TECHNICAL PROGRAM GRID

Room	Monday-November 5 AM	Monday-November 5 PM	Tuesday-November 6 AM	Tuesday-November 6 PM	Wednesday-November 7 AM	Wednesday-November 7 PM	Thursday-November 8 AM
102	ASM – TSS – HTS – IMS – TMS Room: 500 Ballroom Keynote Session: Materials and Processes for the New Millennium				GA: Chemistry & Physics of Materials-I	GA: Chemistry & Physics of Materials-II	
105		GA: High Temperature Alloys-I	GA: High Temperature Alloys-II				
108						GA: Surface Engineering	
111		Appl of Computational Thermodynamics in Materials Processing-I	Appl of Computational Thermodynamics in Materials Processing-II	Appl of Computational Thermodynamics in Materials Processing-III	Radiation and Thermal Degradation of Polymer Materials	GA: Polymers	
112						Surface Composite Coatings-I	Surface Composite Coatings-II
113						GA: Alloy Phases	
115		Friction Stir Welding and Processing-I	Friction Stir Welding and Processing-II	Friction Stir Welding and Processing-III	Friction Stir Welding and Processing-IV	GA: Mechanical Metallurgy and Composite Materials	
117		GA: Industrial Innovations					
139		Affordable Metal-Matrix Composites for High Performance-I	Affordable Metal-Matrix Composites for High Performance-II	Affordable Metal-Matrix Composites for High Performance-III	Affordable Metal-Matrix Composites for High Performance-IV	Affordable Metal-Matrix Composites for High Performance-V	Affordable Metal-Matrix Composites for High Performance-VI
140		Modeling the Performance of Engr Structural Matls-I	Modeling the Performance of Engr Structural Matls-II	Modeling the Performance of Engr Structural Matls-III	Modeling the Performance of Engr Structural Matls-IV	Modeling the Performance of Engr Structural Matls-V	
201			Mech Behavior & Constutive Modeling of Polymers, Fiber & Particulate Reinforced-I	Process/Property Relationships in Polymer Matrix Comp & Nanocomp-I	Process/Property Relationships in Polymer Matrix Comp & Nanocomps-II	Mech Behavior & Constutive Modeling of Polymers, Fiber & Particulate Reinforced-II	
202			Polymers & Other Organics for Electronic Appl: Micro-to-Nano Technology	Lead-Free, Lead-Bearing Solders & Alternative Surface Finishes for Electr Packaging-I	Lead-Free, Lead-Bearing Solders & Alternative Surface Finishes for Electr Packaging-II	Lead-Free, Lead-Bearing Solders & Alternative Surface Finishes for Electr Packaging-III	
203						GA: Ferrous Metallurgy	
204		Materials Design Approaches and Experiences-I	Materials Design Approaches and Experiences-II	Materials Design Approaches and Experiences-III	Materials Design Approaches and Experiences-IV	Materials Design Approaches and Experiences-V	Materials Design Approaches and Experiences-VI
206					GA: Non-Ferrous and Powder Metallurgy	Full Density Powder Processing	
208		Microstr Modeling & Prediction during Thermomechanical Processing-I	Microstr Modeling & Prediction during Thermomechanical Processing-II	Microstr Modeling & Prediction during Thermomechanical Processing-III	Microstr Modeling & Prediction during Thermomechanical Processing-IV	Microstr Modeling & Prediction during Thermomechanical Processing-V	
209		Powder Materials: Current Research & Industrial Practices-I	Powder Materials: Current Research & Industrial Practices-II	Powder Materials: Current Research & Industrial Practices-III	Powder Materials: Current Research & Industrial Practices-IV	Powder Materials: Current Research & Industrial Practices-V	
212		GA: Refractory Metals					
500 Ballroom		Polymer Plenary Polymer Poster Session					
White River BR			Fundamentals of Solidification-I	Fundamentals of Solidification-II	Fundamentals of Solidification-III	Fundamentals of Solidification-IV	
Wabash Ballroom 1						2:00–5:00 PM Joint ASM/TMS Governmental Affairs	

Technical Program

2001 TMS FALL MEETING TABLE OF CONTENTS

ASM – TSS – HTS – IMS – TMS

Monday AM Room: 500 Ballroom
November 5, 2001

Keynote Session:
Materials and Processes for the New Millennium

Affordable Metal-Matrix Composites for High Performance Applications - I: Aerospace, Space and Land Vehicle Applications

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: Awadh B. Pandey, Pratt & Whitney, Liquid Space Propulsion, West Palm Beach, FL 33410-9600 USA; Kevin L. Kendig, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Watson, Pratt & Whitney, East Hartford, CT 06108 USA

Monday PM Room: 139
November 5, 2001 Location: Indiana Convention Center

Session Chair: Awadh B. Pandey, Pratt & Whitney, Liquid Space Propulsion, W. Palm Beach, FL 33410-9600 USA

2:00 PM Keynote
Metal Matrix Composites for Space Systems: Current Uses and Future Opportunities: *Daniel B. Miracle*[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA

Metal matrix composites (MMC's) have fulfilled important space systems requirements for over 20 years. Current applications include structural members in the space shuttle cargo bay, a multi-functional antenna mast/signal waveguide in the Hubble Space Telescope, and electronic packaging and thermal management for the Iridium constellation of satellites. The growing maturation of MMC technology, along with the expansion of new space systems concepts, now provides an unusual range of opportunities for the use of MMC's in space. Enabling capabilities in liquid rocket propulsion are now being pursued. Both particulate- and fiber-reinforced MMC's are being developed and evaluated for pump motor housings of liquid fuel rocket engines. Other pump components which are being pursued as a result of very attractive trade studies include inducers and impellers, cryogen ducts, lines and flanges, and the combustion chamber structural jacket. A novel approach to the manufacture of continuously-reinforced Al MMC's is being developed to produce cryogen tankage. The suite of material characteristics that make MMC's attractive for on-orbit applications include very good specific stiffness and strength, high thermal and electrical conductivity, low-to-moderate thermal expansion, excellent resistance to UV radiation and good resistance to atomic oxygen. This balance of properties, and the fact that many of these properties are fully tailorable, offer unique opportunities for multi-functional use. Finally, the cost of MMC's, especially particulate-reinforced metals, can offer signifcant improvements in affordability over competing monolithic metals and organic composites. The current materials systems offering the highest payoffs will be presented, and the impact on space systems will be described. Technical challenges to be overcome will be listed and programmatic opportunities will be suggested.

2:30 PM
Evaluation of Al/SiC Composites for Application in the AAAV: *David M. Bowden*[1]; Hsin Nan Chou[1]; Stephen Evans[2]; [1]The Boeing Company, Phantom Works, PO Box 516, MC S276-1017, St. Louis, MO 63166 USA; [2]Eck Industries, Inc., PO Box 967, Manitowoc, WI 54221-0967 USA

We have investigated the feasibility of utilizing Al/SiC composites for the road wheel wear ring of the Advanced Amphibious Assault Vehicle (AAAV). This application requires good wear resistance, as well as the ability to withstand track loads in service. Objectives are to provide a low cost, lightweight component that meets vehicle requirements. In this paper, we will review property goals, and present the results of screening tests on a variety of candidate materials. We will also review our approach to component fabrication, and the results of full-scale laboratory tests of prototypes.

2:50 PM
Development of a New Discontinuously-Reinforced Aluminum for Space Applications: *Awadh B. Pandey*[1]; Kevin L. Kendig[2]; Daniel B. Miracle[2]; [1]Pratt & Whitney, Liquid Space Propulsion, PO Box 109600, MS 702-06, W. Palm Beach, FL 33410-9600 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, Dayton, OH 45433 USA

Discontinuously-reinforced aluminum (DRA) has been used in aerospace structures such as Ventral Fins and Fan Exit Guide Vanes owing to their superior specific stiffness, specific strength, wear resistance, and thermal resistance as compared to the unreinforced aluminum alloys. In order to reduce the weight of the engine, DRA materials are now being considered for space applications. The higher specific strength at ambient and elevated temperature is one of the main requirements in certain rocket applications. The conventional DRA materials use commercial precipitation hardened aluminum alloys as matrices which have limited strengths particularly at elevated temperatures due to coarsening of precipitates. Therefore, an aluminum alloy which can provide significantly higher ambient and elevated temperature strengths is required. In this paper, a new aluminum alloy based on Al-Mg-Sc with improved ambient and elevated temperature strengthening capability is proposed. The monolithic alloy and the composite with 15 volume percent SiC particles were processed using a powder metallurgy approach. The influence of processing parameters on the microstructures and mechanical properties of the monolithic and composite materials is discussed. The thermal stability of the monolithic and composite materials is also evaluated. The strength of the composite is discussed in terms of solid solution strengthening, Orowan strengthening, and load transfer models.

3:10 PM Invited
Yes, This is Rocket Science: MMCs for Liquid Rocket Engines: *J. S. Shelley*[1]; Richard LeClaire[1]; Daniel B. Miracle[2]; James Nichols[1]; [1]US Air Force, AFRL/PRSE, 4 Draco Dr., Edwards AFB, CA 93524-7160 USA; [2]US Air Force, AFRL/MLLM, 3000 P St., Bldg. 655, Wright Patterson AFB, OH 45433 USA

The Air Force's Integrated High-Payoff Rocket Propulsion Technologies Program (IHPRPT) has established aggressive goals for both improved performance and reduced cost of rocket engines and components. Achieving these goals relies on developing affordable Metal Matrix Composite (MMC) technologies for application to Liquid Rocket Engines (LREs). Efforts are being conducted on three types of MMCs: Aluminum, Copper, and Nickel matrix material systems. Potential applications include turbopump housings, rotating machinery, and high stiffness flanges and ductwork. This presentation will review the IHPRPT goals as well as the current material requirements for MMC technologies being developed for application to LREs.

3:40 PM Break

3:55 PM Invited
High-Strength Al-Based Nanophase Alloys Developed by Use of Quenching-Induced Metastable Phase: *Akihisa Inoue*[1]; [1]Tohoku University, Inst. for Matls. Rsrch., Sendai 980-8577 Japan

