials Issues in Additive Powder-Based Manufacturing Processes: Coatings and Deposition

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Powder Materials Committee
Program Organizers: David Bourell, University of Texas; James Sears, South Dakota School of Mines and Technology; Pavan Suri, Mississippi State University

Tuesday PM Room: 3004
February 17, 2009 Location: Moscone West Convention Center

Session Chair: Pavan Suri, Heraeus MTD

2:00 PM
Issue Involved With Applying WC MMC's to Tooling Surfaces through Laser Additive Manufacturing: *James Sears*[1]; Casey Bergstrom[1]; [1]South Dakota School of Mines and Technology

Laser Additive Manufacturing (LAM) of tungsten carbide metal matrix composites (MMCs) has been evaluated for surface modification of: hot die forming tools, cutting edges, glass tooling, extrusion mandrels, and other abrasive wear applications. This work is focused on transitions from tool steel (H-13) to these MMCs through a single pass laser powder deposition operation. Issues related to the application of various metal powders and carbides used include: surface hardness, porosity, cracking and dilution. These issues along with factory results that were obtained during this project are discussed. This work was performed under a grant from the U.S. Department of Energy (DOE), Office of Industrial Technology under contract DE-PS07-03ID14425: Industrial Materials for the Future Program.

2:25 PM

Combined Analyses of TEM and Atom Probe Tomography for Superfine WC-Co Coatings: *Nam Suk Lim*[1]; Seong Yong Park[2]; Chan Gyung Park[3]; [1]POSTECH; [2]The University of Texas at Dallas; [3]POSTECH and NCNT

The microstructure of WC-Co coatings fabricated by using superfine(0.1~0.5μm) carbide particles and detonation gun spraying method has been investigated. In order to find exact phase and unique 3D distribution of constituent elements, ultra high-resolution analysis using transmission electron microscope (TEM) and 3 dimensional atom probe tomography (3D-APT) has been performed. The analytical samples of WC-Co powders and coatings were made by using the focused ion beam (FIB).The microstructure of WC-Co coating layers fabricated with superfine carbides was identified as the combined phases of unmelted, partially melted and fully melted regions. TEM results revealed clearly that WC phase was decomposed crystalline W2C, W phase and complex amorphous phase, due to an exposure of carbide particles to high temperature detonation flame during the spraying and rapid quenching followed. The results will be compared with the APT results of distribution of constituent elements in each phase.

2:50 PM

Cold Sprayed Aluminum and Aluminum Alloy Coatings: *Shaodong Wang*[1]; Lijue Xue[1]; Jiaren Jiang[1]; [1]NRC-IMI (London)

A downstream radial powder injection cold spray process has been developed and validated using aluminum-based (Al, Al-12Si and Al-7075) powders. Numerical simulation is performed to simulate the gas flow and particle movement behavior in the spray nozzle. Particle velocities at the nozzle exit were measured using an optical diagnostic method. The numerical simulation agrees well with the particle velocity measurement results. Microstructures and microhardness of the aluminum coatings deposited on Al-6061 aluminum substrates were investigated and were compared with results from the literature using upstream axial powder injection cold spray systems.

3:15 PM

High Performance Titanium Osteoconductive Coatings for Medical Implant Applications: James Sears[1]; Dana Medlin[1]; *Jacob Fuerst*[1]; [1]South Dakota School of Mines and Technology

Laser Additive Manufacturing (LAM) is being evaluated for an improved methodology for creating a grid surface coating on osteoconductive implants. Titanium powder was directed into a small molten bead on a Titanium substrate produced with a focused laser operating from 200 to 350 watts with a beam diameter of 600 microns in an inert Ar environment. Evaluation of the deposition showed near 50% void formation within 100% dense grid structure. Metallurgical analysis shows low total heat input and fast bead cooling rate result in a fine microstructure in the grid structure with a very thin heat affected zone in the substrate. Developments with finer powders (20 to 50 microns) and a narrower beam (100 microns) result in increased void area and a finer grid structure. The formation of titanium interlocking structures with this technology has also been explored.

3:40 PM Break

4:05 PM

Electrically Insulating Phosphate Coatings for Iron Powder Based Electromagnetic Core Applications: W. Rane Nolan[1]; Francis Hanejko[2]; Howard Rutz[2]; *Mitra Taheri*[3]; [1]Massachusetts Institute of Technology; [2]Hoeganaes Corporation; [3]Drexel University

Powdered metals, such as iron, are a common building block for electromagnetic cores. An iron powder was reacted with phosphoric acid to create an electrically insulating iron phosphate layer on each particle, which could lead to significant reductions in eddy current losses in alternating current applications. The electromagnetic properties of this phosphate-coated powder material were examined as a function of heat treatment. Additionally, SEM and EDS were used analyze the particle interfaces and composition in compressed bar-shape samples that were heat treated at temperatures ranging from 315°C to 540°C. Initial results show that after high temperature treatments (required for stress reduction, sintering, increased magnetic permeability, and decreased coercivity), the bulk resistivity is reduced. Correlation of structure and composition with trends in resistivity is discussed. Ultimately, our analysis will aid in the development of high temperature coatings with ideal properties for electromagnetic core applications.

4:30 PM

Effect of Oxide Dopants on Densification, Microstructure and Mechanical Properties of Alumina-Mullite Ceramic Foams Prepared by the Polymeric Sponge Method: *Xiao Jin*[1]; Feng Zhou[1]; Song-yun Deng[1]; Jie Li[1]; Ye-xiang Liu[1]; [1]School of Metallurgical Science and Engineering, Central South University

The densification behaviour, microstructural development and mechanical properties of the ceramic foams were studied by four different oxide dopants. It was found that CaO, MgO,Y2O3 and CeO2 enhanced the densification of foams, which can be explained by a liquid phase assisted sintering process. The calcium oxide and magnesium oxide doped ceramic foam could be sintered at 1350°C. Doping with MgO, Y2O3 or CeO2 refines the grain size of the matrix alumina-mullite whereas calcium oxide addition induces exaggerated grain growth. The fracture hardness, compressive strength, thermal shock resistance were evaluated. Doped CaO exhibit lower fracture hardness and thermal shock resistance values, but doping with MgO, Y2O3 and CeO2 exhibit higher fracture hardness, compressive strength, thermal shock resistance values than that of the undoped foams. CeO2 doping slightly improves fracture hardness and thermal shock resistance lower than Y2O3 dopings whereas the improvement in compressive strength was 1.2 times than the other oxides.

4:55 PM

Influence Of Processing Conditions On The Microstructure And Mechanical Properties Of A Polycrystalline Nickel Alloy Created By Powder Bed Laser Deposition: Xinhua Wu[1]; Fude Wang[1]; *Daniel Clark*[2]; [1]University of Birmingham; [2]Rolls-Royce Plc

Developments in powder bed laser deposition process capability enable structures with efficient material utilisation in response to service loading. Systematic research has been carried on the influence of processing conditions on microstructure and mechanical properties of a polycrystalline nickel alloy, which was direct laser melted (DLMed) via a laser powder bed machine. The processing parameters: laser power, scan speed, scan spacing and nominal laser power density, were studied. Assessment was made of: density, dimension accuracy, surface roughness, cracking and top-surface concavity across a range of processing conditions. Tensile samples were tested in order to assess the difference in mechanical properties induced by the difference in build location. Some DLMed samples were HIPped(hot isostatic pressed). It has been found that nominal laser power density has the dominating effects on the above characteristics however the influence of scan spacing and scan speed can sometimes be significant on some of those characteristics.

5:20 PM

Uniform-Droplet Spray Forming of $Mg_{97}Zn_1Y_2$ Alloy: *Hiroki Fukuda*[1]; Pengtao Wang[2]; Hongwei Sun[2]; Peter Wong[3]; Charalabos Doumanidis[4]; Teiichi Ando[1]; [1]Northeastern University [2]University of Massachusetts-Lowell; [3]Tufts University; [4]University of Cyprus

A $Mg_{97}Zn_1Y_2$ alloy was spray-deposited using uniform (mono-size) droplet sprays produced by the controlled capillary jet breakup. Consisting of mono-size droplets of desired diameter chosen in the range of 500 - 1000 μm, and no fine, pyrophoric droplets, the uniform droplet sprays permitted stringent control of the thermal state of depositing droplets while assuring safe spraying. The solidification of traveling droplets was simulated with a model that accounted for the rapid crystallization during recalescence. A level-set-method based numerical model was used to simulate the deformation and solidification of a depositing droplet. Mg-Zn-Y deposits produced under optimal spraying conditions had a uniform microstructure consisting of equiaxed grains of hcp Mg-rich solid solution and $Mg_{12}ZnY$ precipitates. Subsequent rolling resulted in further microstructural refinement and improved mechanical properties.

Materials Processing Fundamentals: Smelting and Refining

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

Tuesday PM Room: 2016
February 17, 2009 Location: Moscone West Convention Center

Session Chair: K. Morsi, San Diego State University

2:30 PM
Boron Production Via Molten Salt Electrolysis: *Judith Gomez*[1]; Patrick Taylor[1]; Edgar Vidal[2]; [1]Colorado School of Mines; [2]Brush Wellman, Inc.

Boron is an element that is difficult to isolate in its pure form and requires a substantial amount of energy. Typically, boron is synthesized by metallothermic reduction of its oxide with magnesium or sodium; reaction of boron halogenides with hydrogen; or, thermal decomposition of diborane. An alternative method using molten salt electrolysis has been evaluated in this work. A mixture of MgF_2-NaF-LiF-B_2O_3 salts was molten under an inert atmosphere, and the effect of different experimental parameters such as temperature, potential and current density were evaluated. Characterization of the material deposited and efficiency of the process is presented, as well as conclusions and recommendations for future work.

2:45 PM
XRF Anlysis of EAF and LMF Type Slags: *Alexander Seyfarth*[1]; Dan Pecard[1]; [1]Bruker AXS Inc.

Talk and manuscript will detail a case study based tutorial on how LMF and EAF slags are sampled, prepared and analyzed by XRF. We will compare the applicability of both ED and WD XRF. Data derived from the analysis can then be used to optimize the SLAG composition using different models.We will present and compare the casy study of an EDX Benchtop system at the furnace directly operated by the meltshop vs. a laboratory based WD XRF system operated by the QC group. This is aimed to teach the application of XRF and it's limitations to enable discussion and application of results to the process.

3:00 PM
Effect of EAF Slag Carryover on Slag-Metal Equilibrium Calculations for Ladle Degassing Process: *Hamid Doostmohammadi*[1]; Margareta Andersson[1]; Karin Steneholm[2]; Pär Jönsson[1]; [1]KTH, Royal Institute of Technology; [2]Uddeholm Tooling AB

During the tapping of liquid steel from Electric Arc Furnace (EAF), some slag is carried over into the ladle. High levels of FeO and MnO in slag carryover increase the oxygen activity in steel melt leading to oxide inclusion formation during the ladle treatment. The demand on cleaner steels requires minimization of carryover slag. In this work the effect of EAF slag carryover on ladle slag-steel equilibrium calculations for a hot working tool steel was studied. Steel and slag sampling were done at Uddeholm Tooling AB in Sweden. XRF and OES techniques were used to determine chemical composition of samples. The quantity of slag carryover was calculated by mass balance followed by thermodynamic calculations on metal-slag equilibrium for vacuum degassing process using Thermo-Calc software. The agreement with lab analysis will lead to the development of a prediction method for optimizing the production of other tool steel grades.

3:15 PM
Effects of Stoichiometry on Boron Carbide Production via Self Propagating High Temperature Synthesis: *Murat Alkan*[1]; Bora Derin[1]; Seref Sonmez[1]; Onuralp Yucel[1]; [1]Istanbul Technical University

In this study, a self-propagating high-temperature synthesis (SHS) and following acid leaching techniques were carried out to produce boron carbide (B4C) powder. First, B2O3 was obtained through calcinations of H3BO3 at 1073 K for 2 hours. Then, the glassy B2O3 obtained was crushed and sieved. In the SHS experiments, different amounts of B2O3, Mg and C black were used. The SHS product was obtained in the form of black, spongy solid. In the leaching step, the SHS product was leached in a HCl solution to eliminate the MgO and Mg3B2O6. The effect of acid concentration on the selective leaching was studied at different concentration range, solid/liquid (S/L) ratio, and temperature. The products obtained were characterised by using X-ray diffraction, chemical analysis and SEM techniques.

3:30 PM
Corrosion Resistance of High-Alumina Graphite Based Refractories to the Smelting Reduction Melts: *Qingcai Liu*[1]; [1]Chongqing University

The corrosion resistance and microstructure of Al2O3-C based refractories in smelting reduction melts were studied and evaluated by the quasi-stationary immersion and rotary immersion test. The corrosion rate of the Al2O3-C based refractories is decreased with the addition of the graphite carbon and ZrO2. The test results showed that the ZrO2 containing bricks had much better corrosion resistance than the ZrO2-free bricks. The ZrO2 addition improved the oxidization resistance of the refractory and decreased the interaction rate between the melts and the refractory. The corrosion of the Al2O3-C based refractories is caused by both the interaction between melts and refractory and the dissolution of the refractory constituents into the melts.

3:45 PM
Process and Practice of EAF with De-P Hot Metal Charging for Melting Stainless Steel: *Fangyi Zhu*[1]; [1]Baosteel

The article introduce the process of Electric Arc Furnace with Dephosphorized hot metal charging for melting stainless steel in Baosteel stainless steel Branch. Based on the practice of production, The main factors affecting the process of EAF with De-P HM charging are theoretically analyzed, such as using oxygen, the material charging and making slag. The optimization of hot metal charging can advance the use of chemical and physical energy, reduce the consumption of power. The optimization of using oxygen can increase the use of chemical energy. The optimization of material charging can reduce the oxidation of Cr. Making foamy slag can advance the transformer capacity and the use of power. Based on the character of the process EAF with De-P HM charging for Melting Stainless Steel, EAF productivity increased were reached with application of integrated control theory on EAF process in Baosteel stainless steel branch.

4:00 PM Break

4:15 PM
Study On A New Electrolysis Technology Of Preparing High-Nb Bearing TiAl Alloy From Metal Oxides: Fanke Meng[1]; *Huimin Lu*[1]; [1]Beijing University of Aeronautics and Astronautics

Electrolyzing oxides of titanium, aluminum and niobium could prepare high-Nb bearing TiAl alloy(Ti-45Al-8.5Nb) in the temperature between 1700°C and 1800°C. The chosen electrolysis voltage ranges from 2.3V to 2.5V, which is lower than reducing voltage (~4.7V) of electrolyte CaF2, but higher than that of titanium, aluminum and niobium oxides. The investigation shows that compared with traditional methods, this method for preparing high-Nb bearing TiAl alloy is of many advantages such as few steps, friendly environment, low energy consumption, low cost and high current efficiency.

4:30 PM
A Study On Nickel Containing Iron Alloy Production From West Anatolian Region Lateritic Ores: *Cem Colakoglu*[1]; Bora Derin[1]; Onuralp Yucel[1]; [1]Istanbul Technical University

In this study, nickel containing iron alloys were produced by Carbothermal reduction of lateritic nickel ores obtained from Manisa -Caldag region of Turkiye. In the experiments, the lateritic ore containing 1.58 % Ni and % 0.12 % Co was first calcined in a semi-pilot scale rotary furnace at 1100°C for 1 hour. Then, a mixture of calcined ore and metallurgical grade charcoal was reacted in sintered alumina crucibles using a graphite resistance tube furnace at different time (0- 60 min) and temperature (1450-1600°C). The raw materials, alloys and slags were characterized by using wet chemical analyses, XRD (X-Ray Diffractometry), and EPMA (Electron Probe Micro Analyzer) techniques.

4:45 PM
Reduction Kinetics of Iron Ore Pellet Used for Corex C-3000: *Nan Wang*[1]; [1]Northeastern University

In order to provide the basic kinetics parameters for the numerical simulation of the pre-reduction shaft furnace of Corex C-3000 in Baoshan Steel Iron and Steel Corporation, China, investigations into the reduction kinetics of iron ore pellet (CVRD pellet) were conducted under the conditions of H2-CO-CO2 mixture gas and 750~850° temperature, and the reaction kinetic parameters were determined based on the thermogravimetric experimental data. The experimental results indicated that the reduction rate increased with the increment of hydrogen content and temperature, and the reduction reaction of CVRD ore pellet was mainly controlled by both chemical reaction at the oxide/iron interface and diffusion through porous product layer. Consequently, the activation energy values of CVRD ore pellet were also estimated.

5:00 PM
Manufacture of High Nitrogen Austenitic Stainless Steels by Pressurized Electro-Slag Remelting: *Jiang Zhouhua*[1]; Cao Yang[1]; Li Huabing[1]; [1]Northeastern University

The attempt has been made to manufacture high nitrogen austenitic stainless steels by pressurized electro-slag remelting furnace with maximum nitrogen pressure of 7MPa. To obtain high nitrogen content in steels, the compound electrodes with different nitrogen sources (FeCrN, Si3N4) were prepared. Using the Si3N4 as the nitrogen alloying source, the silicon contents in ingots were prone to be out of the specification range, and the electric current fluctuated greatly and the surface qualities of the ingots were poor. The surface qualities of the ingots were improved using FeCrN as nitrogen alloying source. The sound and compact macrostructure ingot with the maximum nitrogen above 1.0wt% could be obtained. A series of high nitrogen austenitic stainless steels have been successfully developed by this method. The mechanical and pitting corrosion properties of high nitrogen austenitic stainless steels were investigated. The results show that the steels exhibit excellent mechanical and pitting corrosion resistance properties.

5:15 PM
An Efficient Method of Stirring Melt with a Modulated Traveling Magnetic Field: *Xiaodong Wang*[1]; Rene Moreau[2]; Yves Fautrelle[2]; [1]McGill University; [2]EPM-SiMAP-CNRS

This study examines a liquid GaInSn metal flow generated by a magnetic field whose traveling direction is periodically reversed. Ultrasonic Doppler velocimetry probes the generated fluid flow. Depending on the modulation frequency, a number of characteristic flow features are exhibited. A transition frequency (~4 Hz) exists, below this transition point, the amplitude of the velocity oscillation increases and below fm = 0.05 Hz, it saturates around a value close to that observed without any modulation. The role of this electromagnetically driven flow is to transport the solute rejected by the solidifying interface at significant distances in the melt, and to periodically reverse its circulation such that macro-segregation is minimized. An analytical electromagnetic model and a flow dynamic model for the recirculating fluid flow are derived, assuming that in this central region the flow is quasi-parallel to the main axis. The analytical velocity field achieves a satisfactory agreement with the measurements.

5:30 PM
Carbothermic Reduction of Ilmenite Concentrate: Chengjun Gao[1]; Na Hou[1]; *Hongmin Zhu*[1]; [1]Beijing University of Science and Technology

Carbon thermo-reduction of ilmenite were performed with various ratio of carbon, at temperatures up to 1600°C. The thermodynamic possibility of selective carbothermic reduction was discussed in detail. Experiments for testing the reduction order of the oxides of iron, titanium, silicon, and magnesium were carried out. The products were characterized by X-ray diffraction, scanning electron microscope and chemical analysis. The results showed that it is possible to reduce all iron oxide to metal, and titania to titanium carbide(TiC), or oxicarbide(TiC_xO_y), without the reduction of silica and magnesia. The separation of the reduction product of TiC_xO_y-Fe-MO_x was also carried out.

Mechanical Behavior of Nanostructured Materials: Strengthening Mechanisms at Small Length Scale

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: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Xinghang Zhang, Texas A & M University Andrew Minor, Lawrence Berkeley National Laboratory; Xiaodong Li, University of South Carolina; Nathan Mara, Los Alamos National Laboratory; Yuntian Zhu, North Carolina State University; Rui Huang, University of Texas, Austin

Tuesday PM   Room: 3024
February 17, 2009   Location: Moscone West Convention Center

Session Chairs: Rui Huang, University of Texas; Thomas Buchheit, Sandia National Laboratories

2:00 PM Invited
Mechanical Properties of Nanocrystalline Thin Films: Daniel S. Gianola[1]; Yixiang Gan[2]; Tim Rupert[3]; John Sharon[4]; *Kevin Hemker*[4]; [1]Forschungszentrum Karlsruhe, Institute for Materials Research II ; [2]Forschungszentrum Karlsruhe, Institute for Materials Research II; [3]Massachusetts Institute of Technology; [4]Johns Hopkins University

Microtensile testing techniques have been developed and employed to characterize the small-scale and scale-specific mechanical behavior of materials for MEMS and nanocrystalline thin films. Geometric concentrators can be used to introduce spatial variations in the stress and strain states in these films, providing insight on how deformation is accommodated. Combining microstructural analysis with continuum descriptions of stress and strain gradients allows one to decouple the effect of stress and strain on deformation processes, however, use of multiple stress concentrators will lead to wrinkling and must be accounted for. Here we describe experiments and finite element simulations of freestanding submicron Al thin films with patterned interior holes. Stretching these films induces periodic and symmetric wrinkling that is imaged directly during tensile testing and faithfully reproduced by the simulations. An example will be presented using these techniques that elucidated the mechanisms of mechanical grain growth in nanocrystalline Al thin films.

2:20 PM
Characterization of Nanostructured Films Synthesized by High Power Pulse Ion Ablation: *Thomas Buchheit*[1]; Timothy Renk[1]; Somuri Prasad[1]; Paul Kotula[1]; [1]Sandia National Laboratories

A unique Repetitive High Energy Pulse Power (RHEPP-1) facility at Sandia National Laboratories offers the capability for deposition of low stress multilayer films with layer thicknesses less than 10 nm. The facility generates an intense ion-beam of 10 J/cm2 that ablates alternating targets to synthesize the films, with a wide range of multilayer films possible. Driven by a previous study focused on MoS2-Ti nano-laminate films for tribological applications, this investigation broadened the scope of deposited Mo-based nanolayered films. Targets were chosen to produce Mo-bearing laminates with a wide range of characteristics, although particular focus was directed toward choosing target materials with a range of differing shear modulii. Nanolaminate films investigated include Mo-Ir, Mo-Ti, and Mo-W. Each film contained at least one hundred alternating layers and all were compared against a uniform Mo deposition. Subsequent material and mechanical characterization revealed series of hard, well-ordered, semi-crystalline bilayer films. Details will be discussed.

2:35 PM
Temperature Dependence Of Mechanical Properties In Ultra Thin Au Films With And Without Passivation: *Patric Gruber*[1]; Sven Olliges[2]; Eduard Arzt[3]; Ralph Spolenak[2]; Oliver Kraft[4]; [1]Universität Karlsruhe, Institut für Zuverlässigkeit von Bauteilen und Systemen; [2]ETH Zurich, Laboratory for Nanometallurgy, Department of Materials; [3]INM Leibniz Institute for New Materials; [4]Forschungszentrum Karlsruhe, Institut für Materialforschung II

Mechanical testing of thin metallic films at elevated temperatures is difficult. Here, we present a systematic study of the mechanical properties of 80 to 500 nm thick polycrystalline Au films with and without SiNx passivation layers in the temperature range from 123 to 523 K. The films have been tested by a novel synchrotron-based tensile testing technique. All film systems show a very strong temperature dependence of the flow stress. For passivated Au films the temperature dependence of the flow stress can be rationalized by thermally activated dislocation glide. For pure Au films on polyimide, an analysis on the basis of a model for grain-boundary diffusional creep of a freestanding foil gives values for the activation energy of 0.3 to 0.6 eV, which indicates that diffusional creep is strongly pronounced in these films. This is remarkable because the testing temperatures are at most 0.3 of the homologous temperature.

2:50 PM Invited

Evolution Of Intrinsic Stresses In Thin Film Growth Via Coupled Surface And Grain Boundary Diffusion: Tanmay Bhandakkar[1]; Eric Chason[1]; *Huajian Gao*[1]; [1]Brown University

In order to explain experimental observations on the evolution of intrinsic stresses during Vomer-Weber growth of thin metal films of high surface mobility, Chason et al. (2002) have proposed a model based on the assumption that a higher chemical potential near the film surface during deposition provides a driving force for a flow of adatoms into the grain boundaries (GB). Here we build upon the previous models of Gao et al. (Acta Mat, 1999), Chason et al. (PRL, 2002) and Guduru et al. (JMPS, 2003) on coupled surface and GB diffusion and extend these models to include the effect of GB diffusion heterogeneity. Our model considers a layer of active diffusion near the surface during deposition. Inside the active layer, both surface and GB diffusivities are assumed to be higher than their normal values. The simulation results show excellent comparisons with experimentally measured stress evolution in various films.

3:10 PM Invited

Nanoscale Deformation In Multilayered Nanocomposite Thin Films: In- Situ And Ex-Situ TEM Analyses: *Jeff DeHosson*[1]; Changqiang Chen[1]; Yutao Pei[1]; [1]University of Groningen

Although high-resolution transmission electron microscopy is the most direct method to observe the effects of a mechanical response at nanometer scales, a common problem encountered in the HRTEM examination of deformed amorphous or uniformly nanostructured materials is the lack of intrinsic markers tracing microstructural evolutions. We will demonstrate that nanocystallites may serve as perfect interior markers for distinguishing various deformation patterns in MeC/DLC nanocomposite coatings.This paper concentrates on the nanoindentation behavior and corresponding deformation mechanisms of the nanocomposite coatings under depth sensing indentation, using combined ex situ nanoindentation followed by XTEM and also in situ TEM nanoindentations. Through the examination of the displacement of the nanocrystallites an interparticle deformation process involving rearrangement of TiC nanocrystallites and displacement of the DLC a-C matrix is demonstrated that dominates the deformation mechanism at length scales ranging from tens of nm down to 1 nm.

3:30 PM

Microstructure – Property Relationships in TiN-based Coatings on Steel Substrates Prepared by Pulsed Laser Deposition: Andreas Jahja[1]; *Paul Munroe*[1]; [1]University of New South Wales

A range of sub-micron thick TiN coatings were deposited on a H13 hot worked tool steel substrate via pulsed laser deposition as a function of processing conditions. The coatings were subject to detailed microstructural characterization, including FIB and cross-sectional TEM studies. Coatings prepared at high substrate temperatures (450ºC) and reactive gas conditions exhibited very fine nanoscale grain sizes, whilst slightly coarser structures were prepared in inert environments. Mechanical behaviour was assessed through nanoindentation using a spherical indenter. The coatings exhibited high hardness values and significant resistance to cracking, even at high loads. Examination of the indented layers revealed intercolumnar cracks within the TiN coatings, together with shear steps at the coating-substrate interface, whilst inclined cracks were observed at the periphery of the indentations.

3:45 PM Break

3:55 PM Invited

Deformation of Nanotube Arrays for Contact Switches in MEMS: *David Bahr*[1]; Ryan Johnson[1]; [1]Washington State University

The properties of large assemblages of CNTs are not controlled by the individual tubes, but by the collective topological behavior of the "turf", consisting of many CNTs attached to an inflexible substrate. This presentation focuses on a range of experimental efforts using nanoindentation (including ECR) and compression testing in situ in an SEM to assess the properties of turfs. The stress required to form a collective buckle structure in the turf is dependant only on the ratio of tangent modulus to applied stress, and not the aspect ratio of the structures. Adhesion to diamond is strong and metallic coatings dramatically reduce the adhesion between the CNTs and diamond indenter tip. The results will be used to demonstrate a low temperature thermocompression bonding technique that demonstrates the flexibility of these materials. The CNT data will be contrasted to silica nanowires and springs, which follow more macroscopic models of fiber based deformation.

4:15 PM

Interfacial Fracture in Scandium Deuteride Films from Micro to Nano Scales: Marian Kennedy[1]; *Neville Moody*[2]; David Adams[2]; E. David Reedy[2]; Nancy Yang[2]; David Bahr[3]; [1]Clemson University; [2]Sandia National Laboratories; [3]Washington State University

Performance and reliability are extremely important issues for scandium deuteride films used in neutron tube applications where high residual stresses during processing can lead to premature failure. As a result, we have begun a program combining small volume property and thin film fracture tests to determine fracture susceptibility in these film systems. Samples were fabricated following a two-step procedure to create films on fused silica monitors that ranged in thickness from 150 nm to 6 μm with a uniform grain size of 400 nm. On cool-down, high thermal mismatch led to spontaneous delamination in the thick films at an interfacial fracture energy near 4 J/m2. Surprisingly these values matched four point bend results on the thinnest films tested. In this presentation, we will discuss how structure, properties, and stress affect interfacial fracture in these films from micro to nanoscales. This work supported by Sandia National Laboratories under USDOE contract DE-AC0494AL85000.

4:30 PM

Mechanical Behavior Of Single-Layer Graphene: Qiang Lu[1]; *Rui Huang*[1]; [1]University of Texas at Austin

The unique structure and properties of single-layer graphene has drawn tremendous interests recently. This paper presents a theoretical study of the mechanical behavior of graphene and associated morphological structures. By combining atomistic and continuum modeling, lattice deformation of graphene sheets under both in-plane forces and bending moments is analyzed. The model predicts a nonlinear and anisotropic mechanical behavior of graphene under large strains. Using a two-atom unit cell, the theoretical strength of single-layer graphene under macroscopically homogeneous in-plane deformation is investigated. Heterogeneous deformation with characteristic strain localization is observed in large-scale atomistic modeling. It is found that the bending properties of single-layer graphene are fundamentally different from those predicted by continuum plate or shell models. Furthermore, a buckling instability is predicted by atomistic simulations for graphene sheets under compression or shearing, leading to a periodic morphology that depends on the size and boundary conditions.

4:45 PM

Nano-Scale Tribology Of Polycrystalline Silicon Structural Films In Ambient Air: *Daan Hein Alsem*[1]; Ruben van der Hulst[2]; Eric Stach[3]; Michael Dugger[4]; Jeff DeHosson[2]; Robert Ritchie[5]; [1]Lawrence Berkeley National Laboratory; [2]University of Groningen; [3]Purdue University; [4]Sandia National Laboratories; [5]University of California, Berkeley

Dynamic coefficients of friction (COF), nano-scale wear volumes and morphology have been studied for polysilicon MEMS (Sandia SUMMiT V) in ambient air at different relative humidity (%RH). Half of the devices show an increase in the COF by a factor of three with increasing number of wear cycles with failure after ~10e5 cycles. The other half of the devices displayed similar behavior, but after peaking reached a lower steady-state COF showing no failure after millions of cycles. In this regime increasing the %RH resulted in a linear increase of the COF. Additionally, the wear coefficient and surface roughness sharply increased in the first ~10e5 cycles and then decayed to a lower value over several million cycles. Electron microscopy shows that abrasive wear is the governing mechanism, and failures are being attributed to differences in local surface morphology. Re-oxidation of worn polysilicon only affects the friction coefficient after periods of inactivity.

5:00 PM Invited
In-Situ Atomic Scale Nanomechanics Enabled by a TEM-SPM Platform: *Jianyu Huang*[1]; [1]Sandia National Laboratory

By using a sharp scanning tunneling microscopy (STM) probe integrated into a transmission electron microscopy (TEM), in-situ atomic scale nano mechanical studies can be achieved. In this talk, I will review our recent progress in using a TEM-STM platform to probe the atomic scale deformation mechanisms of carbon nanotubes and nanowires. It is postulated that nanotubes accommodate no plastic deformation even beyond the elastic limit or before breakage at room temperatures. I report here our recent discoveries of plastic deformation, as characterized by the superplastic elongation, kink motion, and dislocation climb, in carbon nanotubes at about 2000 °C. These discoveries indicate that there are rich nanomechanics in carbon nanotubes at high temperatures. I will also discuss our progress in using the TEM-SPM platform to probe the mechanical properties of nanowires, and designing a MEMS platform to enable in-situ thermal and thermoelectric measurements of carbon nanotubes and nanowires.

5:20 PM
A Novel Method for Preparing Nanoscale Tensile Test Specimens for In-Situ TEM Tensile Testing: Kai-Ming Ho[1]; Kristen Constant[1]; *Alan Russell*[1]; Wai Leung[1]; Joong Park[1]; [1]Iowa State University

Currently available methods to fabricate nanoscale compression and tensile testing specimens are often time-consuming techniques that carry the risk of introducing impurity atoms or dislocations into the specimen. A new technique for producing in-situ TEM tensile test specimens is being developed that uses a lithographic patterning method to rapidly produce filaments with typical dimensions of 100 nm by 400 nm by 1 cm. Such small sample dimensions allow modeling, (e.g., three-dimensional dislocation dynamics) to be validated by experiment. Results will be presented from preliminary TEM tensile tests performed on several pure metals and intermetallic compounds, including nanocrystalline and amorphous specimens.

5:35 PM
In-Situ Nanomechanical-Electrical Testing of One-Dimensional Materials: *Reza Shahbazian Yassar*[1]; Chee Lee[1]; Jiesheng Wang[1]; Yoke Yap[1]; [1]Michigan Technological University

One-dimensional nanomaterials including nanotubes are building blocks for constructing various complex nanodevices. Boron nitride (BN) nanotubes with structure similar to carbon nanotube are known to have the highest mechanical strength among the insulators. In this work, deformation of an individual BN nanotube is performed inside a high-resolution transmission electron microscope (TEM) using a piezo-driven atomic force microscope (AFM) and scanning tunneling microscope (STM)–TEM holder. The electrical and mechanical properties of individual BN nanotubes are obtained from the experimentally recorded I-V and force-displacement curves.

5:50 PM
Young's Modulus Measurement of Alkaline Earth Metal Hexaboride Nanowires with Atomic Force Acoustic Microscopy: *Xiaoxia Wu*[1]; Terry Xu[1]; [1]University of North Carolina Charlotte

Young's moduli of alkaline earth metal hexaboride (MB6; M = Ca, Sr and Ba) nanowires, a new group of one-dimensional nanostructures for thermoelectric energy conversion, was studied by Atomic Force Acoustic Microscopy (AFAM). The AFAM, a promising technique for nondestructive test of nanoscale mechanical properties, utilizes the resonance frequency shifts of the AFM cantilever induced by the tip-sample interaction for quantitative mechanical property measurement of nanowires. In this study, factors including (1) diameter of MB6 nanowires, and (2) substrates (e.g., SiO2/Si, Si) used for supporting the nanowires were investigated to examine their effects of Young's modulus measurement. Initial results show that (1) the Young's modulus of BaB6 nanowire (measured on SiO2/Si substrate) decreases from 136 GPa to 90 GPa as diameter decreases from 170 nm to 60 nm; and (2) the 'Receding contacts' mechanics originally introduced by Keer et al can be adapted to study the substrate effect.

Microstructural Processes in Irradiated Materials: Radiation Effects III: He Effects on Microstructural Evolution and Deformation

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee
Program Organizers: Christophe Domain, Electricite De France; Gary Was, University of Michigan; Brian Wirth, University of California, Berkeley

Tuesday PM Room: 2008
February 17, 2009 Location: Moscone West Convention Center

Session Chairs: Yann de Carlan, CEA; Takeshi Toyama, Tohoku University

2:00 PM Invited
Ab Initio Modeling of He and H in W: *Charlotte Becquart*[1]; Christophe Domain[2]; [1]University of Sciences and Technologies of Lille; [2]Electricite De France, Research and Development

To model radiation damage in tungsten with He and H production in order to predict the evolution of the microstructure and the possibility of swelling or blistering, the elementary physical phenomena associated with the point defects created and their interaction with the He and H produced have to be characterised. The role of the impurities most commonly found in tungsten has also to be investigated and in particular the interactions they establish with point defects as well as with the light elements. We have thus used density functional theory based ab initio calculations and the VASP code to determine the investigate the interactions of He and H with point defects, impurities as well as with themselves in W. For both elements the most stable site in interstitial configuration is the same: the tetrahedral site, however their diffusion properties and their tendency to form clusters are completely different.

2:30 PM Invited
Modeling of He Diffusion and Clustering in Irradiated α-Fe: *Christophe J. Ortiz*[1]; Maria José Caturla[2]; Chu Chun Fu[3]; François Willaime[3]; [1]CIEMAT; [2]Universidad de Alicante; [3]CEA/Saclay

High levels of He are expected to be produced in materials under fusion conditions. This element and vacancies generated during irradiation agglomerate into stable He-vacancy clusters that can deteriorate the mechanical properties of materials. Although ferritic/martensitic steels are good candidates for this application due to their low swelling rate, they suffer from embrittlement and the role of He is still not clear. Using a multi-scale strategy we studied the diffusion and clustering of He in the presence of defects and impurities in irradiated α-Fe. Density Functional Theory (DFT) calculations were performed to investigate the migration mechanisms and to determine the migration and binding energies of defects. Rate Theory and kinetic Monte Carlo models were used to reproduce the He kinetics under different conditions of irradiation and temperature. The influence of impurities such as carbon on the migration of He and on the formation of small He-vacancy clusters was also studied.

3:00 PM
Interfacial Stability of He Ion Irradiated and Annealed Cu/V Nanolayers: Engang Fu[1]; Jesse Carter[1]; David Foley[1]; Amit Misra[2]; Lin Shao[1]; Haiyan Wang[1]; *Xinghang Zhang*[1]; [1]Texas A&M University; [2]Los Alamos National Laboratory

Sputtered Cu/V nanolayers with individual layer thickness (h) of 1 to 100 nm were subjected to helium ion irradiation with a peak dose of 0.5-10 dpa. In most cases, Cu/V interfaces retain after radiation. A similar hardening trend has been observed in specimens radiated at different doses, i.e., radiation hardening decreases with decreasing layer thickness. For specimens with $h \geq 5$ nm, radiation hardening seems to reach saturation when peak dose approaches 5 dpa. Hardening is negligible for fine ($h \leq 2.5$ nm) nanolayers at all dose levels. Potential mechanisms of interface-defect (induced by radiations) interactions under the context of length scale and growth of He bubbles will be discussed. In parallel we investigated the interfacial stability of as-deposited nanolayers annealed up to 600°C. Evolutions of microstructure and hardness after annealing are also investigated.

3:20 PM
Modeling the Transport and Fate of Helium in Tempered Martensitic Steels (TMS) and Nanostructured Ferritic Alloys (NFA): *Takuya Yamamoto*[1]; G. Robert Odette[1]; Brian Wirth[2]; Richard Kurtz[3]; [1]University of California, Santa Barbara; [2]University of California, Berkeley; [3]Pacific Northwest National Laboratory

Managing high helium concentrations is an absolute requirement for fusion alloys. Matrix helium bubbles act as nucleation sites for growing voids and helium on grain boundaries leads to severe degradation of fracture toughness at low temperatures and creep strength at high temperatures. Thus viable fusion alloys must trap helium in bubbles that are too small to be void nucleation sites and that also protect the boundaries from helium accumulation. The predictions of a rate theory cluster dynamics model of helium transport and fate in partitioning between and within various microstructural sites to form bubbles in both TMS and NFA are described. The master rate theory model is parameterized by atomistic submodels of helium diffusion and trapping. The model predictions of helium partitioning and bubble formation on dislocations and boundaries in TMS and on Y-Ti-O enriched nanofeatures in NFA are in good agreement with the results of in-situ helium implantation studies.

3:40 PM Break

4:00 PM Invited
An Assessment of Susceptibility to Helium Embrittlement of Nano-Scaled Oxide Dispersion Strengthened Steels: *Akihiko Kimura*[1]; [1]Kyoto University

Microstructure processing has been investigated for ODS steels and a reduced activation ferritic steel (RAFS), which were irradiated with iron and helium ions simultaneously. The void swelling of ODS steels were remarkably smaller than that of the RAFS because of much smaller size and higher density of helium bubbles in the ODS steels. The impact test results after helium implantation (900 appm He) by cyclotron clearly indicated that the ODS steels have a high resistance to helium embrittlement, while the RAFS suffered considerable intergranular embrittlement. This is considered to be due to high trapping capacity for helium atoms at matrix/particles boundaries in the steels. A simulation study based on the experimental results on microstructure processing and fracture mode change from cleavage to intergranular cracking is conducted to estimate the overall helium trapping capacity of the ODS steels, which consists of fine elongated grains and nano-scaled oxide particles in high density. Present study includes the result of "R&D of corrosion resistant super ODS steel for highly efficient nuclear systems" entrusted to Kyoto University by the Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT).

4:30 PM
Absorption of ½<111> and <100> Dislocation Loops on Mmoving Dislocations in bcc Fe: *Dmitry Terentyev*[1]; D.J. Bacon[2]; P. Grammatikopoulos[2]; Yu. N. Osetsky[3]; [1]SCK-CEN; [2]University of Liverpool; [3]Oak Ridge National Laboratory

Neutron-irradiated ferritic alloys typically contain interstitial dislocation loops with Burgers vector equal to either ½<111> or <100>. Their presence obstructs motion of dislocations, leading to an increase in the yield stress and reduction in ductility, and the ability of dislocations to absorb loops assists in the formation of 'clean' channels. The mechanisms controlling dislocation-loop reaction are therefore important. MD simulations have been used to investigate reactions between ½<111> edge dislocations and ½<111> or <100> loops at different locations with respect to the slip plane, with loop size varying from 0.5 to 10nm and temperature from 1 to 600K. Some reactions are complex, but all can be described in terms of conventional dislocation reactions in which Burgers vector is conserved. The fraction of interstitials absorbed varies from 0 to 100%. The nature of these reactions and of those requiring high applied stress for dislocation breakaway has been identified.