Recently, we have succeeded in developing three kinds of high-strength Al-based alloys by warm consolidation of atomized non-equilibrium phase powders. The first group is nanocrystalline Al-Ln-Zr-TM (Ln=lanthanide metal, TM=Fe,Co,Ni) alloys with a mixed structure of nanoscale Al_3Ni and $Al_{11}Ln_2$ compound particles embedded in a fcc-Al phase obtained by crystallization of amorphous or amorphous plus Al phases. The particle size is 50 nm for Al_3Ni and 10 nm for $Al_{11}Ln_2$ and the grain size of the Al phase is 100 to 200 nm. The second group includes Al-Mn-Ln-TM, Al-Cr-Ln-TM and Al-V-Ln-TM alloys consisting of nanoscale icosahedral particles surrounded by Al phase without grain boundary. The icosahedral particle size is 30 to 50 nm. The third group is an Al-Zr-Ni-Ln alloy with a mixed structure of Al_3Zr and $Al_{11}Ln_2$ particles embedded in Al phase. The second and third group alloys were formed by the solidification mode of primary icosahedral or Al_3Zr phase followed by the precipitation of Al or Al plus $Al_{11}Ln_2$, respectively. The tensile strength and elongation at room temperature are 650 to 800 MPa and 2 to 7% for the first group alloy, 500 to 800 MPa and 10 to 30% for the second group alloy and 600 to 650 MPa and 10 to 20% for the third group alloy. These alloys also exhibit high-elevated temperature strength of 300 to 350 MPa at 573K. By use of the good mechanical properties, the bulk Al-based alloys have already been used in many fields which require high strength with light weight.

4:25 PM
Development of a High-Temperature Aluminum-Based Material: *K. L. Kendig*[1]; D. B. Miracle[1]; A. B. Pandey[2]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., WPAFB, OH 45433 USA; [2]Pratt & Whitney, Liquid Space Propulsion, W. Palm Beach, FL 33410 USA

Discontinuously reinforced aluminum has potential to maintain high specific strength and stiffness with sufficient ductility to elevated temperatures. Such a material could have pervasive impact in aerospace applications. An Al-Mg-Sc-Zr alloy with or without reinforcements is being developed for use in full-life applications up to 200ºC. Microstructures of the unreinforced and reinforced alloy in different conditions were characterized. The alloys have been tested in tension and resulting fractures have been examined. Thermal stability data for the alloy has also been measured. The relationship of mechanical properties to relevant microstructural features has been explored. Conclusions and recommendations for alloy improvement will be given.

4:45 PM
Diamond Based Composites for Thermal Management: *S. M. Pickard*[1]; J. K. Kim[1]; J. C. Withers[1]; R. O. Loutfy[1]; [1]MER Corporation, 7960 S. Kolb Rd., Tucson, AZ 85706 USA

MER has developed diamond/Al composites for power electronics applications and other high heat flux thermal management applications. The composite is pressure squeeze cast to provide a porosity free material in near net shape. The composite material has high stiffness and strength exceeding Al/SiC. Thermal conductivity of the composite exceeds 550 W/mK and is isotropic, with coefficient of thermal expansion of 7.5 ppm/K for a 45% Vf loading. Processing issues of net shape casting, machinability, use of alternative matrix and cost are discussed. Use of plating with Ni and Au coatings and their impact on thermal properties of a final module are reported.

5:05 PM
New Heat Resistant Materials Based on the Ti-Al-Si-Zr System: *S. Firstov*[1]; V. Mazur[1]; D. Miracle[2]; L. Kulak[1]; S. Kapustnikova[1]; [1]Institute for Problems of Materials Science–Kyiv, State Metlgcl. Acdy., Dnipropetrovs'k, Ukraine; [2]Air Force Research Laboratory, Wright-Patterson AFB, OH 45433 USA

Alloys of the Ti-Si-X system are good for designing heat resistant alloys based on titanium. Creation of composite strengthened with Ti5Si3 silicides is possible due to eutectic crystallization. The data on structure and properties of Ti-Si, Ti-Si-Al and Ti-Si-Al-Zr alloys are given. It is shown that alloying with Al and Zr results in enhancing heat resistance. The temperature dependence of mechanical properties is studied and it is shown that strength of these alloys in as-cast state is higher of strength of titanium -aluminides in temperature range of 20-700°C. It is found that hypoeutectic alloys have higher heat resistance than eutectic ones. Relatively high volume of strengthening phase (up to 26%) is limiting significantly low temperature plasticity of alloys. The possible ways of enhancing plasticity are discussed. Heat treatment allows increasing remarkably both strength and plasticity. It is developed new class of engineering materials based on the Ti-Al-Si-Zr system where the alloyed Ti matrix is reinforced with silicide phases. The material shows low density (4.5 g/cm3), fracture toughness about 20 MPam1/2 in temperature region 20-800°C as cast, and 40-50 MPam1/2 as deformed, the Young modulus is 140 GPa, ultimate tensile strength at 800°C is 200-400 MPa, hardness is 40-50 HRC, good oxidation resistance at elevated temperature, low heat conductivity (4-9 W/m K) and nonflammable at high temperature. These materials are competitive with -TiAl-based materials and MMC reinforced with SiC fibers. Their costs are the practically comparable with of conventional titanium alloys. Destination of Materials in automotive, aerospace and space, including: Structural materials with high specific strength, Adiabatic diesel engine parts, Rotor turbo-chargers, Wear resistant and brake parts, Corrosion and abrasion resistant parts (seawater, acids, alkalis, etc).