4:50 PM
The Role of Irradiation Microstructure in Localized Deformation in Austenitic Alloys: *Zhijie Jiao*[1]; Gary Was[1]; [1]University of Michigan

Localized deformation has emerged as a potential factor in irradiation assisted stress corrosion cracking of austenitic stainless steels in LWR environments. The degree of localized deformation is very likely controlled by the irradiation microstructure. Seven austenitic alloys with various Cr and Ni content were irradiated using 2-3 MeV protons to doses of 1 and 5 dpa at 360C. The irradiation microstructure consisting of dislocation loops, precipitates and voids was characterized using transmission electron microscopy (TEM). The degree of localized deformation was characterized using atomic force microscope (AFM) on the deformed samples after the constant extension rate tension test performed in argon. The contribution of irradiation microstructure to localized deformation will be discussed.

5:10 PM
Strain Induced Evolution of Grain Boundary Character and Taylor Factor in 316L Stainless Steel: *Elaine West*[1]; Gary Was[1]; [1]University of Michigan

Irradiation assisted stress corrosion cracking depends on grain boundary structure and deformation mode. The character of a grain boundary describes the degree of alignment between two adjacent grains, and the Taylor factor describes the propensity of a grain to undergo slip, and both may affect IASCC susceptibility. Restricted grain deformation under tensile strain alters both the grain orientation and the misorientation across a grain boundary, causing both the grain boundary character and Taylor factor to evolve with strain. Samples of 316L stainless steel were irradiated with 2.0 MeV protons at 400°C to a dose of 7 dpa and strained in supercritical water at 400°C. EBSD analysis was used to determine how the grain boundary character distribution and the Taylor factors of grains in irradiated and unirradiated 316L stainless steel evolved with strain. EBSD analysis results will be presented in the context of potential mechanisms for stress corrosion crack nucleation.

5:30 PM
Effects of Dynamic Strain Aging and Cyclic Loading on Fracture Behavior of A516 Grade 70 and Other Steels: Indrajit Charit[1]; Chang-Sung Seok[2]; *Korukonda Murty*[3]; [1]University of Idaho; [2]Sungkyunkwan University; [3]North Carolina State University

Ferritic steels used for fabricating nuclear reactor pressure vessels and reactor supports exhibit ductile-brittle transition temperature (DBTT). These steels show radiation embrittlement in terms of decreased toughness and increased DBTT following exposure to neutron irradiation. Recent work revealed decreased toughness during dynamic strain aging (DSA) as well as during reverse-cyclic loading. These have important implications on reliability of these structures under seismic loading conditions. We summarize here our work on these aspects along with synergistic effects of interstitial impurity atoms and radiation-induced defects under certain test conditions. Effect of DSA on ductility and toughness were investigated in pure iron, Si-killed mild steel, reactor support (A516) and pressure vessel steels (A533B). Temperature dependence of fracture toughness revealed plateau during DSA in A516 steel while A533B steel exhibited distinct dips. The effects of load ratio on J versus load-line displacement curves for A516 steel indicated decreased J1C as load ratio is decreased.

Nanocomposite Materials: Metallic Nanocomposites

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee, TMS: Nanomaterials Committee
Program Organizers: Jonathan Spowart, US Air Force; Judy Schneider, Mississippi State University Bhaskar Majumdar, New Mexico Tech; Benji Maruyama, Air Force Research Laboratory

Tuesday PM — Room: 3018
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Rajarshi Banerjee, University of North Texas; Bhaskar Majumdar, New Mexico Tech

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2:00 PM Introductory Comments

2:05 PM Invited
Carbon Nanotube Reinforced Nickel Matrix Nanocomposites: Junyeon Hwang[1]; Antariksh Singh[1]; Soumya Nag[1]; Jaimie Tiley[2]; *Rajarshi Banerjee*[1]; [1]University of North Texas; [2]Air Force Research Laboratory

Nanocomposites based on multi-walled carbon nanotubes (MWCNT) dispersed in a nickel matrix have been processed using the laser-engineered net shape (LENS) processing technique. The advantage of using LENS is that that while using a powder feedstock, the composites are processed via a liquid metal route involving rapid solidification. The present study focuses on the survivability of nanotubes during melt processing using LENS in a liquid nickel matrix.

Furthermore, the stability of MWCNT versus graphite powders in liquid nickel, processed under identical conditions, has been compared. These nanocomposites have been characterized in detail using scanning electron microscopy (SEM), transmission electron microscopy (TEM), 3D atom probe tomography, and, micro-Raman spectroscopy in order to determine the state of the nanotubes post processing as well as the nature of the nanotube/matrix interface. Preliminary results of wear and micro-mechanical testing of these MWCNT reinforced nanocomposites will also be discussed.

2:30 PM
Reinforcement with Atom-Infiltrated Carbon Nanotubes in Aluminum Matrix Composites: *Hyunjoo Choi*[1]; Donghyun Bae[1]; [1]Yonsei University

Reinforcing effects of multi-walled carbon nanotubes (MWNTs) in aluminum-based composites have been investigated. The composites are produced by hot rolling of the ball-milled mixture of aluminum powders and MWNTs. We present a new fabrication approach in constructing tight bonding between the MWNTs and the metal matrix by infiltrating metal atoms into the MWNTs with a controlled mechanical milling process, producing the network structure of metal atoms around the MWNTs. Furthermore, each of MWNTs is dispersed and mainly located inside the metal powders, providing an easy route of consolidation via conventional hot rolling processes. The composites exhibit remarkably enhanced strength at room temperatures. The composite sheets containing 4.5 vol. % exhibit around 600 MPa of tensile strength with ductile failure. Reinforcing effects of MWNTs in tensile properties at elevated temperature, fracture toughness, and tribological properties will be also presented.

2:50 PM
Strengthening Mechanisms in Tri-Modal 5083 Al Based Composite: *Ying Li*[1]; Yonghao Zhao[1]; Julie Schoenung[1]; Enrique Lavernia[1]; [1]University of Carlifornia Davis

We have performed systematic investigations on the microstructural origin of the previously published super-high yield strength (up to 1065 MPa) of a bulk composite with 10 wt.% boron carbide (B4C), 50 wt.% coarse-grained (CG) and 40 wt.% ultrafine-grained (UFG) 5083 Al. In principle, the strength of this composite conforms well to the rule-of-mixtures prediction, an observation that is attributed to the presence of clean metallurgical interfaces as revealed by high-resolution transmission electron microscopy (HRTEM). The grain size and distribution of the 5083 Al matrix were characterized. Al6(Mn, Fe) precipitates have been observed in CG interiors and at both UFG boundaries and interiors by scanning TEM (STEM) and electron dispersive X-ray spectroscopy (EDX) mapping. The presences of grain refinements, precipitates in the 5083 Al matrix, B4C particles and a high density of dislocations in the CG region are thought to represent the four principal factors responsible for the reported strength levels.

3:10 PM
An Investigation into the Thermal Stability of an Aluminum Based Nanocomposite: *Leyla Hashemi*[1]; Rustin Vogt[1]; Zhihui Zhang[1]; Enrique Lavernia[1]; Julie Schoenung[1]; [1]University of California, Davis

A nanocomposite of Al 5083-14.3%B_4C has been prepared by mechanically milling powders of Al 5083 and B_4C in liquid nitrogen medium; a process referred to as cryomilling. This material, when consolidated using conventional powder metallurgy techniques, is well known to exhibit high strength due to the nanocrystalline aluminum matrix and boron carbide reinforcement. It is also expected for this nanocomposite to show high thermal stability. The effect of nitrogen content on grain growth was taken into account by cryomilling powders in liquid nitrogen for different durations, while also cryomilling in argon as a benchmark data point. The cryomilled powders were characterized for homogeneity, grain size, and other microstructural features. They were then annealed at various times and temperatures and a comparison of grain growth behavior was made on the basis of nitrogen content.

3:30 PM
Microstructural Characterization of Tri-Modal Aluminum Alloy Composites: Bo Yao[1]; Helge Heinrich[1]; *Yongho Sohn*[1]; Cory Smith[2]; Mark van den Bergh[2]; Kyu Cho[3]; [1]University of Central Florida; [2]DWA Aluminum Composites; [3]US Army

Tri-modal aluminum alloy composites exhibit excellent strength and impact resistance for a variety of end-uses including survivability related applications. We have examined the microstructural characteristics of commercially produced tri-modal Al-5083 composites reinforced with B4C particulates. Mixtures of 5083 aluminum powder and boron carbide were processed with a commercial scale cryomill in liquid nitrogen and blended with coarse grain inert gas atomized 5083 aluminum powder to produce a trimodal 5083 aluminum composite. Billets were fabricated via hot-vacuum degassing followed by vacuum hot pressing. Samples were taken from the vacuum hot pressed billet for evaluation. X-ray diffraction, scanning electron microscopy, transmission electron microscopy, and related complementary analytical characterization techniques were employed for microstructural characterization with emphasis on the size, composition and distribution of microstructural features. Results and analysis from the microstructural analysis will be discussed with respect to processing conditions and variables of the trimodal aluminum alloy composites process sequence.

3:50 PM Break

4:05 PM
Aluminum Coated Carbon Nanofibers Reinforced Metal Matrix Composites: *Chitoshi Masuda*[1]; Yu-suke Nishimiya[1]; Fumio Ogawa[1]; Seiji Itabashi[1]; Minoru Oda[1]; [1]University of Waseda

Carbon nanofibers and carbon nanotubes are very attractive reinforcements of composites. Nanotubes and nanofibers are very difficult to disperse in the metal matrix using ball mill. Now the surface active agents to disperse the carbon nanotubes and nano fibers were usefule. After carbon nano fibers were coated by Al using CVD method, they were mixed with Al powders by ball mill under 200rpm in Argon atmosphere for 180 min. The mixed powers were consolidated by SPS method under 50 MPa in vacuum at 550 and 600°C. On the coated carbon nanofibers (about 150nm in diameter)examined by XRD the aluminum carbide was detected. The diameter of carbon nano fiber was about 300nm. Tensile strength of pure aluminum, Al+CNF(non-coated), Al+CNF(coated) consolidated at 600°C were 125, 185, 193MPa, respectively. On the tensile fracture surfaces many dimple patterns were observed and carbon nano fibers were also seen.

4:25 PM
Consolidated Cryomilled Al Stabilized with Diamantane: *Khinlay Maung*[1]; James C. Earthman[1]; Farghalli A. Mohamed[1]; [1]University of California Irvine

Thermal stability has been one of the primary issues for cryomilled nanocrystalline (nc) materials. In this paper we will present the effect of high temperature exposures on the nano-scale grain size of cryomilled Al stabilized with diamantane, the second smallest naturally occurring diamondoid particle. As-cryomilled powders having an average grain size of 22 nm were hot isostatically pressed (HIP'd) for consolidation at 723K (0.7 Tm of Al).The consolidated stabilized Al composite exhibited an average grain size of 50 nm. A very high grain growth exponent was estimated which was found to be consistent with Burke's model based on drag forces exerted by dispersion particles. The high value of n suggests the operation of strong pinning forces on boundaries during high temperature processing and HIPping. An examination of the microstructure by means of transmission electron microscopy (TEM) showed evidence for recovery of low angle boundaries during HIP process.

4:45 PM
A Novel Method for Preparation of Metal Matrix Nanocomposites: *Payodhar Padhi*[1]; [1]Hitech Medical College and Hospital

Particulate metal matrix composites (MMCs) can involve ceramic particulates ranging in size from few nanometers to 500 µm. Particulates are added to the metal matrix for strengthening. In particular, addition of nanoparticles, even in quantities as small as 2 weight percent can enhance the hardness or yield strength by a factor as high as 2. Solidification processing is a relatively cheaper route. However, during solidification processing nanoparticulates tend to agglomerate. To overcome these difficulties a non-contact method, where the ultrasonic probe is not in direct contact with the liquid metal, was attempted to disperse nano-sized Al2O3 particulates in aluminum matrix. From HRTEM studies it is seen that the Al2O3 particles are distributed uniformly. Both hardness and micro hardness were measured at different locations. It was found that the variations in hardness from location to location are not so significant. In micro scale the hardness is uniform throughout the sample.

5:05 PM
Dispersing of Nano-Particles in Molten Aluminum Using High-Intensity Ultrasonic Vibrations: Clause Xu[1]; Lu Shao[1]; *Qingyou Han*[2]; [1]Hans Tech; [2]Purdue University

The most inexpensive method for processing particle reinforced aluminum matrix composites involves the use of a stirrer for dispersing particles in molten metal. The method is successful in making composites containing particles larger than a few microns but is difficult in dispersing particles in the nano-size range (<100 nm). This article discusses an enabling technology for dispersing nanoparticles in molten metal using high-intensity ultrasonic processing technology. The use of high-intensity ultrasonic vibration breaks up the nanoparticle clusters and disperses the individual nanoparticles into the melt. The resultant nanocomposites are of superior mechanical properties especially tensile and creep resistance at elevated temperature. Issues associated with the dispersing of nano-particles are addressed.

5:25 PM
Friction Stir Processed Nanocomposite Surface Layers for Aluminum Alloys: *Jun Qu*[1]; Hanbing Xu[1]; Zhili Feng[1]; D. Alan Frederick[1]; Peter Blau[1]; [1]Oak Ridge National Laboratory

Previous work has demonstrated the feasibility of using a friction stir process (FSP) to form a nanocomposite layer on a pure aluminum surface to improve the hardness and wear-resistance without sacrificing the bulk ductility and conductivity. This study applied this surface engineering technique to the Al 6061-T6511 alloy. Nano- or micro-sized reinforcement materials (Al2O3, SiC, TiO2, LaB6) in different shapes (particles or fibers) and sizes (30 nm – 5 um) were friction stirred into the Al 6061 alloy surface to form a composite layer up to 3 mm thick. The concentration of the hard phase was in the range of 10-20 vol%. Compared with a non-processed Aluminum surface, the FSP-formed nanocomposite surface exhibited a moderate increase in hardness and a substantial improvement on wear-resistance by more than one order of magnitude when rubbed against a hardened bearing steel. A post-FSP heat treatment (T6) afforded further enhancement of the wear performance.

Near-Net Shape Titanium Components: Casting, Welding and Beam Processes

Sponsored by: The Minerals, Metals and Materials Society, TMS: Titanium Committee
Program Organizers: Rodney Boyer, The Boeing Company; James Cotton, The Boeing Company

Tuesday PM — Room: 2010
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Rodney Boyer, The Boeing Company

2:00 PM
The Boeing Approach to More Cost Effective Ti Components: *Rodney Boyer*[1]; Kevin Slattery[1]; Todd Morton[1]; James Cotton[1]; [1]The Boeing Company

The competition in commercial airframes, the present economy (fuel prices) and the continuing technology development globally mandate the development of new alloys and technologies to reduce the cost of titanium hardware. Obviously this applies to all materials, but with the high cost of titanium it is even more imperative for this technology area. Reducing the cost of titanium embraces the entire value stream including raw materials, melt and meltless technologies, machining, and reduction of the buy:fly ratio. The latter could involve modeling, near-net shape forging, casting, extrusions, welding, etc. Some of the Boeing approaches to reducing the cost of titanium hardware will be discussed.

2:20 PM
Development Of Databases To Relate Composition, Microstructure, And Properties For The Production Of Near-Net Shape Functionally Graded a+ß and ß-type Ti-Based Materials: *Peter Collins*[1]; Dan Huber[1]; Brian Welk[1]; Hamish Fraser[1]; [1]Ohio State University

Functionally graded Ti-based components offer the potential to engineer location-specific properties in unitized structures. However, the microstructures and properties arising from composition landscape between the two terminal compositions will often remain largely unexplored. This work explores the microstructural evolution that can occur at various compositions along binary (Ti-xMo and Ti-xFe) and multi-component (e.g., Ti to Ti-1Al-8V-5Fe or Ti to Ti-1Al-7Fe) gradients produced using laser engineered net shaping (LENS™) and begins to populate databases that may be probed to relate composition with microstructure and properties.

2:40 PM
Advanced Titanium Welding Processes for Improved Material Utilization in Aerospace Manufacturing: *Paul Edwards*[1]; Chris Swallow[1]; Dan Sanders[1]; Kevin Slattery[1]; Amy Helvey[1]; [1]The Boeing Company

The use of Titanium by the aerospace industry has recently been driven to unprecedented levels, which has resulted in price escalations and temporary supply shortages. Most titanium parts are machined out of plate, blocks, forgings or extrusions, which all result in wasted scrap material and unnecessarily high fabrication costs. In order to reduce the buy-to-fly ratio of titanium parts, more efficient manufacturing techniques must be implemented. Laser Welding, Linear Friction Welding and Friction Stir Welding of titanium 6Al-4V have all been developed in order to produce low cost aerospace structural components. For each of these welding technologies, process parameters have been identified for producing very repeatable, high quality welds on a variety of material thicknesses and joint configurations. Extensive metallurgical examinations and preliminary mechanical property evaluations have been performed to qualify these process for fabricating structural aerospace parts.

3:00 PM
Microstructure-Properties of Alloy Ti-5Al-5Mo-5V-3Cr Castings: *E. Chen*[1]; L. Weihmuller[2]; D. Bice[1]; G. Hall[2]; W. Thomas[2]; [1]Transition45 Technologies Inc; [2]Bell Helicopter Textron

Alloy Ti-5Al-5Mo-5V-3Cr-0.5Fe (Ti-5553) is an emerging high-strength titanium alloy with improved static mechanical properties compared with the industry workhorse Ti-6Al-4V. Studies to date have shown that this material also has comparable or better fatigue properties to 4340 steel and Ti-6Al-4V, respectively, thus could be a replacement candidate for these alloys to achieve weight savings and/or enhanced durability. The ability to cast complex net shapes from a high strength titanium as this also offers the potential to save both cost and weight over traditionally forged components. This presentation covers work being conducted on characterizing the microstructure-properties of Ti-5553 castings. Mechanical properties covered here include tensile, toughness, and fatigue behavior for microstructures achieved under different thermo-mechanical processing conditions. The results show outstanding strength and fatigue properties relative to both wrought and cast Ti-6Al-4V. This work was supported by the Naval Air Warfare Center.

3:20 PM
Optimization of Layered Additive Manufacturing Processes: *Raghavan Srinivasan*[1]; Anil Chaudhary[2]; Matthew Keller[2]; [1]Wright State University [2]Applied Optimization Inc.

Layered additive manufacturing offers a flexible approach for the production of complex near-net- and net-shaped components by building up three dimensional objects by selectively adding material on successive two dimensional layers. A recently developed software based tool, SAMP ® (Simulation of Additive Manufacturing Processes) provides the opportunity to simulate laser (or electron beam) based powder deposition processes. Using Ti-6Al-4V as the model material, SAMP ® will be used to conduct a systematic study of the effect of parameters, such as laser power, beam traverse rate, powder flow rate, and deposition schedule on the predicted microstructure, and to optimize the layered manufacturing process. Optimization of the process schedule has several cost benefits, such as decreased certification costs resulting from a uniform microstructure, decreased "buy/fly" ratio, and more effective use manufacturing equipment. Several part geometries ranging from a simple thin wall on a plate to more complicated geometries, such as "T", "D", "H" or box shapes, will be investigated.

3:40 PM
The Effect Of Powder Production Process On Microstructure And Mechanical Properties Of Electron Beam Deposited Ti6Al4V: *Jonathan Nguyen*[1]; Baolong Zheng[1]; Troy Topping[1]; Yizhang Zhou[1]; Scott Gilley[2]; James Good[3]; Enrique Lavernia[1]; [1]University of California, Davis; [2]Tec Masters, Inc.; [3]Teledyne Brown Engineering

Arcam Electron beam melting (EBM) is an emerging technique that utilizes an electron beam to melt metal powder in a layered powder additive process. Samples produced are near net shaped as well as have comparable or superior mechanical properties, thereby reducing costs associated with machining time and post heat treatment processing, respectively. Currently, there have been no published results on the effects of the method of prealloyed powder production processes. In this article, square columns were fabricated at a voltage of 60 kV and a current of 9.1 mA, using gas atomized (GA) and plasma rotating electrode process (PREP) powders to evaluate the effects of the initial powder on microstructure and mechanical properties. The microstructure of the as-deposited columns was characterized using optical and scanning electron microscopy (SEM). The amount of porosity resulting from the type of prealloyed powder used and its implication on the mechanical properties will be discussed.

4:00 PM
Research of TiAl Alloy Complex Component Precision Casting: *Nan Hai*[1]; [1]Beijing Institute of Aeronautical Materials

In this paper the fludity of TiAl alloy was compared with Ti6Al4V in the graphite mould.By wax pattern preparing,ceramic mould making,centrifugal casting,and microstructure and property analysing,TiAl alloy complex component precision casting technology was introduced.Turbo charger and incresing pressure case were cast. The smallest wall thickness is 1mm.

4:20 PM
Effect Of Microstructure Variations On Fatigue Of Investment Cast Ti-6Al-4V: *Adam Pilchak*[1]; James Ault[2]; James Williams[1]; [1]Ohio State University [2]Precision Castparts Corp

Investment castings of titanium are being used in increasing numbers. In the cast and hot isostatic pressed condition the material has a coarse, fully lamellar microstructure characterized by poor fatigue crack initiation resistance, but excellent crack growth resistance. Friction stir (FS) processing can improve the crack initiation resistance of castings. These FS processed castings may be able to replace forged components resulting in cost reduction for parts with complex geometries. In the course of studying the effects of FS processing on fatigue, several microstructural variations have been observed. These include tungsten contamination from the FS tool and regions of equiaxed α grains formed as a result of HIP pore closure. Furthermore, the effect of yttrium rich particles on fatigue crack initiation in cast and wrought Ti-6Al-4V has been examined. This talk will describe the sources of these microstructural variations and their effect on fatigue behavior.

Neutron and X-Ray Studies of Advanced Materials: Advances in Line Profile

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Titanium Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter Liaw, The University of Tennessee; Jaimie Tiley, US Air Force

Tuesday PM — Room: 3016
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Yan-Dong Wang, Northeastern University; Peter Liaw, University of Tennessee

2:00 PM Keynote
Advances In Diffraction Line Profile Analysis Of Nanocrystalline And Heavily Deformed Materials: *Paolo Scardi*[1]; [1]University of Trento

In the past ten years diffraction line profile analysis evolved from single-peak methods, with more or less arbitrary steps to remove background and to separate overlapping peaks, to one-step full pattern modeling methods. In particular, the Whole Powder Pattern Modeling is a new paradigm for the analysis of diffraction line profiles, according to which the diffraction pattern from polycrystalline materials can be analyzed on the basis of physical models of the microstructure, without using arbitrary analytical profile functions. The present contribution shows recent advances in the study of nanocrystalline materials, where crystalline domain shapes and size distribution can be analyzed in detail, as well as of heavily deformed materials, where grain refinement and lattice defect type and density can be obtained. Results are compared with those by other approaches (e.g. based on Debye formula) and different techniques (e.g., TEM). The specific case of dislocation containing materials is discussed in detail.

2:30 PM Invited
Your Synchrotron Powder Diffraction Instrument: 11-BM at the Advanced Photon Source: *Brian Toby*[1]; [1]Argonne National Laboratory

Synchrotron powder diffraction has revolutionized powder diffraction in that it makes possible data collection with tremendous resolution and signal to noise, or allows for extremely rapid collection (<1 second) collection of entire, high quality, powder diffraction patterns. The high penetration and data sensitivity over wide Q range even allows synchrotrons to make inroads into territory that previously demanded neutrons: extreme sample environments and site occupancy studies. The problem has been that access to synchrotrons has been difficult. The 11-BM synchrotron powder diffractometer at the APS now offers easy mail-in access with quick turnaround for routine structural analysis, providing truly first-quality data. This talk will present the capabilities of the instrument and how these kinds of data can solve real-world materials problems. Also to be discussed is how to obtain access.

2:50 PM Invited
Defects And Strains By X-Ray And Neutron Diffraction: Modeling And Applications: *Davor Balzar*[1]; [1]University of Denver

With the constant improvement of experimental techniques, especially with the advent of new-generation synchrotron and neutron sources with superior resolution, sometimes subtle effects of lattice strains and low defect concentrations on diffraction lines, become easier to study. Furthermore, modern analysis methods, such as Rietveld refinement, increasingly go beyond the determination of structural parameters and include refinable parameters with a physical significance within accurate line-broadening models. Some recent improvements in modeling of line broadening and line shift in Rietveld refinement will be discussed. In the second part, several examples will be presented. In particular, I will talk on the determination of a complete strain tensor from Rietveld refinements of multiple TOF neutron-diffraction spectra, size-strain round-robin results, and applications of line-broadening and line-shift analyses to several materials of technological interest.

3:10 PM
Development of Intergranular Thermal Residual Stresses in Beryllium During Cooling from Processing Temperatures: *Thomas Sisneros*[1]; Donald Brown[1]; Mark Bourke[1]; Bjorn Clausen[1]; Sven Vogel[1]; Brian Smith[1]; Steve Abeln[1]; Michael Steinzig[1]; [1]Los Alamos National Laboratory

The intergranular thermal residual stresses in randomly textured solid polycrystalline beryllium have been determined by comparison of crystallographic parameters in solid and powder samples measured by neutron diffraction during cooling from 800ºC. The stresses have been calculated by an Eshelby type polycrystalline model for comparison. The internal stresses are not significantly different from zero above 575ºC and increase nearly linearly below 525ºC. At room temperature the c-axis perceives roughly -200MPa of compressive internal stress; the a-axis 100MPa of tensile stress.

3:25 PM Invited
Defect-Related Physical-Profile Based X-Ray And Neutron Line Profile Analysis: *Tamas Ungar*[1]; Levente Balogh[1]; Gábor Ribárik[1]; [1]Eotvos University

Diffraction line broadening is caused by different defects present in crystalline materials: (i) small coherent domains, (ii) dislocations, (iii) other types of microstrains, (iv) twin boundaries (v) stacking faults, (vi) chemical inhomogeneities and (vii) grain-to-grain second order internal stresses. Line profile analysis provides qualitative and quantitative information about defect types and densities, respectively. Line profiles can broaden, be asymmetric, can be shifted and these features can be anisotropic in terms of hkl indices. A few thumb rules help qualitative selection of lattice types. If the beradths are globally not increasing with hkl, the defects are of size type, i.e. either domain size is small or twinning or faulting, or both are present. Whenever the beradths increase globally, the defects produce microstrains. It will be shown that physically based profile functions can be determined for each defect type and the hkl anisotropy allows evalaution of the different defect types and density.

3:45 PM Break

4:00 PM Invited
Microstructure of Ultrafine-Grained Metals after Severe Plastic Deformation and Its Thermal Stability Studied by XRD in Combination with PAS, EBSD and TEM: *Radomir Kuzel*[1]; Miloš Janecek[1]; Jakub Cížek[1]; Milan Dopita[2]; [1]Faculty of Mathematics and Physics, Charles University; [2]Technical University Freiberg

Different samples of cubic (Cu, Ni, Fe, Nb) and hexagonal (Mg) metals and their alloys or composites deformed by equal-channel angular pressing (ECAP) and high-pressure torsion (HPT) were studied by XRD line profile analysis and back-scattering photos, transmission electron microscopy (TEM), electron backscatter diffraction (EBSD) and positron annihilation spectroscopy (PAS). XRD revealed changes in both dislocation density (line broadening) and dislocation correlations (line shape) with number of ECAP paths, abnormal grain growth and bimodal grain-size distribution for Cu samples with annealing, grain fragmentation for iron, in-depth and in-plane microstructural inhomogeneities for HPT samples. TEM and EBSD indicated changes of grain boundaries with ECAP paths (transition from low angle to high angle grain boundaries). XRD and EBSD could characterize variation of the texture. PAS detected the presence microvoids and enabled determination of their size. PAS was also complementary with XRD for dislocation density determination (lower and higher values, respectively).

4:20 PM
Synchrotron X-Ray Study of Nanocrystalline Ni during Cold Rolling: *Li Li*[1]; Yan-Dong Wang[2]; Tamas Ungar[3]; Yang Ren[4]; Hahn Choo[1]; Peter Liaw[1]; [1]University of Tennessee; [2]Northeastern University; [3]Eötvös University; [4]Argonne National Laboratory

Two foils of nanocrystalline Ni metal (with nominal grain size of 10 - 20 nm) were rolled at room temperature (RT) and liquid-nitrogen temperature (LNT), respectively. Synchrotron-based high-energy x-ray diffraction was employed in the undeformed and deformed specimens to study the microstructure evolution. Quantitative microstructures, such as the area-average mean crystallite size, dislocation density, and dislocation character, were investigated by the x-ray line-profile analysis. X-ray line profile analysis is especially useful to the study of submicron-grained or nano-grained materials where the x-ray line broadening becomes well pronounced, and the observation of defects with a very large density is often challenging using conventional TEM. Texture experiment was also performed in the experiment. This research is supported by NSF-IMI program (DMR-0231320) and NSF-MRI program (DMR-0421219).

4:30 PM Invited
Total Scattering: The Key to the Local and Medium Range Structure of Complex Materials: *Thomas Proffen*[1]; [1]Los Alamos National Laboratory

Structural characterization is mainly based on the measurement of Bragg intensities and yields the average structure of the crystalline material. However, this approach ignores any defects or local structural deviations that manifest themselves as diffuse scattering. It also fails in case of disordered materials and nano-crystalline. The total scattering pattern, however, contains structural information over all length scales and can be used to obtain a complete structural picture of complex materials. Suddenly one has access to a new parameter, the real-space range of the refinement and structures can be analyzed as function of length scale straight forwardly. Here we present different applications of this technique including data taken on the high resolution neutron powder diffractometer NPDF located at the Lujan Neutron Scattering Center. This instrument is design for total scattering studies using the Pair Distribution Function (PDF) approach and length scales in excess of 200Å can be accessed.

4:50 PM Invited
X-ray Microdiffraction Study of the Ni-based Superalloys After Friction Stir Processing: *Oleg Barabash*[1]; Rozaliya Barabash[1]; Gene Ice[1]; Zhili Feng[1]; [1]Oak Ridge National Laboratory

Advanced 3D polychromatic X-ray micro diffraction at the APS synchrotron was applied to study structural changes in the Ni-based superalloy caused by Friction Stir Processing (FSP). Spatially resolved 3D Laue diffraction allowed following the changes in dislocation arrangement with depth in different regions of the FSP alloys. X-ray diffraction results are complemented by SEM and EBSD. Formation of several specific zones was established: friction stir zone, and thermomechanicaly and heat affected zones. It was shown that FSP generates a large number of geometrically necessary dislocations. Anisotropy of all stir processing zones is demonstrated. Ultrafine grain size is observed in the stir zone. Research is sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Science U.S. Department of Energy.

5:10 PM Invited
Dynamic Recrystallization and Grain Refinement in a Friction Stir Processed AZ31B Mg Alloy: An X-Ray Line Profile Analysis: *Hahn Choo*[1]; Zhenzhen Yu[1]; Levente Balogh[2]; Tamás Ungár[2]; Zhili Feng[3]; [1]University of Tennessee; [2]Eötvös University; [3]Oak Ridge National Laboratory

Friction stir processing (FSP) has a great potential as a novel method for the fabrication of bulk ultrafine-grained structural materials. While it is known that the combined effects of severe plastic deformation and temperature result in fine grain sizes through dynamic recrystallization (DRX) process during FSP, the exact mechanism of DRX and the detailed understanding of the resulting microstructure are still unclear. In this study, a series of FSP was performed on AZ31B magnesium alloy under different strain rate and temperature by varying the processing parameters. High-resolution x-ray line profile analysis was used, in combination with electron microscopy, to investigate the dynamically recrystallized microstructure as a function of the processing parameters. The effects of changing the processing parameters on the resulting microstructures; such as grain size, sub-grain size, dislocation density, and texture; in the dynamically-recrystallized zone will be discussed in the context of deformation and recrystallization mechanisms.

5:30 PM
Grain Structure and Dislocation Densities in a Friction Stir Welded Aluminum Alloy Studied by X-ray Line Profile Analysis: *Wan Chuck Woo*[1]; Levente Balogh[2]; Tamas Ungar[2]; Hahn Choo[3]; Zhili Feng[1]; [1]Oak Ridge National Laboratory; [2]Eötvös University; [3]University of Tennessee

Severe plastic deformation and recrystallization process results in the dislocation-embedded 'grain structure'. The dislocation density and grain structure of a friction stir welded 6061-T6 aluminum alloy was examined as a function of distance from the weld centerline using high-resolution micro-beam x-ray diffraction. Theoretically simulated and directly measured diffraction patterns are compared to each other and determined the microstructural characteristics. The results of the x-ray peak profile analysis show that the dislocation density is about 1.2×10^{14} m^{-2} inside, 4.8×10^{14} m^{-2} outside of the weld region. The average subgrain size is about 180 nm in both regions. Compared to the base material, the dislocation density was significantly decreased in the dynamic recrystallized zone of the friction stir welds, which is in good correlation with the TEM observations. The influence of the dislocation density on the strain hardening behavior during tensile deformation is also discussed.

5:45 PM
The Effect Of Welding Pressure On Microstructural Evolution In Dissimilar Steel Friction Welds Studied By Synchrotron X-Ray Diffraction: *Richard Moat*[1]; Michael Preuss[1]; Mallikarjun Karadge[1]; Martin Rawson[2]; Simon Bray[2]; [1]University of Manchester; [2]Rolls Royce Plc

The 2D microstructural evolution across inertia friction welded high strength steels of Aermet 100 against S/CMV has been studied as a function of axial forging pressure. High energy synchrotron x-ray diffraction was used to record 2D maps of retained austenite within the heat affected zone (HAZ), identify the thermomechanically affected zone (TMAZ) from peak broadening maps and use peak shape analysis to identify regions of martensitic phase transformation. Since a number of complete diffraction rings were recorded, it was also possible to calculate the crystallographic texture at each measurement point. Results show that with increasing weld pressure the amount of martensite formed within the heat affected zone is reduced significantly while the amount of retained austenite seems to increase. The texture mapping allowed relating hardness troughs to recrystallised regions. The results are discussed in terms of consequences for residual stress generation during joining.

6:00 PM
Recovery of Ultra-Fine Grained Materials by Severe Plastic Deformation: *Yonghao Zhao*[1]; Thomas Ungar[2]; Y. Li[3]; Ruslan Valiev[4]; Yuntian Zhu[5]; Yizhang Zhou[3]; Enrique Lavernia[3]; [1]Los Alamos National Laboratory; [2]Eotvos University; [3]University of California, Davis; [4]UFA State Aviation Technical University; [5]North Carolina State University

It is well known that ultrafine-grained (UFG) metals prepared by severe plastic deformation (SPD) have nearly zero uniform elongation due to their low dislocation accumulation capability which was consumed during SPD process. Recovery by low temperature annealing is an effective way to regain the lost strain hardening/dislocation accumulation and therefore the ductility. In this work, we employed X-ray diffraction (XRD) combined with transmission electron microscopy (TEM) techniques to systematically investigate the recovery processes of the UFG Cu, Al, CuZn30, UFG Ti by equal-channel-angular (ECAP) process. Evolutions of dislocation density and grain size during annealing were analyzed. Our work will provide basic guidance for optimize mechanical properties of UFG materials.

Pb-Free Solders and Emerging Interconnect and Packaging Technologies: Tin Whisker Formation and Mechanical Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Sung Kang, IBM Corp; Iver Anderson, Iowa State University Srinivas Chada, Medtronic; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Albert Wu, National Central University

Tuesday PM — Room: 2020
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Laura Turbini, Research In Motion; K. Subramanian, Michigan State University

2:00 PM Invited
Structure-Property Evaluations in Sn-Based Solders with Nano-Structured Chemicals: *K. Subramanian*[1]; Deep Choudhuri[1]; Andre Lee[1]; [1]Michigan State University

The integrity of solder joints deteriorates with repeated thermal excursions due to stresses arising from mismatch of coefficient of thermal expansion between the entities present in the packages and the anisotropic nature of Sn. Impact of this thermomechanical fatigue (TMF) depends on parameters such as imposed temperature differences, dwell times, ramp-rates, etc. Our recent investigations have shown that it also depends on the temperature extremes experienced during TMF, since the deformation characteristics of Sn-based solders are highly temperature sensitive. Strongly-bonded inert nano-structured reinforcements significantly improve the TMF resistance of Sn-based electronic solder interconnects. During reflow, active radicals present at the surface of these nano-structured inert structures facilitate strong bonding with the solder. Such strongly bonded inert structures do not coarsen, or de-bond from solder matrix, during service. Roles of various active radicals on the reflowed microstructure, and the resultant performance of SAC 305 and eutectic Sn-Ag solder joints are presented.

2:20 PM
Grain Orientation Effects On The Temperature Cycling Stability Of 180µm Pitch Pb-Free Bump Interconnects: John Osenbach[1]; *Dongmei Meng*[1]; Chis Richardson[1]; Patrick Variot[1]; Chris Richardson[1]; [1]LSI Corporation

The crystallographic orientation of Pb-free solder has been shown to impact the thermo-mechanical and possibly electromigration reliability of the joint. In this paper, results on the influence of crystallographic bump orientation on the temperature cycling performance of Pb-free-180µm pitch-flip chip joints are presented. EBSD results of individual 180µm pitch bumps indicate that individual bumps contain multiple grains. In many cases the grains are highly oriented, separated by low angle grain boundaries. This type of microstructure produces a bump that from a thermo mechanical perspective can, to first order, be treated as a single crystal bump. In this paper, we show that the crystallographic orientation of the bump strongly influences the stability of individual bumps on flip chip devices subjected to extensive temperature cycling testing at temperatures between -55C to +125C.

2:35 PM
The Performance and Fracture Behavior of Low Silver Lead-free Solder Joints upon Micro-Impact Test: *Ya Ling Huang*[1]; Kwang Lung Lin[1]; D. S. Liu[1]; [1]National Cheng Kung University

Various low silver lead-free solder alloys were examined herein to investigate the joint strength, fracture energy, and fracture behavior with micro-impact test at impact velocity of 1m/sec. The solder investigated include Sn-1Ag-0.5Cu (SAC105), Sn-1.2Ag-0.5Cu-0.05Ni, Sn-1Ag-0.5Cu-0.05Ni, Sn-1Ag-0.5Cu-0.05Co, and Sn-1Ag-0.1Cu-0.02Ni-0.05In (SAC101). Most of the solder joints, except SAC105 and SAC101, exhibit mixed brittle (fracture at interface) and ductile (fracture in bulk solder) fracture modes. The morphology of intermetallic compounds were found to affect fracture behavior of the solder joints. Needle-type Ni3Sn4 and (Cu,Ni)6Sn5 intermetallic compounds were observed in SAC101 and SAC105 solder joints, respectively. The SAC101 exhibits ductile fracture behavior while the SAC105 exhibits brittle fracture behavior. Layer-type intermetallic compounds were observed in the Sn-1.2Ag-0.5Cu-0.05Ni, Sn-1Ag-0.5Cu-0.05Co, and Sn-1Ag-0.5Cu-0.05Ni solder joints which exhibit mixed fracture behavior. The ductile fracture behavior has higher fracture energy, displacement and force than brittle fracture.

2:50 PM
Effects of Temperature and Strain Rate on Mechanical Properties of Lead-Free Solders: *Hongtao Ma*[1]; [1]Cisco Systems

Due to the high homologous temperature of solder alloys (Th > 0.5Tm), the mechanical properties of solder alloys are strongly temperature and strain rate dependent. The investigation of this dependence on temperature and strain rates is therefore important in order to fully understand the materials behavior of solder alloys, and accurately predict the reliability of solder joints. There have been various studies on the temperature and strain rate dependence. However, none of the currently available documented data has considered the possible room temperature contribution in their data. In our prior work on aging effects (Ma, et al., ECTC 2006), we demonstrated that the observed material behavior variations of SAC405 and SAC305 lead free solders during room temperature aging (25°C) were unexpectedly large and universally detrimental to reliability. In this study, in order to reduce any room temperature aging contribution, all specimen tested were preconditioned under the same conditions.

3:05 PM
Finite Element Analysis Of Stress Evolution In Sn Films Due To Intermetallic Growth: *Eric Buchovecky*[1]; Nitin Jadhav[1]; Allan Bower[1]; Eric Chason[1]; [1]Brown University

Although mechanical stress induced by growth of an intermetallic phase has been shown to be an important driving force for the formation of whiskers in pure Sn coatings on Cu, the mechanisms by which stresses are generated and transmitted through the Sn film are not fully understood. In this study, we perform three-dimensional finite element simulations to quantitatively model the stress evolution due to elastic and plastic deformation coupled with stress-driven grain boundary diffusion (Coble creep) within a polycrystalline Sn film. We explore the effects of grain size, film thickness and microstructure in the Sn film, as well as the morphology, distribution and growth rate of the intermetallic particles. We find that both dislocation plasticity in the Sn immediately surrounding the intermetallic particles and mass transport along grain boundaries are necessary to produce stress histories in the Sn film consistent with experimental measurements from our Laboratory

3:20 PM
Stress Distribution In Sn-Cu Layers And Its Relation To Whisker Formation: *Nitin Jadhav*[1]; Vivett Fawal[1]; Evan Laprade[2]; Eric Buchovecky[1]; Jae Wook Shin[1]; Eric Chason[1]; [1]Brown University; [2]RPI

Stress is generally believed to be the driving force for Sn whisker formation so measuring its time evolution and its distribution through the layers is useful for understanding its role in whisker growth. We have used a real-time wafer curvature technique to monitor the evolution of stress in bilayers of Sn and Cu deposited on glass substrates. By monitoring a series of samples with different initial Sn and Cu layers thicknesses, we can estimate the distribution of stress through the Sn, Cu and IMC layers. We additionally measure the change in curvature when the Sn layer is removed by selective etching to separate the stress in the Sn from the Cu/IMC layers. Our results suggest that the Sn layers have a relatively uniform compressive stress, the Cu layers have tensile stress confined to a region near the Cu/Sn interface and the stress in the IMC layer is small.