5:25 PM Invited
Metal Matrix Composite LOX Turbopump Housing Via Novel Tool-Less Net-Shape Pressure Infiltration Casting Technology: Sandeep Shah[2]; Jonathan Lee[2]; Biliyar Bhat[2]; Doug Wells[2]; Wayne Gregg[2]; Matthew Marsh[3]; Gary Genge[3]; John Forbes[3]; Alex Salvi[1]; *James A. Cornie*[1]; Michael Sung[1]; Shi-yu Zhang[1]; [1]Metal Matrix Cast Composites, Inc. (MMCC), USA; [2]NASA Marshall Space Flight Center, Eng. Direct., USA; [3]NASA Marshall Space Flight Center, Transportation Direct., USA

Metal matrix composites (MMC) offer relatively higher specific strength, specific stiffness, lower coefficient of thermal expansion (CTE) and lower density as compared with conventional alloys. These unique properties make them very attractive for aerospace turbomachinery applications where there is ever increasing emphasis to reduce weight and cost, and increase engine performance. Through a joint effort between NASA and MMCC, Inc., a complex liquid oxygen (LOX) compatible turbopump housing is being redesigned and manufactured from hybrid (particulate and fibers) Aluminum MMC. A revolutionary tool-less pressure infiltration casting technology is being perfected. Ceramic preforms for the composite are 3-dimensionally printed using a stereolithography file, acquired from a CAD model. The preforms are then invested into a refractory material and pressure infiltrated with liquid metal. After casting, the refractory material is washed away leaving behind a near net-shape composite part. Benefits of this process include increased composite uniformity, no mold machining, short time from design to part, properties matching traditional methods, ability to create previously impossible to manufacture parts and no size limitations with newly developed joining technology. The results of materials, manufacturing and design optimizations, preform joining, and sub-element tests will be presented.

Applications of Computational Thermodynamics in Materials Processing - I

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee
Program Organizers: Qingyou Han, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; Ravi Vijayaraghavan, Ford Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Monday PM — Room: 111
November 5, 2001 — Location: Indiana Convention Center

Session Chairs: Ravi Vijayaraghavan, Ford Motor Company, Scientific Rsrch. Lab., Dearborn, MI 48121-2053 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Rsrch. Lab., Dearborn, MI 48121-2053 USA

2:00 PM Opening Remarks: Qingyou Han

2:05 PM Invited
Guiding Alloy Design and Processing Via a Hybrid First-Principles/Computational Thermodynamics Approach: *Christopher Mark Wolverton*[1]; John Allison[1]; Xin-Yan Yan[2]; [1]Ford Motor Company, MD 3028/SRL, PO Box 2053, Dearborn, MI 48121 USA; [2]University of Wisconsin, Dept. of Matls. Sci., 1509 University Ave., Madison, WI 53706 USA

Computational thermodynamic approaches have become a valuable tool in the calculation of complex, multicomponent phase equilibria in industrial alloys. These methods rely on databases of thermodynamic information for the wide variety of phases which can occur during processing. However, many phases of practical interest, such as metastable precipitate phases, are often absent from current databases, and obtaining thermodynamic functions for these phases from experiment is often problematic due to their metastability. We demonstrate that first-principles, quantum-mechanical calculations provide a means to obtain thermodynamic functions of phases absent from present databases. Hence, a hybrid first-principles/computational thermodynamics approach can be used to predict metastable phase equilibria of industrial alloys during heat treatment. We illustrate our experiences with this novel approach in the heat treatment of the casting alloy, Al 319. This hybrid approach can yield phase fraction information of metastable precipitates, which may, in turn, be used in mechanical properties models.

2:35 PM
Coupling CALPHAD Databases and the Phase Field Approach to Predict Interdiffusion Microstructures in Multi-Component Systems: *Kaisheng Wu*[1]; John E. Morral[2]; Yunzhi Wang[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 344 MacQuigg Lab., 2041 College Rd., Columbus, OH 43210 USA; [2]University of Connecticut, Dept. of Metall. & Matls. Eng., 97 N. Eagleville Rd., Box U-136, Storrs, CT 06269-3136 USA

The microstructures arising from interdiffusion between two multi-component and multiphase materials are usually complicated and simulations were limited to simplified conditions. To deal with the problem in a more general way, a novel method was developed by integrating databases obtained by the CALPHAD (CALculation of PHase Diagrams) method into the phase field approach. The model was first applied to proto-type two-phase diffusion couples containing Type 0 boundaries. The effects of concentration dependence of diffusivity, capillarity, thermodynamic properties and coherent stress on the evolution of interdiffusion microstructures and diffusion paths were investigated. A real alloy system was then considered by using the CALPHAD database for the thermodynamic properties. In this presentation the simulation results will be compared with experimental observations and the results of an error function analysis. The practical application of the approach to the design of NiCrAlY coating materials for gas turbine engines will also be discussed.

3:00 PM
Development of Consistent Multicomponent Thermodynamic Databases: *Fan Zhang*[1]; *Fanyou Xie*[2]; Stephen L. Daniel[1]; Shuanglin Chen[1]; Y. Austin Chang[2]; [1]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA; [2]University of Wisconsin–Madison, MS&E, 1509 University Ave., Madison, WI 53706 USA

The best way to speed up development of new materials is to predict alloy properties with thermodynamic calculations. Thermodynamic calculations can be used to select alloy chemistry and to optimize processing parameters such as heat treatments. Thermodynamic calculations, if successful, will save considerable time and money during materials research and development. The success of this computational approach depends on the reliability of both thermodynamic databases and the phase equilibrium calculation software. Our experience in the construction of multicomponent database for aluminum based alloys has revealed that many poor system descriptions have been obtained through the use of software which is not able to calculate the stable phase diagrams automatically. Examples of these shortcomings will be presented. It will be shown that how a consistent reliable database can be developed when the appropriate software for phase equilibrium calculation is available.