3:35 PM
Failure Mechanisms in Pb-Free Interconnects Resulting from Temperature and Mechanical Cycling: *Brent Fiedler*[1]; Jared Fry[1]; Morris Fine[1]; [1]Northwestern University

The quantitative effects of temperature and mechanical cycling on failure mechanisms in Sn-4.0Ag-0.5Cu (SAC405) solder ball grid array (BGA) interconnects are examined at the component level. The microstructure and composition changes – due to mechanical and temperature cycling - in the lead-free solder BGA are studied by scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS). The studies focus on the interface of the solder, intermetallic compounds (IMC) and Cu pad. Cracking from temperature cycling is most often observed at interfaces with IMC, while mechanical cycling primarily initiates cracks in the solder. In-situ crack initiation and propagation is evaluated using the resistance of the daisy-chained arrays for structural health monitoring (SHM) of interconnects.

3:50 PM Break

4:05 PM

Metal Whisker Formation in Multiphase Electronic Solder Joints under Electromigration: *Guangchen Xu*[1]; Mengke Zhao[1]; Hongwen He[1]; Fu Guo[1]; [1]Beijing University of Technology

Numerous electronic system failures have been attributed to short circuits caused by metal whiskers that bridge closely-spaced circuit elements maintained at high current density. Typically, in the single phase of interconnect atoms are driven from the cathode to the anode and a compressive stress built up at the anode end of the stripe form the hillocks. However, the electronic solders used in interconnects are multiphase materials where primary and secondary diffusion entities exist. In this study, various current densities and ambient temperatures were applied to multiphase solder joints to accelerate metal whisker growth. Either Sn or Bi whiskers was observed depending on the field parameters applied. Such service parameters also affect the site and length of the metal whiskers. It was found that whiskers of the primary diffusion entity tend to form at the anode side, while those of the secondary diffusion entity tend to form at the cathode side.

4:20 PM

Mitigation Of Tin-Whiskers Growth By Applying Multiple Ni/Sn Plating Prior To The Final Tin Finish: *Aleksandra Dimitrovska*[1]; Dechao Lin[1]; Radovan Kovacevic[1]; [1]SMU

World-wide research on the formation of Sn-whiskers has agreed that presence of the compressive stresses built up in the Sn-film is one of the driving forces for Sn-whiskers growth which cause failures of various components in the electronic industry. Migration of Cu-element from the substrate to the Sn-film generates Sn-Cu inter-metallic compounds at the Sn/substrate interface and in the Sn-film which triggers the growth of the compressive stresses. To reduce this stress level a multiple composite Ni/Sn layering was developed in this study. A Ni-layer was first deposited onto the brass substrate and then followed by multiple Sn/Ni layers with the thickness of several microns each. Experimental results demonstrated that this new layering procedure on a brass substrate significantly reduces both the volume fraction of Sn-Cu inter-metallic compounds and the Cu content in the final tin finish, resulting in tin whisker's growth mitigation.

4:35 PM

Competing Mechanism between Intermetallic Compounds Formation and Whisker/Hillock Growth in Pb-free Solder Joints: *Jung-Kyu Han*[1]; Luhua Xu[1]; King-Ning Tu[1]; [1]University Of California, Los Angeles

Whisker/hillock is formed at the anode side in flip-chip solder joints due to the accumulation of Sn. Besides, the electromigration of Cu causes the formation of intermetallic compounds at the anode side in solder joints. In order to see the relationship between whisker/hillock growth and intermetallic compounds formation, the cross-sectioned flip-chip SnAgCu samples were studied with current stressing (1.41 X 10^4 A/cm^2) at 150 deg C. As a result, whisker/hillock was formed at the anode side and was gradually grown at the beginning. The growth of whisker/hillock, however, was hindered by intermetallic compounds formation as time goes by. It seems that Cu which was migrated along the electron flow consumed Sn to form intermetallic compounds at the anode side and blocked Sn source for whisker/hillock growth. The schematic diagram and competing mechanism between intermetallic compounds formation and whicker/hillock growth is proposed.

4:50 PM

Copper Dissolution and Tin Whisker Growth in Lead-Free Solders: *Lizabeth Nielsen*[1]; Dana Medlin[1]; [1]South Dakota School of Mines and Technology

The purpose of this research was to study and understand the tin whisker growth mechanism and the issue of copper dissolution during thermal treatments of lead-free solders. A brief history of tin whiskers and a literature review of copper dissolution issues will also be discussed. An analysis of the tin whiskers was performed by electron backscatter diffraction (EBSD) on the individual whiskers and the base materials where the whiskers originated to determine the crystal orientation of both the whisker and the base material to determine the relationship between base material crystal orientation, residual stress and whisker origin. The EBSD analysis was also able to determine the tin phases for both the whiskers and the base solder material. Solder samples were analyzed through the use of traditional metallographic techniques and scanning electron microscopy to determine the amount of copper dissolution that was occurring with different printed circuit board process parameters.

5:05 PM

Whiskers, Hillocks, And Film Stress Evolution In Electroplated Sn And Sn-Cu Films: *Aaron Pedigo*[1]; Patrick Cantwell[1]; John Blendell[1]; Carol Handwerker[1]; [1]Purdue University

The spontaneous growth of surface defects, including whiskers and hillocks, on lead-free tin electroplated films is believed to be a stress relief phenomenon. Previous research has shown that it is possible to plate pure tin and observe only hillock growth. Whisker growth, however, can be biased over hillock growth with the addition of copper contamination to the electrolyte. In this work, hillock and whisker growth was correlated to measured stress in electroplated tin and tin-copper films using cantilever beams. Cross-sections and morphologies of these defects were observed using SEM and FIB. A transition in short-term film behavior between 0.5 and 1.3% copper in the film was characterized by an increase in plating stress, long-term stress, and propensity to whisker. A transition in hillock morphology was also observed. These results support a growth model where the ratio of surface uplift to grain boundary motion determines defect type.

5:20 PM

IMC Formation to Block Whisker and Hillock Growth in Lead-Free Flip-Chip Solder Joints under Electromigration: *Shih-Wei Liang*[1]; Chih Chen[2]; J. K. Han[3]; Luhua Xu[3]; K. N. Tu[3]; [1]National Chiao Tung University ; [2]National Chiao Tung University; [3]University Of California, Los Angeles

In this study, the flip chip was cross-sectioned for in-situ observation. At current density of 1.3 x 104 A/cm2 and at 100°C, we observed that the hillock squeezed out at the board side was more serious than the whisker grew at the chip side. Accompanying the hillock growth, the Cu-Sn IMCs were spread and grown in the anode after long stressing time. The distribution of IMC was investigated by a second cross-sectioning of the flip chip sample. We speculate that the board side has supplied enough copper to react with tin to form Cu6Sn5. The IMC may create compressive stress to form the hillocks. However, the excess IMC formed in the tin grain boundaries may block the diffusion path of Sn and slow down the growth of tin whisker and hillock. Our results show that the more IMC formation, the slower hillock and whisker growth.

5:35 PM

Stress-Strain Behavior Of Pb-Free Solders over Strain Rates Ranging from 10^-6/s to 10^2/s: *Xu Nie*[1]; Dennis Chan[1]; Weinong Chen[1]; Ganesh Subbarayan[1]; Indranath Dutta[2]; [1]Purdue University; [2]Washington State University

An important need in predicting the reliability of electronic systems is to quantitatively understand the mechanical response of Pb-free solder joints to external loads ranging from creep to shock. In this research, we describe mechanical tests on Pb-free solders over a strain-rate range from 10^-6/s to 10^2/s using both quasi-static mechanical testers and a modified split Hopkinson pressure bar. Loading conditions in the dynamic experiments were controlled to subject the specimens to desired (constant) strain rates. We compare the saturation stress resulting from high-strain rate tests to that from previously conducted low-strain rate tests. We describe creep and viscoplastic constitutive models fit to the experimental data over nine decades of strain rates with reasonable agreement.

5:50 PM

Stress Relaxation Behavior In Sn And Pb-Sn Layers And Its Relation To Whiskering: *Jae Shin*[1]; Eric Chason[1]; [1]Brown University

We have used real-time wafer curvature measurements to study the relaxation kinetics of thermally-induced stresses in Sn and Pb-Sn thin films. We find that the relaxation behavior in Sn is well-described by a power law creep mechanism, with an exponent similar to that found in bulk Sn. However, the yield stress of the thin film is much higher than that of the bulk material. Additionally, the relaxation kinetics are thickness dependent so that thick layers relax more quickly than thin layers. Pb-Sn layers exhibit even faster relaxation behavior than pure Sn layers. The implications of the relaxation kinetics for whisker formation and mitigation will be discussed.

Peirce-Smith Converting Centennial Symposium: New Converting Technologies and Panel Discussion

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
Program Organizer: Joel Kapusta, Air Liquide Canada Inc

Tuesday PM Room: 2009
February 17, 2009 Location: Moscone West Convention Center

Session Chair: To Be Announced

2:00 PM
Continuous Converting of Copper Matte in Packed Bed Reactor: *Andrzej Warczok*[1]; Gabriel Riveros[1]; Juan Vargas[2]; Roberto Saez[2]; Arturo Tapia[3]; [1]Universidad de Chile; [2]ENAMI; [3]Pyros Ingenieros SA

The new process of continuous converting of copper matte directly to blister copper in packed bed reactor states an attractive option due to its very low investing cost, flexibility of operation with a liquid, solid or liquid and solid copper matte charge. Based on fundamental measurements of the rate of matte oxidation the mathematical model of matte converting in the ceramic packed bed has been developed. The alumino-olivine slag has been chosen because the auto-melting properties of the mix of fluxes: clay, quartz and lime. The results of tests in the laboratory demonstrative installation showed process feasibility and effective matte oxidation up to the production of over-oxidized copper. Currently, the industrial-pilot installation of capacity 5 t/h of copper matte is completed.

2:25 PM
The Validation of the Codelco-Chile Continuous Converting Process: *Alex Moyano*[1]; Carlos Caballero[1]; Claudio Toro[2]; Pedro Morales[1]; Jonkion Font[2]; [1]CODELCO - Chile; [2]IM2 a Codelco Subsidiary

Codelco-Chile in about a decade has carried out industrially-oriented research as an alternative to the traditional batch-type converting process. In a first stage, carried out preliminary evaluations in a rearranged PS converter, and then, an exploratory test in a CT (Teniente Converter) for the Codelco-Chile Continuous Converting Process. However, the validation test for this process in the CT, was delayed up to the second quarter of 2007 where the validation studies were started. Hence, the objective of this paper is to present the results of the campaign tests validating the Codelco-Chile Continuous Converting Process in the CT1 of the Codelco Norte smelter. The results of charging the solid or liquid matte or “white metal” by garr-gun, or by mouth of the 22 m x 5 m CT1 are presented. Some features of the industrially validated process are discussed, and the pilot test results of the matte injection are also presented.

2:50 PM
Continuous Improvement in P-S Converter Design - Kumera’s Approach: *Shaolong Chen*[1]; Hannu Mansikkaviita[1]; Markku Rytkonen[1]; Ilpo Kylmäkorpi[1]; [1]Kumera Technology Center

Though P-S converter is a traditional type of furnace, continuous improvement in its mechanical and electrical design is still Kumera’s approach. The efforts particularly made on the following aspects: converter head shape for easy fabrication and structural strengthening, oxidation air swivel joint and wind box for low pressure drop and low noise level and for even air distribution to tuyeres, drive units and its controls for reliable and handy operation. These improvements have successfully been applied in large units for copper and nickel smelters.

3:15 PM
Advantages of Continuous Copper Fire Refining in a Packed Bed: *Gabriel Riveros*[1]; Andrzej Warczok[1]; Daniel Smith[2]; Ariel Balocchi[2]; [1]Universidad de Chile; [2]ENAMI

Blister copper from converting of copper matte is processed in fire refining prior to electrorefining. The fire refining consists of two steps: copper oxidation with impurities slagging and copper reduction. The classical batch process is carried out in vascular or reverberatory furnaces called anode furnace. The new process of continuous copper fire refining has been proposed. The copper flows through two reactors in cascade, where is oxidized and reduced. The flow of a liquid copper through a ceramic packed bed increases the surface area and the rate of oxidation as well as the rate of reduction in the packed bed of charcoal. Mathematical model allowed for design a laboratory scale demonstrating installation and industrial-pilot installation in Hernan Videla Lira Smelter (Paipote). The results demonstrated the process feasibility and flexibility in operation.

3:40 PM Break

3:55 PM
Operation of the Air Liquide Shrouded Injector (ALSI) Technology in a Hoboken Siphon Converter: *Romeo Pagador*[1]; Noparut Wachgama[1]; Chumnoom Khuankla[1]; Joel Kapusta[2]; [1]Thai Copper Industries Public Company Limited; [2]Air Liquide Canada Inc

Thai Copper Industries (TCI) is a custom copper smelter and refinery in Thailand with an annual capacity of 165,000 MT cathodes. It employs the El Teniente Technology as the primary smelting reactor to produce white metal from copper concentrates. The white metal is further treated in a Hoboken Siphon Converter to produce blister copper, refined in anode vessels and cast into anodes for the tankhouse. TCI is the first copper smelter to install and operate in commercial scale the Air Liquide Shrouded Injectors (ALSI) Technology on the Hokoben converters for sonic injection of oxygen enriched air. This paper will present the actual operational results in terms of blowing cycles and blowing efficiencies, reverts recycling, tuyere brick life and other parameters pertaining to the converting operation. The outstanding operation of Hoboken converter with the ALSI technology complemented the TCI Plant production to reach over 75% capacity within four months.

4:20 PM
Flash Converting: Ilkka Kojo[1]; *Markku Lahtinen*[1]; Elli Miettinen[1]; [1]Outotec Oyj

The recent trends of decreasing energy consumption and environmental emissions and utililisation of economies of scale are strong drivers favoring continuous copper converting processes. Flash Converting benefits from low off-gas volume and investment and operational cost to off-gas treatment. Separate matte and blister furnaces allow adoption to concentrate quality changes and flexibility in layout and maintenance. The stationary blister copper bath in Flash Converting furnace is less aggressive to the furnace linings than the agitated processes, resulting in low refractory consumption, long campaign times and high on-line availability. Copper Flash Conveting has been successfully applied in Kennecott with campaign time now exceeding five years. A second FCF was started at Yanggu in 2006.The process itself is proven, as its features are similar in FSF, Directto Blister and Nickel Flash Smelting processes, with one significant difference: it is easiest to operate of all of them.The paper presents differences with continuous Flash Converting and conventional converting based on recent experiences and studies.

4:45 PM
Ausmelt C3 Converting: *Jacob Wood*[1]; Robert Matusewicz[1]; Markus Reuter[1]; [1]Ausmelt Limited

Over the last 20 years, significant improvements in copper smelter productivity have been realised through the advent of continuous smelting processes. Although Peirce-Smith converting has been the dominant technology to date, continuous converting permits large scale throughput in single vessels and improved environmental control. A number of continuous converting technologies are currently in use or are being developed in the copper industry, mostly based on calcium-ferrite slags. However, a notable exception is the Ausmelt Continuous Copper Converting (C3) technology which is based on olivine type slags.This paper discusses the merits of the Ausmelt C3 process and the advantages offered in terms of operational flexibility and process control arising from adoption of the ferrous calcium silicate slag system. Based on theoretical considerations the paper discusses key findings and observations relating to slag chemistry which have been taken from pilot-scale testwork and commercial operating experience.

5:10 PM Panel Discussion

5:50 PM Concluding Comments

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials VIII: Session IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic 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; Daniel J. Lewis, Rensselaer Polytechnic Institute; Kejun Zeng, Texas Instruments; Wojciech Gierlotka, AGH University of Science and Technology; Yee-wen Yen, National Taiwan University of Science and Technology

Tuesday PM February 17, 2009 — Room: 2022 — Location: Moscone West Convention Center

Session Chairs: Clemens Schmetterer, University of Vienna; Yee-wen Yen, National Taiwan University of Science and Technology

2:00 PM Invited
Interesting Phenomena Observed In The Sn/Co Interfacial Reactions: *Sinn-wen Chen*[1]; Chao-hong Wang[2]; [1]National Tsing Hua University [2]National Chung-cheng University

The CoSn3 phase was formed along the Sn/Co interface in the Sn/Co couples reacted at 150 to 200°C. The reaction layers grew linearly with reaction time in the early stage, and then it changed to a parabolic growth when the layer reached a critical thickness. However, at the corners of the Co substrates in the Sn/Co couples, the reaction phase was the CoSn4 phase at 180°C; cracking was observed and there were no reaction phases at 200°C. The reaction phase layer showed a unique cruciform pattern. The cruciform pattern was formed either by cracking or transformation to the CoSn4 phase at the corners where stress was most intensified. With the passage of electric current from the Co toward the Sn side, the CoSn3 phase was thicker than that without the passage of electric current. A significant Peltier effect has been observed which is responsible for the different reaction layer growth.

2:20 PM
Solid/Solid Interfacial Reactions between Sn and Ni-Co Alloys: *Chih-chi Chen*[1]; Jenq-Gong Duh[2]; Sinn-wen Chen[2]; [1]Chung Yuan Christian University; [2]National Tsing Hua University

Nickel is a commonly used barrier layer material of under bump metallurgy (UBM) in flip chip packaging because of its low reactivity with solders. Tin is the primary constituent element of solders. Recent investigations indicate cobalt is a potential diffusion barrier material for copper. Therefore, Ni-Co alloys are potential diffusion barrier materials of UBM for integrated circuits (I.C.) with Cu/low k process. There have been some studies upon Sn/Co and Sn/Ni-Co interfacial reactions. However, Sn/Ni-Co interfacial reactions at solid state have not been examined. This work investigates the interfacial reactions between Sn and Ni-Co alloys at solid state. Reactions at 120, 150 and 200oC are carried out, and the compositions of Ni-Co alloys examined are Ni-5at%Co, Ni-20at%Co and Ni-40at%Co, respectively. The preliminary experimental result shows that a meta-stable phase, Co3Sn7, is formed. Based on the available Sn-Ni-Co isothermal section, the reaction paths of the Sn/Ni-Co couples can be determined.

2:35 PM
Interfacial Reaction Between Sn Solder And NiCo UBM: *JyunWei Cheng*[1]; Chengyi Liu[1]; [1]National Central University

Cu-based UBM has been widely used in the electronic package. During soldering, Cu would dissolve seriously into the molten solder, which would cause reliability issue of the solder joints. Therefore, a reaction barrier often requires to prevent the fast reaction between solder and Cu pad. Ni(P) is the one used as the reaction barrier layer for last decade. Ni(P) also cause many reliability issues, for example, black pad and Ni3P crystalline layer formation. In the work, we study soldering reaction between Ni-Co alloy layer and Sn Pb-free solders. Different Co concentrations in Ni-Co alloy layer were electroplated on Cu foils. Then, Sn solders were reflowed on the Ni-Co alloy layers to investigate the interfacial reactions. We found that different Co concentration in Ni-Co alloy layers will result in different the reaction phase at the interface. The formation kinetics of Ni-Co-Sn ternary intermetallic compound (IMCs) at the interface will be reported.

2:50 PM
A Study of Interfacial Reaction between Molten Sn-Ag Solder and Te Substrate: *Yen-Chun Huang*[1]; Chien-Neng Liao[1]; [1]National Tsing Hua University

Telluride-based thermoelements can react with Sn-contained solders and form SnTe intermetallic compounds that may deteriorate electrical and mechanical properties of soldered junctions. In addition to Ni barrier approach, we may also change the recipe of solder alloys to suppress or slow down the formation of SnTe compounds. In this study the effect of Ag addition (0.1, 1, 3.5, 5 wt%) in pure Sn on the interfacial reaction between molten solder and Te substrate is explored. It is found that the thickness of SnTe compound is reduced after soldering reaction when Ag is added into Sn solder. The suppression of SnTe compound formation may be associated to the presence of Ag3Te2 and Ag-Sn-Te ternary compounds that are located in between the SnTe compound and the Te substrate. Besides, the thickness of the planar-type Ag3Te2 compound is found to increase with increasing Ag content.

3:05 PM
Effect of Cu Addition in Sn Solder on the Interfacial Reaction with Elemental Te Substrate: *Ching-Hua Lee*[1]; Chien-Neng Liao[1]; [1]National Tsing Hua University

Conventional telluride based thermoelectric elements can easily react with Sn-contained solder and form SnTe intermetallic compounds. The interfacial compounds are rather brittle and may lead to the failures of the soldered junctions under normal heating-to-cooling operations. Thus, modification of solder alloy recipe may suppress the interfacial compound formation. In this study the reaction of pure Te elements with Sn(Cu) molten solder is explored. The preliminary results showed that a tiny amount of Cu addition can suppress the vigorous Sn/Te interfacial reaction effectively. Moreover, a compound phase, CuTe, tends to form in between the SnTe phase and the Te substrate. The effect of Cu content in Sn(Cu) solder alloy on the growth mechanism of SnTe and CuTe interfacial compounds will be investigated. The influence of the interfacial compounds on the electrical properties of the soldered junction is also a subject of interests.

3:20 PM Break

3:40 PM Invited
Interfacial Reactions and Microstructures of Sn-0.7Cu-xZn Solders with Ni-P UBM during Thermal Aging: Moon Gi Cho[1]; Sung K. Kang[2]; Da-Yuan Shih[2]; *Hyuck Mo Lee*[1]; [1]Korea Advanced Institute of Science and Technology [2]IBM T.J. Watson Research Center

The effects of Zn addition to Sn-0.7Cu are investigated, focusing on their interfacial reactions, microstructure and hardness when reacted with Ni-P. The Zn content in Sn-0.7Cu-xZn varies as 0.2, 0.4 and 0.8 (in wt %). In the reaction with Ni-P, $(Cu,Ni)_6Sn_5$ intermetallic compounds(IMCs) are formed at the interface, regardless of the Zn content. As the Zn content increases, the growth of $(Cu,Ni)_6Sn_5$ during aging is gradually reduced, yielding a reduction of 40-50% for 0.8% Zn. $(Cu,Ni)_6Sn_5$ IMCs are also commonly observed in the solder matrix. In Sn-0.7Cu, $(Cu,Ni)_6Sn_5$ particles coarsen largely during aging, while in the Zn-added solders, they are much smaller initially and resistant to growth. This explains the stable microhardness of the Zn-added solders during aging. To understand the $(Cu,Ni)_6Sn_5$ IMCs formed in the Zn-added solders, TEM studies are conducted. The microstructure and hardness of the Zn-added solders are further discussed with thermodynamic calculations and analytical works.

4:00 PM
TEM Observations of Cu3Sn Growth at SnBi/Cu Interface: P. Shang[1]; Z. Liu[1]; *J. Shang*[2]; [1]Institute of Metal Research; [2]University of Illinois at Urbana-Champaign

Transmission electron microscopy (TEM) studies were made to observe the growth of Cu3Sn intermetallic phase at SnBi/Cu interface during reflow and solid-state aging process. Two types of Cu-Sn intermetallics, Cu6Sn5 and Cu3Sn, were found at the interface after reflow. In the early stage of solid state aging, Cu3Sn assumed columnar growth grains along [100] direction of Cu. With further aging, new triangle Cu3Sn grains were uncleated at the triple junction sites of Cu/Cu3Sn interface, resulting in two distinct Cu3Sn layers between Cu and Cu6Sn5 layer. Along the Cu3Sn/Cu interface, Bi segregation was detected following prolonged solid-state aging.

4:15 PM
Oscillatory Growth Of The Ni3Si2 Intermetallic Compound In Reactive Interdiffusion Of Thin Ni Film On Si Substrate: *Delphine Borivent*[1]; Bernard Billia[1]; [1]IM2NP

The formation of Ni3Si2 in solid-state reactive diffusion of a Ni film deposited on a <100>-oriented silicon substrate was followed in time by Bragg-Brentano X-ray diffraction, and further investigated by X-ray microdiffraction, optical and scanning electron microscopy. Concentric rings form from nucleation centres in the lateral propagation of the Ni3Si2 phase revealing an oscillatory growth velocity. Similar ripples have already been observed in the explosive crystallization of amorphous films of Si. Yet, the propagation of the Ni3Si2 front is too slow for heat-transport control to hold, which suggests that it is rather the diffusion of chemical species that is driving the patterning of the Ni3Si2 phase into concentric rings. The synthesis of the experimental data allows us to propose a model the Bradley's way (R.M. Bradley, J. Appl. Phys. 60 (1986) 3146) based on a source-sink mechanism for silicon possibly leading to oscillatory instability of disk growth of Ni3Si2.

4:30 PM
Interfacial Reaction Effect on Mechanical and Electrical Reliability in Cu Pillar Bump: *Gi-Tae Lim*[1]; Byoung-Joon Kim[2]; Ki-Wook Lee[3]; Jae-Dong Kim[3]; Young-Chang Joo[2]; Young-Bae Park[1]; [1]Andong National University [2]Seoul National University [3]Amkor Technology Korea, Inc.

Flip chip solder bump has been widely used as a key interconnection technology of high performance devices. As the integration of devices increased, the size of solder bump became smaller with fine pitch. And increase of current density due to miniaturization of solder bump size with fine pitch causes serious reliability issues. Cu pillar bump is one of candidates to solve reliability issues because it provides the fine pitch and uniform current distribution. However, excessive intermetallic compound and Kirkendall void growth in Cu pillar bump can degrade the mechanical reliability of solder joints. Therefore, it is necessary to understand the growth kinetics of intermetallic compound and Kirkendall void of Cu pillar bump. In this work, intermetallic compound and Kirkendall void growth kinetics in Cu pillar bump have been studied using in-situ scanning electron microscope during annealing and electromigration. Also, their effects on the mechanical reliability will be discussed in detail.

4:45 PM
Analysis of Sintering Aids for High Melting Rare Earth-Iron-Boron Magnet Alloys: *Nathaniel Oster*[1]; Iver Anderson[2]; Wei Tang[2]; Yaqiao Wu[2]; Kevin Dennis[2]; Matthew Kramer[2]; R. McCallum[2]; [1]Iowa State University [2]Ames Laboratory

Many Rare Earth (RE)-Iron-Boron magnet alloys display a relatively low-melting (650-700°C) ternary eutectic. Nd-Fe-B is among these. The liquid formed from the eutectic promotes sintering of the particulate during processing of aligned, fully-dense sintered magnets. However, several alternative RE-Fe-B magnet alloys, such as Dy-Fe-B, do not exhibit this low-melting eutectic. Since the dominant $RE_2Fe_{14}B$ phase in such particulate is a brittle intermetallic that melts much higher (about 1250°C), solid state sintering is difficult. Cu-base, Al-base, and Nd-Fe alloys have been proposed as possible systems that could be added to the magnet particulate in order to aid sintering. The wetting, bonding, and diffusive interaction of these alloys with a RE-Fe-B magnet alloy particulate will be the object of microstructure analysis of the sintered compacts by SEM, X-ray diffraction, and electron microprobe. Additionally, magnetic properties of the sintered magnets will be measured. Supported by DOE-EERE-FCVT Office through Ames Lab contract DE-AC02-07CH11358.

Recycling of Electronic Wastes: General Recycling

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS Materials Processing and Manufacturing Division, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Lifeng Zhang, Missouri University Fay Hua, Intel Corp; Oladele Ogunseitan, University of California; Gregory Krumdick, Argonne National Laboratory

Tuesday PM — Room: 2024
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: Fay Hua, Intel Corp

2:00 PM Introductory Comments

2:05 PM
Assessment of Public Health Impacts of Open Air Incineration of Electronic Wastes: Kathleen Hibbert[1]; Oladele Ogunseitan[1]; [1]University of California, Irvine

Electronic waste (E-waste) is the fastest growing source of hazardous solid waste. Americans discarded about 2 million tons of E-waste annually. The U.S. lacks comprehensive federal policies to reduce toxic materials in electronics manufacturing or for E-waste disposal. Furthermore, the U.S. is not a signatory to international regulation of transboundary shipment of hazardous waste, posing risks in developing countries, where waste processing is decentralized. Studies conducted by US EPA suggested that incineration of e-waste emits polybrominated dibenzofurans (PBDFs), and toxic metals such as lead, cadmium, copper, manganese and beryllium. We are analyzing potentially toxic exposures from incineration of cellular phones because they contain analogous components for generic e-waste. A representative sample of 100 cellular phones is being tested under conditions simulating open-air incineration. Emitted gases and ash residue will be analyzed for priority pollutants according to Occupational Safety and Health Administration standards, and the Clean Air Act.

2:25 PM Question and Answer Period

2:35 PM
Separation of Nickel, Cobalt and Magnesium from the Spent Mh-Ni and Cd-Ni Battery Anode Materials: Xu Shengming[1]; Wang Gehua[1]; *Zhang Lifeng*[2]; Li Linyan[1]; Xu Gang[1]; Liu Xiaobu[1]; [1]Tsinghua University; [2]Missouri University of Science and Technology

Reductive leaching kinetics of LiCoO2 from spent lithium ion battery were investigated in sulfur acid solutions. The leaching efficiency of LiCoO2 increased with increasing temperature, and concentration of H2SO4, but with increasing the ratio of liquid to solid. With the addition of H2O2 or Na2SO3 as a reducing agent,the leaching efficency of Li and Co from LiCoO2 can obtain over 95% due to the reduction of Co3+ to Co2+ which can be readily dissolved. Apparent activation energies in the two reductive leaching systems were obtained for Co and Li, respectively.

2:55 PM Question and Answer Period

3:05 PM
LCD (Liquid Crystal Display) Separation Aiming Recycling: *Viviane Tavares*[1]; Jorge Tenório[1]; Denise Espinosa[1]; [1]Escola Politecnica da Universidade de São Paulo

The liquid crystal display (LCD) are currently known by its vast application in electro – electronic devices, amongst them can be detached the TV's devices, laptops, electronic date, calculators and even though the mobile. The mobiles are changed more frequent of the TV, this happens due to the technology advance. With the constantly mobile exchange, the batteries, printed circuit board and liquid crystal display accumulation its become an environmental problem. To minimize the environmental impacts caused by mobile telephones residues are considered the segregation process of the LCDs main components, to goal to recover the recycle material. This process involves the use of treatment ore techniques with disc, hammers and balls mills. The glass recovered can be used in the glass recycling and the sand substitution in the production of concrete blocks.

3:25 PM Question and Answer Period

3:35 PM
Evaluation of Alkaline Battery End-of-life Strategies: *Elsa Olivetti*[1]; Edgar Blanco[1]; Jeffrey Dahmus[1]; Jeremy Gregory[1]; Randolph Kirchain[1]; [1]MIT

Approximately 80% of batteries manufactured worldwide are so-called alkaline dry cells with a global annual production exceeding 10 billion units. Today, the majority of these batteries go to landfills at end-of-life. An increased focus on environmental issues related to battery disposal, along with recently implemented battery directives in Europe and Canada, has intensified discussions about end-of-life battery regulations globally. The logistics of battery collection are intensive given the large quantity retired annually, their broad dispersion, and the small size of each battery. Careful evaluation of the economic and environmental impacts of battery recycling is critical to determining the conditions under which recycling should occur. This work compares a baseline scenario involving landfilling of alkaline batteries as municipal solid waste with several collection schemes for battery recycling through pyrometallurgical material recovery. Network models and life cycle assessment methods enable the evaluation of various end-of-life collection and treatment scenarios for alkaline batteries.

3:55 PM Question and Answer Period

4:05 PM
Green Recycling of EEE: Special And Precious Metal Recovery From EEE: *Christina Meskers*[1]; Christian Hagelueken[1]; [1]Umicore

EEE contains a range of components made of a wide variation of metals, plastics and other substances. Over 40 elements can be found in complex electronic equipment: base metals, precious metals and special metals in circuit boards and batteries. Because of its amount WEEE represents a considerable metal resource with a higher special and precious metal content than found in ores. The Umicore integrated smelter and refinery recovers and supplies back to the market 17 different metals from EEE. Pre-treated materials enter state of the art, material and energy efficient metallurgical processes for environmentally sound recovery of metals and treatment of off gasses and hazardous substances. The organics in the feed function as reducing agent and alternative energy source during smelting for precious metal recovery. Lithium-ion batteries are treated using a dedicated process. As a result the environmental footprint of metals produced from EEE is much smaller than primary production.

4:25 PM Question and Answer Period

4:35 PM
Research On The Recovery Of Organic Acid From Cyclohexanone Waste: daowu yang[1]; *Linping Yu*[1]; Ping Yu[2]; Yunbai Luo[2]; [1]Changsha University of Science and Techonology; [2]Wuhan University

A simplified ED process was developed for organic acids recovery from cyclohexanone waste in this paper. In this regard, the one-stage ED was investigated directly without pretreatment, such as ion-exchange or nanofiltration to removal of foulants. The current efficiency and the energy consumption in the ED process for the recovery of carboxylic acids from cyclohexanone waste is theoretically analyzed and experimentally tested. The concentration of recovered carboxylic acid is related to many parameters and still needs more work to be determined.

4:55 PM Question and Answer Period

Shape Casting: Third International Symposium: Novel Methods and Applications

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Solidification Committee, TMS: Aluminum Processing Committee
Program Organizers: John Campbell, University of Birmingham; Paul Crepeau, General Motors Corp; Murat Tiryakioglu, Robert Morris University

Tuesday PM Room: 2011
February 17, 2009 Location: Moscone West Convention Center

Session Chair: Mahi Sahoo, CANMET Materials Technology Laboratory

2:00 PM Introductory Comments

2:10 PM
Ablation Casting Up-Date: John Grassi[1]; *John Campbell*[1]; Martin Hartlieb[2]; Fred Major[2]; [1]Alotech Limited; [2]Rio Tinto Alcan

Light alloy castings made in an aggregate mold are ablated with coolant to erode away the mold by dissolving the mold binder. The coolant thereby gains access to the surface of the casting prior to extensive solidification, avoiding normal limitations to heat transfer via the ‘air gap’, and thus confers otherwise unattainable rates of cooling. The process was first announced at the previous TMS Annual Congress, and is here updated after this additional year. The unique structures and properties are described.

2:35 PM
The Nemak Cosworth Casting Process - Innovation: *Glenn Byczynski*[1]; Robert Mackay[2]; [1]Nemak Europe GmbH; [2]Nemak Canada

The Cosworth Process is well recognized for its ability to produce high quality, dimensionally accurate aluminum castings. The process was designed from first principles with casting quality as the main focus. Ford transformed this process into a high volume production system in the early 1990’s at its Windsor Aluminum Plant and this plant continues to manufacture world-class aluminum cylinder block castings today as Nemak Canada. The one drawback (if any) of the original process is the resultant microstructure due to the relatively slow solidification rate in heavy sections in the sand mould. The secondary dendrite arm spacing combined with typical automotive grade alloys limits the mechanical properties in certain areas of the casting. The innovation discussed in this paper is an augmentation of the original Cosworth Process to include an integral chill that increases local solidification rates and drives casting performance to new levels.

3:00 PM
Development of an Aluminum Alloy for Elevated Temperature Applications: *Kumar Sadayappan*[1]; David Weiss[2]; Mahi Sahoo[1]; Gerald Gegel[3]; [1]CANMET - Materials Technology Laboratory; [2]Eck Industries; [3]Material and Process Technologies

The need to reduce the exhaust emissions of medium and heavy-duty diesel engines has lead to the use a two-stage series turbocharger design for the air system. The current single stage compressors run at an outlet air temperature of approximately 175°C at sea level. This is the approximate temperature limit for the currently used 354-T61 aluminum alloy impellers. The second stage outlet air temperatures are predicted to reach 260°C or higher at sea level conditions and this temperature will increase with altitude. The maximum operating temperature of most current structural aluminum alloys is about 200°C which is equal to their aging temperatures (approximately 200°C). Efforts were made to develop an Al-Cu alloy with scandium addition which is able to retain its strength at 250°C. The details of the alloy development and the results are presented and discussed in this publication.

3:25 PM
Controlled Diffusion Solidification (CDS): Conditions for Non-Dendritic Primary Aluminum Phase Al-Cu Hypo-Eutectic Alloys: *Abbas Khalaf*[1]; Peyman Ashtari[1]; Sumanth Shankar[1]; [1]LMCRC - McMaster University

Controlled Diffusion Solidification (CDS) is a novel process wherein a non-dendritic primary aluminum phase is obtained in Al-Cu hypo-eutectic alloys by controlled solidification of two precursor liquid alloys and casting the resultant alloy. In CDS, the non-dendritic primary Al phase will enable a continuous network of inter-dendritic liquid during solidification of the mushy zone, thereby mitigating the hot-tearing tendencies typically exhibited by Al-Cu alloys with low solute concentrations. Hence, the process will enable near-net shaped casting of Al-Cu based wrought alloy compositions which exhibit superior mechanical and performance properties. In this paper, a hypothesis explaining the complex mechanism of nucleation and growth of the primary Al phase to result in a non-dendritic morphology will be presented. Critical parameters such as temperatures, mass ratios and rate of mixing of the two precursor liquids to obtain various compositions of Al-Cu hypo-eutectic alloys will be presented to support the suggested mechanism.

3:50 PM Break

4:00 PM
Favorable Alloy Compositions and Melt Temperatures to Cast 2XXX and 7XXX Al alloys by Controlled Diffusion Solidification (CDS): *Peyman Ashtari*[1]; Gabriel Birsan[1]; Sumanth Shankar[1]; [1]LMCRC-McMaster University

Controlled diffusion solidification (CDS) is an innovative rheocasting (Semi-solid) processing route to obtain a cast part with a non-dendritic morphology of the primary Al phase. The process involves mixing two alloy melts with specific individual compositions and temperatures to produce the desired final alloy by mixing and immediately casting in a mold. The process enables the shape casting of Al based wrought alloy along with their superior cast properties and performance. The present work defines process conditions to enable shaped casting of the 2XXX and 7XXX series of Al based wrought alloys, specifically, 2024, 7005 and 7075 alloys.

4:25 PM
The Application of Positron Emission Particle Tracking (PEPT) to Study the Movement of Inclusions in Shape Castings: William Griffiths[1]; *Y. Beshay*[2]; D. J. Parker[1]; X. Fan[1]; M. Hausard[3]; [1]University of Birmingham; [2]University of Birmingham - and - Beshay Steel; [3]Formerly of the University of Birmingham; currently at Centre de Calcul de l'Institut National de Physique Nucléaire et de Physique des Particules

Positron Emission Particle Tracking (PEPT) was used to track radioactive particles entrained into castings during mould filling. The purpose of these experiments was to test the technique for its application to the study of inclusion movement in castings, and so provide a method for validation of computer simulations of inclusion behaviour. Two types of experiments were carried out, one using Al alloy plate castings made in resin-bonded sand moulds, into which were entrained radioactive alumina particles of size 325 to 710 µm. A second type of experiment used smaller alumina and resin particles, around 50 to 100 µm in size, entrained into a low melting point In alloy, (Field's Metal), cast at 80°C into an acrylic die. In each experiment the particles locations were recorded in real time, using a positron detection camera. The particle paths were obtained for each casting and the reproducibility of the technique determined.

4:50 PM
Microstructural and Surficial Characteristics of Lead Free Bismuth Bronze Produced through the Frozen Mold Casting Process: *Shuji Tada*[1]; Hiroyuki Nakayama[1]; Toshiyuki Nishio[1]; Keizo Kobayashi[1]; [1]National Institute of Advanced Industrial Science and Technology

The frozen mold is a kind of sand mold which is produced by freezing the mixture of sand and water. The frozen mold casting process has the possibility to reduce the environmental load and the rapid cooling effect on cast products is expected. The effect of cooling rate on the microstructure of produced bronze cast was investigated. The frozen mold indicated better cooling property compared with conventional green sand mold. The microstructure of bronze cast produced through the frozen mold casting process was refined in the thinner sample but the quenching effect did not work well in the thicker sample. The surface condition of bronze cast was also examined. The surface of bronze cast produced using frozen mold consisting of only sand and water was rather rough. The surface roughness, however, was improved by adding colloidal silica solution into the sand mixture.

5:15 PM
High Pressure Die Cast of Semi-Solid Ductile Cast Iron: Bashir Heidarian[1]; *Mahmoud Nili-Ahmadabadi*[1]; Marzieh Moradi[1]; Jafar Rassizadeghani[1]; [1]University of Tehran

The processing of metals in the semi-solid state is becoming an innovative technology for the production of globular structure and high quality cast parts. Ductile irons because of spherical graphite have specific properties such as good mechanical properties, strength and toughness together and suitable castability. This engineering alloy along with growing application has several shortcoming which had limited its applications such as non-formability, dendritic structure and alloying element segregation, micro-porosity resulted from solidification mode and fabrication of thin section parts. It seems that replacing dendritic structure with globular structure and thixoforming, results in improving of mechanical properties, controlling of alloying element segregation, decreasing of micro-porosity and increasing of ability to thin section filling. In this paper high pressure mold filling of ductile iron contains Mn and Mo in semi-solid state has been investigated. Filling properties, fluidity, liquid segregation, alloying element segregation and defects like shrinkages holes and cracks were characterized.