3:25 PM
Thermodynamic Modeling of the Nb-Hf-Si Ternary System: Ying Yang[1]; [1]University of Wisconsin–Madison, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53705 USA

The thermodynamic description of the Hf-Si system was modified by considering the critical DTA results of Hf-Si binary system and by analyzing the solidification path in the Nb-Hf-Si ternary system. Combining the thermodynamic description of the Nb-Si and Nb-Ti systems available in literature with that of the modified Hf-Si system, a thermodynamic modeling of the Nb-Hf-Si system was performed via the CALculation of PHAse Diagram (CALPHAD) approach. The optimization and the calculation were carried out in the Thermo-calc software. The calculated results using the optimized parameters show reasonable agreement with the experimental information at 1500°C. The liquidus projection close to Nb-Hf binary side is validated through Scheil solidification simulation, which yields good agreement with the observed microstructure of the as-cast alloys.

3:50 PM Break

4:00 PM
Multicomponent Phase Equilibria: Application in Microscopic Modeling of Multicomponent Aluminum Alloys: *Xinyan Yan*[1]; Shuanglin Chen[2]; Y. Austin Chang[1]; [1]University of Wisconsin–Madison, Dept. of Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA; [2]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA

To accurately predict the microstructure and microsegregation in multicomponent alloys, multicomponent phase equilibrium information is required, such as concentrations at liquid-solid interface, liquidus slope and partition coefficients. PanEngine, a recently developed multicomponent phase equilibrium calculation interface, meets this requirement. This interface provides customers with flexibility to create user application programs such as solidification and heat treatment simulation. Examples on applying this interface in microscopic modeling will be given. Using this interface, thermodynamic calculation was directly linked to micromodel codes that consider main kinetic and thermodynamic effects, such as solid back diffusion, dendrite arm coarsening and thermodynamic correction of interface concentration. Model predicted phase fractions, secondary dendrite arm spacing and solute concentration profiles are in good agreement with the experimental data for commercial aluminum alloys. The micromodel, combined with Al-database, can be used as a powerful tool to predict microstructure and microsegregation in aluminum alloys under different cooling rates.

4:25 PM
Thermodynamics-Based Semi-Empirical Description of Partition Coefficients in Ternary Al-Cu-Si and Al-Mg-Si Systems: *Janos Farkas*[1]; Andras Roosz[1]; George Kaptay[2]; Janos Szoke[1]; [1]University of Miskolc, Dept. of Phys. Metall., Egyetemvaros, Miskolc 3515 Hungary; [2]University of Miskolc, Dept. of Phys. Chem., Egyetemvaros, Miskolc 3515 Hungary

Equilibrium partition coefficients of solutes between solid and liquid solutions serve as basic thermodynamic information for the calculation of solidification paths in multi-component alloys. In the present paper a semi-empirical thermodynamic formalism and the appropriate algorithm has been developed to describe the partition coefficients for the family of multi-component alloys having the same solvent. Although the semi-empirical coefficients of this model are found from the best-fit analysis to experimental data, there are certain relations between some of these coefficients, i.e. the model for the n-component system can be built step by step from the binaries via ternaries and (n-1)-component systems. The algorithm is verified for two ternary systems (Al-Cu-Si and Al-Mg-Si). The semi-empirical coefficients are found using the information from the binary diagrams and from the eutectic valleys of the ternary diagrams. The part of semi-empirical constants for the description of the partition coefficient of Si appears to be identical for the two ternary systems.

4:50 PM
PANDAT: Multi-Component Phase Diagram Calculation Software: *Shuanglin Chen*[1]; *Stephen L. Daniel*[1]; *Fan Zhang*[1]; Y. Austin Chang[2]; [1]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA; [2]University of Wisconsin–Madison, MS&E, 1509 University Ave., Madison, WI 53706 USA

PANDAT is a Windows-based software program for calculating phase diagrams and thermodynamic properties in multi-component systems. Combining a powerful phase equilibrium calculation engine, PanEngine, with a user-friendly Microsoft Windows-based graphical interface, PANDAT performs complex calculations with only a few mouse clicks. It does not require initial values from the user, not even to find a miscibility gaps. Functions and applications of PANDAT for real alloy systems will be presented, such as calculating phase diagrams, liquidus projections, and simulating solidification paths.

Friction Stir Welding and Processing - I: Process Modeling and Modifications

Sponsored by: Materials Processing and Manufacturing Division, Shaping and Forming Committee, Texture & Anisotropy Committee
Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; Shigeo Saimoto, Queens University, Ontario K1L 3NG Canada; S. L. Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Monday PM — Room: 115
November 5, 2001 — Location: Indiana Convention Center

Session Chairs: Michael Uchic, Air Force Research Laboratory, WPAFB, OH 45433-7817 USA; D. Nicholas, The Welding Institute, Innovation Unit, Cambridge, Cambridgeshire CB1 6AL UK

2:00 PM Opening Remarks

2:05 PM
Friction Based Technologies for Joining and Processing: *W. M. Thomas*[1]; S. W. Kallee[1]; E. D. Nicholas[1]; [1]The Welding Institute, Innovation Unit, Granta Park, Great Abington, Cambridge, Cambridgeshire CB1 6AL UK

Friction, which requires relative motion, deformation and time, is an efficient thermal energy source for the welding and reprocessing of materials. Friction based technology is now extensively used in industries as divergent as sub-sea and aerospace. This paper will describe some of the variants of friction welding and material processing techniques, with particular reference to their relationship to Friction Stir Welding and Friction Stir Processing.

2:35 PM
Tool Technology for Friction Stir Welding Applications in the Automotive Industry: *Jorge F. dos Santos*[1]; [1]GKSS Forschungszentrum, Inst. for Matls. Rsrch., Joining Tech., Max-Planck-Str, Geestahcht D-21502 Germany

In friction stir welding (FSW), plasticised material is stirred and forged behind the trailing face of the pin tool, where it consolidates and cools down to form the solid state weld. In the present study alternative tool designs are proposed which, due to their self-contained construction, absorb the process forces reducing the requirements for clamping and fixturing devices. Two basic tool designs have been developed for standard configurations, particularly butt and overlap joints. Furthermore, these tools are optimised for long and short welds as found in real components and assemblies in the automotive industry. Results of preliminary trials with different tool designs are presented and discussed based on actual bodywork and suspension components welded by robotic FSW.

3:05 PM

Circumferential FSW using "Bobbin Tool" Technology: *Douglas J. Waldron*[1]; [1]The Boeing Company, Phantom Works, 5301 Bolsa Ave., MS H013-C317, Huntington Beach, CA 92647 USA

Friction stir welding is a revolutionary method of joining aluminum structures that has reduced the cost of welding launch vehicle pressurized propellant tanks by a factor of six. The first flight ever of friction stir welded hardware, occurred August 17, 1999 with the successful launch on a Boeing Delta II rocket. Boeing has established itself as a world leader in this technology and has maintained this position as over 80,000 inches of longitudinal production welds have been made for Delta II, III and IV family of launch vehicle components. Emerging technology within the friction stir process has now begun to focus on welding circumferential weld joints using the revolutionary self-reacting or "bobbin tool" technology. This presentation will cover the aspects of the use of "bobbin tool" technology and its associated unique microstructures and process benefits and how Boeing is using this technology.

3:30 PM

Alloy, Tool Geometry, and Temper Effects on Resultant FSW Process Parameters and Joint Properties: *Tony Reynolds*[1]; Wei Tang[1]; [1]University of South Carolina, Dept. of Mechl. Eng., 30 Main St., Rm. A224, Columbia, SC 29208 USA

An attempt has been made to systematically examine controlled friction stir weld process parameters (e.g. rpm and welding speed), tool geometry, and alloy temper effects on weld properties and weld process variables (e.g. required forces and torques). Welds have been made in several aluminum alloys using FSW parameters chosen to provide different levels of specific weld energy and the required forces and torques recorded. Tool geometry effects on weld strength and resultant process parameters have been quantified for the aluminum-lithium alloy, 2195. Trends suggested by the data and directions for further exploration will be discussed.

3:55 PM Break

4:05 PM

Material Flow during Friction Stir Welding: *Mario Guerra*[1]; *John C. McClure*[1]; L. E. Murr[1]; A. C. Nunes[2]; [1]University of Texas–El Paso, Metall. & Matls. Eng. Dept., El Paso, TX 79968 USA; [2]NASA, Marshall Space Flight Ctr., Huntsville, AL 35812 USA

The flow of metal during Friction Stir Welding is clarified using a faying surface tracer and a nib frozen in place during welding. It is shown that material is transported in two distinct streams or currents. One stream has its source on the advancing front side of the nib. It is wiped onto a plug of material that rotates and advances with the nib. As the plug rotates and advances the entrained material descends within the plug in the wash of the threads on the nib in a helical motion. After multiple rotations, this material is sloughed off the plug in the wake of the tool primarily on the advancing side. Weld metal volume is conserved by a rising flow outside the plug compensating for the downward flow within. The second stream of material has its source on the retreating front side of the nib. It fills in the wake primarily on the retreating side and intercalates between the sloughed-off areas of the multiply rotated stream. Material from each of these two streams has different mechanical properties. The strength of a weld should depend on the relative dominance of the stream products.

4:30 PM

Modeling Friction Stir Welding of Titanium and Aluminum Alloys: *R. L. Goetz*[1]; Kumar V. Jata[2]; [1]UES, Inc., Dayton, OH, USA; [2]AFRL/MLLM, 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA

Friction stir welding (FSW) is a solid state welding process primarily for aluminum alloys and aerospace applications. However, there is also interest in using FSW for higher temperature aerospace materials such as titanium alloys. In light of this, FSW of Ti-6Al-4V was modeled and compared to aluminum alloys using a finite element model. The model was used to predict the tool and workpiece temperatures for various tool rpm's and plunge rates, as well as loads and deformation behavior. The effect of various tool materials was also examined with respect to tool and workpiece temperature response.

4:55 PM

Modeling, Analysis, and Validation of Friction Stir Welding Processes: *Askari Abe*[1]; Stewart Silling[2]; Blair London[3]; Murray Mahoney[4]; [1]The Boeing Company, PO Box 3707, MC 7L-25, Seattle, WA 96124-2207 USA; [2]Sandia National Laboratories, Computl. Phys. & Mech. Dept., PO Box 5800, MS 0820, Alburquerque, NM, USA; [3]California State University, Matls. Eng. Dept., 1 Grand Ave., San Luis Obispo, CA 93407 USA; [4]Rockwell Science Center, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA

Science-based simulation and modeling toolkits, once validated, can enable expansion of FSW technology by providing process design and optimization. Models reduce cost and cycle time by focusing and guiding subsequent laboratory testing and process qualification. Boeing and collaborators have developed a computer model for FSW using a 3D, thermomechanically coupled finite element analysis. Our model includes important physical effects with a minimum of assumptions. The model includes frictional heating and energy dissipation due to plastic work, thermal convection and conduction, and predicts the metal flow path and mixing between workpiece materials. All relevant geometrical details, including pin shoulder design are included in the model. Detailed post-weld analysis in high-strength aluminum alloys is used to validate the computational model. Placement of tracers in the weld path, post-weld metallographic examination, and computer tomography are used to identify both the in-situ and equilibrium history of metal flow dynamics. With this model, detailed thermal and deformation histories can be predicted and exploited to improve post-weld microstructures and properties.