Structural Materials Division Symposium: Advanced Characterization and Modeling of Phase Transformations in Metals in Honor of David N. Seidman on his 70th Birthday: Kinetics of Phase Transformations I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Robert Averback, University of Illinois, Urbana-Champaign; Mark Asta, University of California, Davis; David Dunand, Northwestern University; Ian Robertson, University of Illinois at Urbana-Champaign; Stephen Foiles

Tuesday PM — Room: 3000
February 17, 2009 — Location: Moscone West Convention Center

Session Chair: David Dunand, Northwestern University

2:00 PM Invited
Coarsening Kinetics With Large Diffusional Mobility Disparity In Two Phases: Guang Sheng[1]; Qiang Du[1]; Kegang Wang[2]; Zikui Liu[1]; *Long Qing Chen*[1]; [1]Pennsylvania State University; [2]Florida Institute of Technology

Precipitation is a basic process underlying the development of many engineering alloys ranging from high-temperature Ni-based superalloys to light-weight Al-alloys. It is generally known that the diffusion coefficients of elements responsible for coarsening are dramatically different in the precipitate phase and in the matrix. It has recently been suggested that such dramatic difference in diffusivities may result in fundamentally different coarsening kinetics. In this presentation, we will discuss our recent simulation results of coarsening kinetics with large diffusional mobility disparity using a variable-mobility Cahn-Hilliard equation. We study the coarsening kinetics of both interconnected two-phase microstructures and isolated precipitate particles embedded in a matrix. Our preliminary simulations for both interconnected morphologies and isolated particles suggest a coarsening power law with an time exponent of ~ 0.30, which is surprisingly close to that obtained by Seidman's group for the coarsening of gamma-prime precipitates in Ni-based alloys using the three-dimensional atom probe technique.

2:30 PM Invited
High Resolution Electron Microscopy of Core/Shell Precipitates in Al-Based Alloys: *Ulrich Dahmen*[1]; M. D. Rossell[1]; R. Erni[1]; M. Watanabe[1]; V. Radmilovic[1]; [1]National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, University of California

Core-shell precipitate structures have recently been demonstrated in Al-based alloys with Sc and Zr. It was shown that a Zr rich shell surrounded an Al3Sc core, acting as a diffusion barrier that reduced the growth rate of the Sc rich core. In this work, we investigate the effect of Li addition to AlScZr alloys. Using aberration-corrected high resolution transmission electron microscopy, the role of Li as a transient nucleating agent for Sc and Zr during heating was documented, and the formation of an Al3Li shell around (Sc,Zr)-rich particles was observed. It was possible to directly image Li atom columns in the shell from the phase of the exit surface wavefunction. Likewise, Sc and Zr-containing columns in the core could be seen directly using high-angle annular dark field imaging. Finally, combining a monochromated source with aberration-corrected energy-filtered imaging, we were able to obtain spectrum images of plasmon peaks that provided a map of Li concentration in these precipitates. Our observations are consistent with a multi-stage precipitation mechanism. Initially, spinodal decomposition serves as a barrier-free process to grow evenly-spaced Li-rich clusters by congruent ordering, acting as heterogeneous nucleation sites for the formation of Sc-rich precipitates at high temperature. During subsequent low-temperature annealing, Li forms a shell around these particles. The resulting microstructure is remarkably monodisperse. This approach to generating precipitate distributions can be applied to a range of alloys and could lead to new types of dispersion-strengthened materials.

3:00 PM
Study of Precipitation Kinetics of Copper in HSLA Steel by TEM Small Angle Neutron Scattering (SANS): *Chandra Pande*[1]; M. Ashraf Imam[1]; [1]Naval Research Laboratory

Precipitation kinetic of copper in a high strength low –carbon ferrous alloy has been studied in the past by Prof. Seidman using field ion microscopy. These precipitates are initially coherent with the matrix and hence are difficult to detect by conventional transmission electron microscopy (TEM). We have therefore used TEM in conjunction with small angle neutron scattering (SANS) to study copper precipitation. Direct measurement from TEM micrographs and integral transform of the SANS data was used to calculate the size distribution for a variety of aging conditions. Maximum entropy principle was used to refine the distribution obtained. The role of these precipitates in hardening of the material will also be considered.

3:15 PM Break

3:30 PM Invited
The Topology and Morphology of Three-Dimensional Bicontinuous Interfaces: A. Genau[1]; Y. Kwon[2]; K. Thornton[3]; *Peter Voorhees*[1]; [1]Northwestern University [2]Samsung Electronics; [3]University of Michigan

We examine the topology and morphology of interfaces produced following phase separation via spinodal decomposition and phase ordering. We employ three-dimensional simulations to examine the evolution of these systems during coarsening. We quantify the morphology of these complex microstructures via the interfacial shape distribution, the probability of finding a patch of interface with a given pair of principle curvatures. We also characterize the spatial correlations of the interfacial curvature. This analysis has indentified new characteristic length scales of these complex structures. In the structure produced following phase ordering, despite the local evolution law governing interfacial motion, long-range correlations develop that lead to a characteristic length scale associated with the distance between high-curvature tunnels. In the structure produced following spinodal decomposition the diffusional dynamics leads to a length scale that is related to correlations and anticorrelations between regions of curvature of opposite sign.

4:00 PM Invited
Solute-Vacancy Interaction in Al and Mg Alloys: *Christopher Wolverton*[1]; Dongwon Shin[1]; [1]Northwestern University

Solute-vacancy binding is a key quantity in understanding diffusion kinetics, and also can have a considerable impact on age hardening response in alloys. Previous efforts to understand solute–vacancy binding in alloys have been hampered by a scarcity of reliable, quantitative experimental measurements. Here, we report a large database of solute–vacancy binding energies determined from first-principles density functional calculations for both Al and Mg alloys. The calculated binding energies agree well with accurate measurements where available, and provide an accurate predictor of solute–vacancy binding in other systems. For both Al and Mg, we have explored the physical effects controlling solute–vacancy binding. We find that there is a strong correlation between binding energy and solute size, with larger solute atoms possessing a stronger binding with vacancies.

4:30 PM
Solute Segregation and Thermal Stability of Ultra-Fine-Grained Al-Mg: *Richard Karnesky*[1]; Nancy Yang[1]; Christopher San Marchi[1]; Enrique Lavernia[2]; [1]Sandia National Laboratories; [2]University of California, Davis

The effect of hot vacuum degassing, consolidation, and annealing on ultra-fine-grained (d≈200 nm) Al-7.5 wt. % Mg, produced from cryomilled, nanocrystalline (d≈50 nm) powders is studied by means of X-ray diffraction and local-electrode atom-probe tomography. Tomographic reconstructions of the powders, mounted and milled with a dual-beam SEM/FIB, show that the majority of grain growth and solute segregation to grain boundaries occurs during the thermal degassing prior to consolidation. The documented Mg segregation provides a possible explanation for the thermal stability of the materials, as the post-consolidated grain size and solute distribution changes very little when the alloy is annealed at 500°C for 2 hours.

4:45 PM Invited
Roles of Interface Width and Chemical Diffusion in Particle Coarsening: *Alan Ardell*[1]; [1]University of California

Experiments conducted by David Seidman and his students and other co-workers have provided valuable insights into the nature of the interfaces between precipitate and matrix phases. In Ni-base alloys containing γ' precipitates, typified by Ni_3Al in binary Ni-Al alloys but also including other binary as well as ternary alloys, the interfaces are not sharp, the composition across them changing over distances the order of 2 nm or so. Chemical diffusion across the interfaces influences the growth rates of individual precipitates, hence ultimately the kinetics of coarsening of the entire ensemble. Since chemical diffusion in the ordered precipitate phase is much slower than in the disordered solid solution, the interface acts as a diffusion bottleneck. A recent theory of coarsening takes into account diffusion across the interface. In this presentation extant data, including measurements originating in Professor Seidman's laboratory, will be examined in light of this new theory.

5:15 PM
Probing the Early Stages of Elemental Partitioning During the Nucleation and Growth of Alpha Platelets in the Beta Matrix of Titanium Alloys: Soumya Nag[1]; *Rajarshi Banerjee*[1]; Junyeon Hwang[1]; Srinivasan Rajagopalan[2]; Hamish Fraser[2]; [1]University of North Texas; [2]Ohio State University

The solid-state precipitation of the alpha phase within the beta matrix of titanium alloys involves both a structural bcc to hcp transformation as well as the diffusional partitioning of the alloying elements. Developments in advanced characterization techniques such as high-resolution scanning transmission electron microscopy (HRSTEM) and 3D atom probe (3DAP) tomography allow for unprecedented insights into the true atomic scale structure and chemistry changes associated with the precipitation of alpha as a function of heat-treatments. Such a coupling of 3DAP and TEM observations, on complex beta titanium alloys, indicate that the structural component of the beta to alpha transformation precedes the diffusional partitioning of the alloying elements. Thus, platelet-shaped alpha precipitates of a composition near that of the beta matrix, far from the equilibrium alpha composition, nucleate and grow by what appears to be a mixed mode (displacive + diffusive) transformation, similar to the bainite transformation in steels.

5:30 PM
Partitioning And Site Preference Of Transition Metals (Cr, Ta, Ru, Re) In Model Ni-Based Superalloys: An Atom-Probe Tomographic And First-Principles Study: *Zugang Mao*[1]; Christopher Booth-Morrison[1]; Yang Zhou[1]; David Seidman[1]; [1]Northwestern University

The site substitution and partitioning behavior of transition metals (Cr, Ta, Re, Ru) in Ni_3Al ($L1_2$) γ'-precipitates of model Ni-Al-Cr superalloys are investigated by first-principles calculations and atom-probe tomography (APT). Measurements of the γ'-phase composition by APT suggest that the investigated transition metals prefer to occupy the Al sublattice-sites in the γ'-precipitates. The calculated substitutional energies of the solute atoms at the Ni and Al sublattice sites indicate that Ta, Re, and Ru have a strong preference for Al site, while Cr has a weak Al site preference. The significant decrease of the substitutional energies of Cr, Re and Ru from the γ'-phase to the γ-phase provide the driving force for the partitioning of these elements to the γ-matrix. In contrast, the substitutional energy of Ta increases from the γ'-phase to the γ-phase, leading to strong partitioning of Ta to the γ'-phase.

5:45 PM
Coarsening in Al-Cu Solid-Liquid Mixtures: *Julie Fife*[1]; Larry Aagesen[1]; Erik Lauridsen[2]; Marco Stampanoni[3]; Peter Voorhees[1]; [1]Northwestern University; [2]Risoe National Laboratory; [3]Paul Scherrer Institut

We examine, in-situ, the morphological evolution of solid-liquid mixtures in the Al-Cu system during isothermal coarsening, with increasing solid volume fraction and varying solidification techniques. Through x-ray tomography, real-time data is collected for the three-dimensional analysis of these complex structures. Phase-field calculations, using this data as initial conditions, are also employed. The morphology and topology of the microstructure are analyzed through interface shape distributions and genus, which determine the probability of finding a patch of interface with a given set of principal curvatures and the topological complexity of the microstructure, respectively. We find that the microstructure evolves in a manner that is strongly influenced by the initial conditions prior to coarsening and that the phase-field models provide important insight into the experimental results. We also examine the formation of topological singularities, specifically tubes of liquid that fission into liquid droplets. An analysis of this process will be discussed.

6:00 PM
Microstructural Evolution during Thermal Aging of IN718 Plus Alloy: *Vibhor Chaswal*[1]; S Mannava[1]; Vijay Vasudevan[1]; [1]University of Cincinnati

IN718 Plus is the latest high temperature candidate material for aero-engine components having improved peak temperature strength and toughness over IN718 attributed to lower Nb, and Fe and higher Al contents which modify its precipitation behavior. Precipitation hardening is controlled by location and extent of γ', γ" and δ precipitates in this alloy, and is strongly influenced by thermal aging during service. Hence, thermal aging studies between 650°C to 850°C have been conducted on a hot rolled IN718 Plus alloy used commonly in industry. Quantitative measurements of precipitate location, extent and evolution at grain boundaries and within the matrix were evaluated with respect to dislocation density and grain orientation by transmission electron microscopy (TEM), X-ray diffraction (XRD) and electron back-scattered diffraction (EBSD). The predictability of temperature-time dependence of γ', γ" and δ phases, and corresponding micro-hardness results were compared with conventional Larson Miller Parameter(LMP) based approach and multi-scale computational method.

Surface Structures at Multiple Length Scales: Surface Properties in Various Length Scales

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University; Sudipta Seal, University of Central Florida; Yang-Tse Cheng, University of Kentucky; Narendra Dahotre, University of Tennessee; Graham McCartney, University of Nottingham

Tuesday PM Room: 3011
February 17, 2009 Location: Moscone West Convention Center

Session Chair: To Be Announced

2:00 PM Keynote
Thin Film Epitaxy Across The Misfit Scale And Its Implications On Integration Of Solid State Devices: *Jagdish Narayan*[1]; [1]North Carolina State University

A unified domain epitaxy model for thin film growth across the misfit scale is presented, specifically addressing phenomena related to dislocation nucleation and propagation and stress relaxation during thin film growth. Due to difficulty in dislocation nucleation and propagation in oxides with high lattice frictional stress, it is argued that it is more difficult to relax smaller misfit strains, particularly tensile strains. Under a compressive strain, surface step is lower which reduces dislocation nucleation barrier at free surfaces. Thus, oxides films, where critical thickness is less than a couple of monolayers, can be grown epitaxially and more relaxed on substrates having a large lattice misfit using the paradigm of domain matching epitaxy. In the domain matching epitaxy, integral multiples of lattice planes match across the film-substrate interface, and the misfit falling in between the integral multiples can be accommodated by the principle of domain variation.

2:40 PM
Characterization And Analysis Of Island Formation Mechanisms On Yttria-Stabilized Zirconia Crystal Surfaces: *Haris Ansari*[1]; Andrew Boyne[1]; Sheikh Akbar[1]; Suliman Dregia[1]; Yunzhi Wang[1]; [1]The Ohio State University

Recently, we have shown that thin film systems of Gadolinium-doped Ceria (GDC) on single crystal Yttria Stabilized Zirconia (YSZ) can spontaneously develop pseudo-periodic arrays of nano-islands. These islands were observed near delaminated regions (sharp interfaces of GDC film and bare substrate), following heat treatment. They showed strong tendencies to self-organize, aligning along elastically soft directions. Presently, we have reproduced the phenomenon via lithographically prepared arrays of GDC patches on bare YSZ. Regular fringes of highly aligned nano-islands form around these deposited features. Micro-raman spectroscopy is employed to examine the nature and role of stress in the islands. Electron microscopy is used to characterize island composition and formation kinetics. Finally, a phase field computational model is used to examine the fundamental basis of the island formation. Direct comparison of modeling and experiment is facilitated by the controlled nature and regular geometry of the lithographic experiments.

3:00 PM
Heteroepitaxial Diffusion of Cu Islands on Ag(111) by the Dislocation Glide Mechanism: *Henry Wu*[1]; Dallas Trinkle[1]; [1]University of Illinois, Urbana-Champaign

Island diffusion dynamics play an important role in determining the morphology and structure of heteroepitaxial thin film growth. In systems with large lattice-mismatch such as Cu islands on Ag(111), we expect dislocation glide as a migration mechanism. To study the size and shape dependence of Cu island diffusion, we optimize an embedded atom method potential. We validate the potential by comparing Cu monomer, dimer, and trimer results with DFT and experiment. A systematic study of diffusion pathways for islands up to 20-atoms shows faster diffusion for islands with the dislocation mechanism than without. This gives a "magic size" effect: non-monotonic behavior of diffusion with island size. The dislocation mechanism is sensitive to surface strain, where neighboring islands break the directional symmetry in diffusion barriers by 10-20meV. A kinetic Monte Carlo model for the dislocation-glide mechanism including island interactions via strain is compared with experimental observations of fast island diffusion.

3:20 PM Break

3:40 PM Invited
From Dynamic Surface Roughening To Dynamic Smoothening Of Nanocomposite Films: *Jeff DeHosson*[1]; Yutao Pei[1]; [1]University of Groningen

This paper reports some striking findings on the breakdown of dynamic roughening in film growth. With increasing energy flux of concurrent ion impingement during pulsed DC sputtering, a transition from dynamic roughening to dynamic smoothening is observed in the growth behavior of MeC/a-C DLC nanocomposite films. In the case of dynamic smoothening, TiC/a C nanocomposite films exhibit a negative growth exponent and ultra-smoothness (RMS roughness ~0.2 nm at film thickness of 1.5 μm). As an experimental indication of the impact-induced downhill flow model an amorphous front layer of 2 nm thickness has been observed with high resolution cross-sectional transmission electron microscopy, always covering the bulk nanocomposite film and consequently leading to ultrasmoothness. Roughness evolution has been described by the linear stochastic equation which contains the second- and fourth-order gradient terms. The predicted interface evolution is in a good agreement with the atomic force microscopy measurements of roughness evolution.

4:10 PM
New Characteristic Length Scale on Surfaces: *Hanchen Huang*[1]; [1]RPI

Surfaces have various well-established characteristic length scales, such as Mullins' wavelengths due to mass transport and atomic islands dimension in epitaxy. Following the discovery of three-dimensional Ehrlich-Schwoebel barrier, we have discovered a new length scale during surface processing such as synthesis. This presentation starts with the physics origin of such length scale, and continues with atomistic simulations demonstrating the variation of the length scale and validation experiments, and ends with design of nanosynthesis based on the knowledge of this new length scale & its experimental validation. It is interesting to note that this length scale is the very reason that nanorods synthesis is possible, even though nanorods had been realized long time ago (and it was patented a decade ago).

4:30 PM
Understanding Strain Localization Through Matrix-Based 3-D Surface Roughness Characterizations: *Mark Stoudt*[1]; Joseph Hubbard[1]; [1]National Institute of Standards and Technology

Since stretch-forming limiting strains are typically determined with complex, deterministic numerical simulations that do not reliably predict true localization strain, a matrix-based, 3-dimensional surface analysis technique has been developed to improve characterizations of the morphological conditions that promote strain localization. This technique quantifies and then maps the changes that are contained in the surface roughness data produced by plastic deformation. The results of analyses on commercial aluminum sheet surfaces subjected to three in-plane stretching modes established that strain localization is dominated by stochastic processes that can be reliably predicted with Weibull statistics. This study also suggests that gross localization may involve a nucleation and growth process that requires regions of the surface to exceed a threshold roughness magnitude before localization occurs. The methodology developed for this approach and the potential impact on models used to predict limiting strains shall be presented and discussed.

4:50 PM

Stochastic Finite Temperature Continuum Modeling With Applications to Film Evolution: *Lawrence Friedman*[1]; [1]Pennsylvania State University

Evolution of surface structure is frequently modeled as an energy dissipating process triggered and augmented by thermal fluctuations. Until recently, a prescription for including general thermal fluctuations in phenomenological models has been lacking and in most instances, random fluctuations are neglected or random initial conditions are used as a surrogate for actual thermal fluctuations. However, Lau and Lubensky (Phys. Rev. E 76, 011123, 2007) showed how to construct finite temperature models using stochastic differential equations with general white noise terms. Their method ensures that an ensemble of systems approaches the Boltzmann distribution. Here, their method is extended to discretized stochastic partial differential equations and then applied to fluctuations in strained and unstrained film surfaces.

5:10 PM

Hydrogen Transport In Fe/Ti Nanometer-Scale Multilayers During In-Situ Thermal Annealing: *Z.L. Wu*[1]; B.S. Cao[1]; J. Gao[1]; T.X. Peng[1]; M.K. Lei[1]; [1]Surface Engineering Laboratory, School of Materials Science and Engineering, Dalian University of Technology

Hydrogen transport in the Fe/Ti nanometer-scale multilayers on Si(100) substrates during in-situ thermal annealing at 463 K was investigated by using x-ray diffraction (XRD), secondary ion mass spectrometry (SIMS), and cross-sectional transmission electron microcopy (XTEM). The Fe/Ti nanometer-scale multilayers constructed with thickness of alternating Fe and Ti sublayers of 16.2 nm and the sublayer thickness ratio of 1:1 were deposited by direct current magnetron sputtering. The composition modulation structure was still maintained in Fe/Ti nanometer-scale multilayers during thermal annealing. After annealing for 10 min, hydrogen permeated through the whole Fe/Ti nanometer-scale multilayers and localized in Ti sublayers to form TiH. With the annealing time increased to 30 min, the concentration of hydrogen increased in the Ti sublayer and TiH transformed to TiH2. It is found that composition modulation structure has a significant effect on the transport of hydrogen during thermal annealing.

5:30 PM

Synthesis and Characterization of Boron Carbide Thin Films Grown by RF Sputtering: *Tolga Tavsanoglu*[1]; Sid Labdi[2]; Michel Jeandin[3]; [1]Istanbul Technical University [2]Université d'Evry Val d'Essonne, Laboratoire d'études des Milieux Nanométriques; [3]MINES ParisTech, Centre des Matériaux

Boron carbide (B_4C) is the third hardest material at room temperature and it combines many other attractive properties such as, high modulus, good wear resistance, and high chemical and thermal stability. Boron carbide films are considered to be promising candidate as hard, protective coatings for cutting tools and other wear resistance applications. In the present study, boron carbide thin films of 500–700 nm were deposited by RF sputtering from a boron carbide target. The elemental composition of the deposited films was measured by EPMA. The mechanical properties, Young's modulus and hardness were determined by AFM coupled nanoindentation technique. FTIR analyses were conducted to evaluate bonding characteristics of boron carbide. The microstructure and crystallinity of the films were characterized by cross–sectional SEM and XRD analysis. Tribological properties were also investigated by pin–on–disc test measurements.

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Nanotribological Properties of Carbon Nanotube Reinforced Plasma Sprayed Aluminum-Silicon Alloy Composite Coatings: *Srinivasa Bakshi*[1]; Kantesh Balani[1]; Arvind Agarwal[1]; [1]Florida International University

Nanoscratch experiments have been carried out on plasma sprayed Al-Si coatings containing 5wt% and 10wt% carbon nanotubes (CNT) as well as Al-Si coating without nanotubes. The effect of CNTs on the wear resistance and friction properties are studied. Scratching has been done both under constant load and increasing load conditions. SEM and AFM have been used to image the wear track under both loading conditions. Microstructure of the coatings has been discussed to delineate the wear resistance mechanisms in CNT reinforced composites.

Synergies of Computational and Experimental Materials Science: Synergies in Nanoscience

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

Program Organizers: Katsuyo Thornton, University of Michigan; Henning Poulsen, Risoe National Laboratory; Mei Li, Ford Motor Company

Tuesday PM — Room: 3003
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: Yunzhi Wang, Ohio State University; Ragnvald Mathiesen, NTNU

2:00 PM Invited

Dislocation Mechanism In Nanocrystalline Metals: Atomistic Simulations And Experiments: *Helena Van Swygenhoven*[1]; Christian Brandl[1]; Steven Van Petegem[1]; Peter Derlet[1]; [1]Paul Scherrer Institut

Molecular dynamics deformation studies of nanocrystalline metals suggest dislocations are nucleated at grain boundaries, travel through the grain to be finally absorbed in the surrounding grain boundaries. Dislocation propagation on the slip plane on which it was nucleated was observed to be hindered by stress intensities in the grain boundaries suggesting that propagation could not be excluded as a rate limiting process. Recent constant strain rate molecular dynamics simulations of nanocrystalline Al demonstrate that dislocations also exhibit cross-slip via the Fleischer mechanism. The grain boundary is found to strongly influence when and where cross-slip occurs, allowing the dislocation to avoid local stress concentrations that otherwise can act as pinning sites for dislocation propagation (PRL 100(2008)235501). In this talk the latest suggestions from molecular dynamics are discussed in terms of new experiments involving strain rate sensitivity measurements, stress dip test, creep studies and in-situ tensile and compressive testing during X-ray diffraction.

2:40 PM Invited

Effects Of Solute Concentrations On Kinetic Pathways In Ni-Al-Cr Alloys: Experiments And Simulations: *David Seidman*[1]; Christopher Booth-Morrison[1]; Zugang Mao[1]; Chantal Sudbrack[1]; [1]Northwestern University

The kinetic pathways resulting from the formation of coherent gamma-prime precipitates from a gamma (fcc) matrix are studied for two alloys with similar gamma-prime (L12)precipitate volume fractions at 873 K. The phase decompositions of Ni-7.5 Al-8.5 Cr at.%, and Ni-5.2 Al-14.2 Cr at.%, for aging times 1/6 to 1024 hours are investigated by atom-probe tomography (APT), and they differ significantly. The morphologies of the gamma-prime precipitates of the alloys are similar, though the gamma-prime precipitate coagulation and coalescence differ. The temporal evolution of the gamma-prime precipitate average radii and the gamma-matrix supersaturations follow the predictions of classical coarsening models. The compositional trajectories of the gamma matrix phases of the alloys are found to follow the equilibrium tie-lines, while the trajectories of the gamma-prime precipitates do not, resulting in significant differences in the partitioning ratios of the solute elements. The experimental APT results are compared with lattice kinetic Monte Carlo simulations.

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Computer Simulations of Precipitate Strengthening of Al-Zr-Sc, as Informed by Local-Electrode Atom-Probe Tomography: *Richard Karnesky*[1]; Keith Knipling[2]; Volker Mohles[3]; David Dunand[4]; David Seidman[4]; [1]Northwestern University and Sandia National Laboratories; [2]Northwestern University and Naval Research Laboratory; [3]RWTH Aachen University; [4]Northwestern University

Local-electrode atom-probe tomography allows structural information about hundreds of nanoscale precipitates, including precipitate size, volume fraction, number density, and edge-to-edge interprecipitate distances, to be measured directly in three dimensions. These data can be used to generate glide planes for use in a continuum dislocation dynamics simulation that calculates the critical resolved shear stress based on the elastic interactions of dislocations with each other, themselves, and with precipitates. Within a single glide plane, some precipitate cross sections may be sheared (creating an antiphase boundary) while others are bypassed via Orowan dislocation looping. When isochronally-aged to 300 °C, Al-0.1 Zr-0.1 Sc (at. %) forms a high number density of nanoscale, coherent $L1_2$) $Al3(Sc_{1-x},Zr_x)$ precipitates. As the aging temperature is increased to 400 °C, a Zr-rich precipitate shell grows, further strengthening the alloy. We compare the ambient-temperature microhardnesses as a function of aging treatment with the strength that is modeled.

3:40 PM Break

4:00 PM Invited
Combined Phase-field Simulations and Experimental Studies of Nanoferroics: *Long Qing Chen*[1]; Darrell G. Schlom[2]; Venkat Gopalan[1]; X. X. Xi[1]; C. B. Eom[3]; S. V. Kalinin[4]; R. Ramesh[5]; X. Q. Pan[6]; [1]Pennsylvania State University; [2]Cornell University; [3]University of Wisconsin; [4]Oak Ridge National Laboratory; [5]University of California, Berkeley; [6]University of Michigan

This presentation will discuss a number of successful examples of coupling phase-field simulations and experimental measurements in the area of nanoferroics with an emphasis on the phase transitions, domain structures, and properties of ferroelectric thin films. It will be shown that one can use phase-field simulations to not only help interpreting experimental observations but also provide guidance to achieve desirable transition temperatures, specific domain states, domain wall orientations, and domain wall mobility. Furthermore, phase-field modeling has also been combined with piezoelectric force microscopy to study the local ferroelectric domain switching. Examples to be discussed include several important oxide systems $BaTiO_3$, $PbZr_xTi_{1-x}O_3$, $BiFeO_3$ and $BaTiO_3$/$SrTiO_3$ superlattices.

4:40 PM
Crystal Plasticity Modeling and Micro-Mechanical Experiments of Gamma-TiAl Based Microstructures: *Claudio Zambaldi*[1]; Franz Roters[1]; Stefan Zaefferer[1]; Dierk Raabe[1]; [1]Max-Planck-Institut für Eisenforschung

The deformation behavior of a gamma-TiAl based alloy is incorporated into a crystal-plasticity formulation coupled with a finite-element solver. The single-phase constitutive behavior is calibrated by nano-indentation experiments in single phase regions. For the discrimination of the orientational variants a newly developed high-precision indexing method for electron backscatter diffraction patterns was applied. Nano-indentation experiments are evaluated by a 3D model. The simultaneous activation of deformation mechanisms is used to assess their relative strengths and cross-hardening. The lamellar microstructure is analyzed in terms of kinematic constraints, which lead to pronounced plastic anisotropy. Secondly, the mechanical behavior of massively transformed microstructures is modeled by applying a lower degree of kinematic constraints. On a grain-scale, this results in less plastic anisotropy and possibly improved ductility. An attempt is described to include the significant micro stresses.The modeling is complemented and validated by mechanical characterization through small-scale tests.

5:00 PM
Modeling the Drawing of Steel Wire with Nano-Engineered Composite Hardmetal Dies: *Ivica Smid*[1]; Daniel Cunningham[1]; Erik Byrne[1]; John Keane[2]; [1]Pennsylvania State University [2]Allomet Corporation

Wire drawing with a novel composite hardmetal comprised of hard Al2O3 core particles encapsulated in a tough WC-Co shell has been studied. Dynamic fracture toughness testing shows that this is an excellent material for machining in the high strain rate environments of metal forming operations such as wire drawing. This material's ability to resist dynamic fracture is due to the toughening mechanisms of its microstructure, such as added binder ductility, crack interactions at core particles, and crack interactions at grain boundaries. Finite element modeling was used to model the microstructural interactions in wire drawing. Using non-linear material properties for steel, permanent strain of the wire due to plastic deformation was found as well as the stress states imposed by them. Future advances of these simulations will include predictions of wear lifetime as well as influence of temperature.

5:20 PM
First-Principles Simulations and Inelastic Neutron Scattering in Thermodynamics Studies: *Oliver Delaire*[1]; Matthew Lucas[2]; Max Kresch[1]; Jiao Lin[1]; Brent Fultz[1]; [1]California Institute of Technology; [2]Oak Ridge National Laboratory

In crystalline materials, low energy excitations around the average configuration of the ions and electrons can be thermally activated, providing entropy. As such, phonons, spin-waves, or electronic excitations can be subdivided into their own entropic contributions. Inelastic neutron scattering is the preferred experimental technique to measure phonons and magnons, but a difficulty in analyzing the neutron scattering results resides in the complexity of the experimental datasets. First-principles simulations of the phonon dynamics have now reached a level of reliability such that the full phonon scattering function can be predicted from quantum mechanics, and compared to experimental data. The synergy between quantum mechanical calculations and experimental measurements has provided us with valuable insights. Fundamental relations between phonon excitations and the underlying electronic band structure are discussed, and in particular, we show that the electron-phonon interaction can influence the thermodynamics of metals to much higher temperatures than was previously assumed.

Transformations under Extreme Conditions: A New Frontier in Materials: Extreme Deformation and Damage

Sponsored by: The Minerals, Metals and Materials Society, ASM International, ASM Materials Science Critical Technology Sector, TMS Materials Processing and Manufacturing Division, TMS/ASM: Phase Transformations Committee
Program Organizers: Vijay Vasudevan, University of Cincinnati; Mukul Kumar, Lawrence Livermore National Laboratory; Marc Meyers, University of California-San Diego; George "Rusty" Gray, Los Alamos National Laboratory; Dan Thoma, Los Alamos National Laboratory

Tuesday PM — Room: 3001
February 17, 2009 — Location: Moscone West Convention Center

Session Chairs: George Gray, Los Alamos National Laboratory; James Stolken, Lawrence Livermore National Laboratory

2:00 PM Invited
A Statistical View of High-Rate Material Instability and Failure: *Thomas Wright*[1]; K.T. Ramesh[2]; [1]US Army Research Laboratory; [2]Johns Hopkins University

All materials have defects distributed throughout their interior and surfaces due to processing, prior deformation, microstructure, etc. As a consequence any applied loading on the material will result in stress concentrations also being distributed throughout the interior and surfaces. In quasi-static loading it is well understood that the weakest locations in the material are at risk for initiating failures through material instabilities. In dynamic loading, however, especially extreme dynamic cases, the situation tends to be far more complex, and it may be necessary to give independent consideration to the processes of damage nucleation, growth, and communication with other sites, each process developing according to its own time scale. Clearly the interaction of all these processes and time scales must be modulated by the statistics of defects within the material. Concrete examples will be given to illustrate these interconnected events.

2:35 PM
Characterization of Incipient Spall Damage in Monocrystalline Copper Targets Subjected to Laser-Driven Flyer Impacts: Stephan DiGiacomo[1]; Sheng-Nian Luo[2]; Darrin Byler[2]; Rob Dickerson[2]; *Pedro Peralta*[1]; Scott Greenfield[2]; Aaron Koskelo[2]; Kenneth McClellan[2]; [1]Arizona State University; [2]Los Alamos National Laboratory

Monocrystalline copper was subjected to low pressure shocks (4 - 6 GPa)along the <100>, <110>, <111>, <123>, and <114> directions using laser-driven flyers. Values for spall strength as determined from pullbacks in the free-surface velocity histories are reported for each orientation. Characterization of untested <100> samples using electron backscatter diffraction (EBSD) revealed very-low-angle (~0.5-2 deg.) sub-grain boundaries that localized damage in shocked specimens, since numerous isolated voids clustered along boundaries oriented parallel to the shock direction. Furthermore, EBSD revealed high misorientation bands parallel to {111} traces. Spall damage was noted to occur at the intersections of these bands and the low-angle sub-grain boundaries. High-resolution EBSD analysis of isolated voids in shocked <100> samples revealed octahedral geometry of individual voids with a characteristic misorientation field. The lattice rotations surrounding the spall voids were analyzed with a kinematic crystal plasticity model to derive the effective plastic strain around the voids.

2:55 PM Invited
Laser-Shock Induced Deformation and Spalling in Metals: *Marc Meyers*[1]; Hussam Jarmakani[1]; Bimal Kad[1]; Bruce Remington[2]; Daniel Kalantar[2]; Brian Maddox[2]; Eduardo Bringa[2]; James McNaney[2]; [1]University of California, San Diego; [2]Lawrence Livermore National Laboratory

High-amplitude lasers producing shock and quasi-isentropic compression are a powerful tool to probe the generation and evolution of damage in metals under extreme pressure and strain rate conditions. Pressures higher than 100 GPa, strain rates in the range 10^6-10^9 s^-1, and durations on the order of 10^-9 seconds are achieved in a controlled and reproducible manner. Monocrystalline and polycrystalline copper, nickel, and vanadium were subjected to laser compression at the Lawrence Livermore National Laboratory Jupiter facility and at the University of Rochester Omega facility (LLE). The generation of dislocations, mechanical twins, stacking faults, and voids was characterized, quantified, and modeled. The transition from slip to twinning is analysed through the constitutive behavior of the two mechanisms, the Rankine-Hugoniot relation, and the Swegle-Grady equation. Analytical predictions are compared with molecular dynamics results. In spalling experiments, experimentally obtained fragment sizes are compared with predictions from the Grady-Kipp model. Support: UCOP ILSA Program.

3:30 PM
Spall (Dynamic Fracture) Strength and Deformation Microstructure of SS304 Alloy at High Strain Rate: Keshaw Joshi[1]; R. Tewari[1]; G. Dey[1]; Satish Gupta[1]; *Srikumar Banerjee*[1]; [1]Bhabha Atomic Research Centre

Spall (dynamic fracture) strength and the deformation microstructure of SS304 alloy have been determined at high strain rate. Spall in SS304 plate has been achieved by impacting it with a parallel SS304 plate at velocity of 0.6 km/s; this impact introduced a shock wave of 11.9 GPa in both the target and the impactor. The interaction of tensile waves resulting from the reflection of shock wave from the target free surface and from the flyer free surface generated large tensile stress in the target which exceeded the spall strength causing spall fracture. The analysis of the velocity history of the target free surface recorded using VISAR reveals the dynamic yield strength of 0.8 GPa and spall strength of 2.6 GPa at strain rate of ~ 10^4 /s. The microstructure examination of the area in proximity of the fractured surface using SEM and TEM revealed new deformation features.

3:50 PM
Three-Dimensional Characterization of Spall Damage in Shock Loaded Metallic Multicrystals: Leda Wayne[1]; Shima Hashemian[1]; Stephan DiGiacomo[1]; *Pedro Peralta*[1]; Heber D'Armas[2]; Shengnian Luo[3]; Scott Greenfield[3]; Dennis Paisley[3]; Robert Dickerson[3]; Darrin Byler[3]; Ken McClellan[3]; [1]Arizona State University [2]Universidad Simon Bolivar; [3]Los Alamos National Laboratory

Correlations between damage and local microstructure were investigated in multicrystalline copper, nickel and titanium samples via impact tests conducted with laser-driven plates. All samples had a large grain size compared to the thickness, to isolate microstructure effects on local response. Velocity interferometry was used to monitor the response of the samples and spall failure. Cross-sectional Electron Backscattering Diffraction (EBSD) was used to relate crystallography to damage at features such as grain boundaries (GBs) and triple points. Preferred damage nucleation and localization sites were identified via statistical sampling in serial sectioned specimens and through 3-D reconstructions obtained from serial cross-sections. Damage distribution and connectivity along the spall plane in 3-D were correlated to GB misorientations, GB inclination to the shock and grain connectivity at particular locations. Results indicate that the tips of terminated twins and locations with high grain connectivity are the preferred locations for intergranular damage in these samples.

4:10 PM Break

4:25 PM Invited
Emergence of Mesoscopic Length Scales through Self-Organization in Alloys Subjected to Severe Plastic Deformation: *Pascal Bellon*[1]; Robert Averback[1]; Pavel Krasnochtchekov[1]; Samson Odunuga[1]; Alfredo Caro[2]; Jung Singh[1]; Wenjun Cai[1]; [1]University of Illinois; [2]Lawrence Livermore National Laboratory

Materials are often subjected to sustained and severe plastic deformation, for instance during extrusion, high-energy ball milling, or when experiencing frictional wear. We recently showed that the chemical mixing forced by dislocation-based plasticity in solids can be superdiffusive in some length scale range. In alloy systems comprised of immiscible elements, the dynamical competition between this superdiffusive forced mixing and thermally activated decomposition can lead to self-organization of the composition, producing mesoscopic composites. We will establish via atomistic simulations, modeling and experiments the parameters that determine the characteristic length scale of these compositional patterns. Self-organization may also impart new and beneficial properties. We will illustrate this point by discussing the important role played by mechanically mixed layers in improving resistance to sliding wear.

5:00 PM
Carbide Decomposition Induced by Severe Plastic Deformation: *Xavier Sauvage*[1]; Yulia Ivanisenko[2]; [1]University of Rouen; [2]Institute of Nanotechnology

The strain induced carbide (cementite) decomposition in pearlitic steels is widely reported in the literature. It is indeed though to be responsible of the formation of the extremely hard white etching layer on rail track surfaces and also to affect the ductility of heavily drawn steel cords. However, the driving force and the kinetic of this phase transformation are still under debate and there are still some doubts about the distribution of carbon atoms resulting from the carbide decomposition. In this report, recent Atom Probe Tomography showing both the strain induced transformation of the cementite and carbon atoms diffusion would be presented. These data demonstrate that the first step of the decomposition is the formation of a thin layer of under-stoechiometric cementite along the cementite/ferrite interface. The role of dislocations on the diffusion and the distribution of carbon atoms in the ferrite would be discussed also.

5:20 PM
Nanocluster Formation in Mechanically-Alloyed Ferritic ODS Steels: *Michael Miller*[1]; Chong Long Fu[1]; David Hoelzer[1]; Kaye Russell[1]; Chain Liu[1]; [1]Oak Ridge National Laboratory

Atom probe tomography of ball-milled powders of a 14YWT ferritic alloy has revealed that the solute atoms from the yttria particles are forced into solid solution during mechanical alloying and there is an excess of vacancies. First principle calculations have revealed a delicate balance between vacancies and the levels of Ti and Y for the formation of nanoclusters and too high levels will result in the formation of $Y_2Ti_2O_7$ or TiO_2. Although micron size oxides are observed, the predominant microstructural feature is a high density of Ti-, O- and Y-enriched nanoclusters that form during extrusion. The nanoclusters are extremely resistant to coarsening at temperatures up to 1400ºC. 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.

5:40 PM
Atomic Scale Investigation of Strain Induced Interdiffusion in the Cu-Fe System: *Xavier Sauvage*[1]; Xavier quelennec[1]; Florian Wetscher[2]; Jean Marie Le Breton[1]; Alain Menand[1]; [1]University of Rouen; [2]Erich Schmid Institute

Strain induced interdiffusion and supersaturated solid solutions are widely reported in the literature, especially for ball milled powders. However, little is known about the physical mechanisms leading to such non-equilibrium structures. Here, we report about specific experiments performed on the Cu-Fe system in the bulk state with an accurate control of the temperature and of the strain rate. A nanostructured Cu-Fe composite was processed by high pressure torsion up to extreme level of deformation. Atom Probe Tomography and Mössbauer spectroscopy reveal the progressive interdiffusion of Cu and Fe and finally the formation of a homogeneous solid solution. The contribution of strain induced vacancies would be discussed.