5:20 PM

Multi-Axis Friction Stir Welding: *Stanley M. Howard*[1]; Jon J. Kellar[1]; Michael Langerman[2]; Carl Larsen[3]; Michael Skinner[3]; [1]South Dakota School of Mines and Technology, Matl./Met. Eng., 501 E. St. Joseph St., Rapid City, SD 57701-3995 USA; [2]South Dakota School of Mines and Technology, Mechl. Eng., 501 E. St., Joseph St., Rapid City, SD 57701 USA; [3]MTS, Eden Prarie, MN, USA

The South Dakota School of Mines and Technology (SDSM&T), working with its industrial partner, MTS Systems Corporation (MTS) of Eden Prairie, Minnesota, has begun a joint research program in Friction Stir Welding (FSW) research. Presently, FSW is done on single axis machines typically performing flat plane welds joining soft metals, primarily aluminum. New alloys need to be developed that are more amenable to this superior joining process and novel machines need to be designed that can join curvilinear seams of thick section aluminum and other materials. The research will take the present FSW technology and expands it to a five-axis machine capable of curvilinear welds. The machine will be designed such that it is capable of performing welds on thick section aluminum and other materials. This presentation will detail the current status of the research effort and give an overview of future research efforts.

General Abstracts: High Temperature Alloys - I

Sponsored by: TMS

Program Organizers: TMS, Warrendale, PA 15086 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Monday PM — Room: 105
November 5, 2001 — Location: Indiana Convention Center

Session Chair: Eric M. Taleff, The University of Texas at Austin, Matls. Sci. & Eng. Prog., Austin, TX 78705 USA

2:00 PM

Deformation Mechanisms in Nb-40Ti-15Al-5Cr Alloy: *Sundar Amancherla*[1]; Gopal B. Viswanathan[1]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA

This paper focuses on the deformation mechanisms observed in Nb-40Ti-15Al-5Cr alloy (referred to as 5Cr alloy). The 5Cr alloy shows planar slip when compressed in solutionized condition at room temperature as has been observed in the ternary alloy Nb-40Ti-15Al (base alloy) despite the fact that the 5Cr alloy work hardens and the base alloy work softens. Slip bands have been observed in the microstructure of the compressed sample, and are oriented in different directions when examined in SEM and TEM. The TEM observations also indicate these slip bands are localized on {110} and {112} type slip planes. In contrast to the base alloy, the 5Cr alloy shows significant tendency for cross slip indicated by diffuseness of slip planes when observed in edge-on orientation in TEM. The 5Cr alloy also exhibits massive dislocation density within slip bands with different sets of dislocations oriented in different directions when observed in planar view in TEM. These alloys also show a phase transformation from B2 to the orthorhombic phase at intermediate temperatures. In addition to the deformation mechanisms within the B2 matrix, this paper also discusses the slip transmission from the B2 to the orthorhombic phase in the base alloy as well as the 5Cr alloy. This research was supported in part by Reference Metals Company, Inc.

2:20 PM

The Ordering Tie Line (OTL) Calculation and Representation Applied to Quaternary B2 Alloys: *Sundar Amancherla*[1]; Rajarshi Banerjee[1]; Hamish L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA

This paper focuses on the calculation and prediction of ordering schemes in quaternary B2 alloys. The computational model is an extension of that proposed previously for ternary B2 alloys based on the framework of Bragg-Williams theory of order-disorder transformations. A chemical reaction rate approach is used to represent the atomic exchange mechanism that takes place in the process of attaining an equilibrium state of ordering at a particular temperature. The Ordering Tie Line (OTL) representation is used to geometrically depict the predicted ordering scheme. Furthermore, the dependence of the six binary interaction parameters representing the quaternary system, on the orientation of the OTL will be discussed in detail. Experimental results from Nb-40Ti-15Al-5Cr alloy will be presented to compare and contrast the predicted OTLs.

2:40 PM

Processing and Microstructure of Mo-Si-B Alloy: *Saeed Bejnood*[1]; Peter K. Liaw[1]; Raymond A. Buchanan[1]; Chain T. Liu[2]; [1]University of Tennessee, Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA

The processing and microstructures of Mo-Si-B alloy have been studied. The arc-melting process, followed by drop casting into cooper chill mold was applied to fabricate the Mo-Si-B specimen. The specimens were annealed at 1200, 1400, and 1600°C for the control of multi-phase microstructures. The microstructures of heat-treated samples were compared with non heat-treated samples regarding the grain size and phase morphology. The investigation of the intermetallic phases, Mo5Si3 (T1), Mo5SiB2 (T2) and Mo3Si is necessary to provide bases for further improvements of the compositions to enhance the ductility and fracture toughness of Mo-Si-B alloy. Moreover, the high-temperature optical floating zone furnace will be used to process the drop-casting materials with the hope of developing lamellar microstructures in the Mo-Si-B alloy, thus improving the ductility and fracture toughness. This research was founded by the National Science Foundation (NSF) through an Integrative Graduate Education and Research Training (IGERT) program (DGE-9987548) with Dr. W. Jennings and Dr. L. Goldberg as contact monitors.

3:00 PM

The Electrical Resistivity and Specific Heat of a Co-Cr Alloy (ULTIMET) from 25 to 1100°C: Douglas Falcon[1]; Wally Porter[2]; *Charlie R. Brooks*[1]; Peter K. Liaw[1]; Dwaine Klarstrom[3]; [1]The University of Tennessee, Matls. Sci. & Eng. Dept., Knoxville, TN 37996 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., Oak Ridge, TN 37831 USA; [3]Haynes International, 1020 W. Park Ave., Kokomo, IN 46904 USA

ULTIMET alloy is a Co-Cr-base alloy containing nominally (wt. %) 26 Cr, 9 Ni, 3 Mo, 3 Fe, 0.8 Mn, 0.3 Si, 0.08 N and 0.06C. At high temperatures (above approximately 900°C) the structure is face-centered cubic alpha, and at lower temperatures the equilibrium structure is hexagonal close-packed epsilon and sigma. Extensive aging is required to form sigma, and it did not appear in this study. The formation of epsilon is suppressed by cooling rapidly from high temperatures. However, the microstructure may show thin sheets of stacking faults and twins, depending on the cooling rate. Plastic deformation induces the formation of these structures, and eventually the epsilon phase forms. The electrical resistivity and specific heat were measured simultaneously from 25 to approximately 1100°C using pulse-heating calorimeter. The heating rate was typically 1000°C/s. Also the specific heat was measured using differential scanning calorimetry at a heating rate of approximately 10°C/min. The resistivity increase smoothly from 108 microohm-cm at 25°C to about 130 at 1100°C. The specific heat increases from about 0.45 J/gm-C at 25C to about 0.7 at 1100°C. However, there is an indication of a phase change near 650°C. The results are discussed in terms of the alpha to epsilon phase change. Part of this research was supported by the National Science Foundation under the Small Grant for Exploratory Research program; Dr. Bruce McDonald is the technical monitor. The material was supplied by Haynes International. The DSC measurements were made at the High Temperature Materials Laboratory user facility at Oak Ridge National Laboratory.

3:20 PM Break

3:40 PM

Gamma Prime Precipitate Morphology in CMSX-2 Superalloy: *Alejandro Mario Ges*[1]; Osvaldo Fornaro[2]; Hugo Aníbal Palacio[1]; [1]Universidad Nacional del Centro de la Provincia de Buenos Aires, Instituto de Física de Materiales Tandil-CICPBA, Pinto 399, Tandil, Buenos Aires B7000GHG Argentina; [2]Universidad Nacional del Centro de la Provincia de Buenos Aires, Instituto de Física de Materiales Tandil-CONICET, Pinto 399, Tandil B7000GHG Argentina

The aim of the present work is to describe the effect of the heat treatment and the thermal exposure upon the gamma prime precipitate in a CMSX-2 superalloy. The samples were obtained from bars vacuum induction melted, which were solidified in polycrystalline way. The application of initial heat treatment such as solution (SHT), post-solution (PSHT) and precipitation heat treatment (PHT) permit us to obtain different size and distribution of gamma prime. Afterwards, the samples were artificially aged at different constant temperature, and studied in absence of applied strength. The evolution of the rate constant k at several temperatures was estimated and subsequently the activation energy for coarsening was determined. The gamma prime size against microhardness was evaluated. These observations and their possible implications on the behavior of these materials at high temperature were discussed.

4:00 PM

Microstructure Control and Tensile Properties of the γ-Fe/k-(Fe,Mn)$_3$AlC Two-Phase Alloys: *Kunio Hayashi*[1]; Yoshisato Kimura[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Eng., 4259 Ngatsuta, Midori-ku, G3-513, Yokohama 226-8502 Japan

The E2$_1$ intermetallic compound (Fe,Mn)$_3$AlC, k phase, would be an excellent strengthener of the fcc γ-Fe based heat resisting alloys, since the E2$_1$ ordered structure quite resembles that of the L1$_2$ which is a well-known strengthener of Ni-base superalloys. In the present work, the γ/k two-phase Fe-Mn-Al-C alloys with relatively high carbon contents (around 8at%) were selected to stabilize k phase above 1000K. A typical microstructure of annealed alloy is that k-E2$_1$ precipitates as cubes and plates in the γ-Fe matrix having about 3% lattice mismatch. Aging treatment after water quenching from the γ-Fe field yields the lamellar-type microstructure formed by discontinuous precipitation. This heat treatment drastically improves tensile ductility of the alloys through microstructural change at temperatures from 300 to 1073K without sacrificing strength. Moreover, effect of the 5th element addition, such as Cr and W, on microstructures, γ/k lattice mismatch and tensile properties were systematically investigated.

4:20 PM

Creep Behavior of Ti-6Al-2Sn-4Zr-2Mo: *Robert Walter Hayes*[1]; Babu Viswanathan[2]; Mike Mills[2]; [1]Metals Technology, Inc., 19801 Nordhoff St., Northridge, CA 91324 USA; [2]The Ohio State University, 2041 N. College Rd., Columbus, OH 43210 USA

We have shown previously that the addition of trace levels of Ni to the alloy Ti-6Al-2Sn-4Zr-2Mo (Ti-6-2-4-2) increases the magnitude of primary creep strain as well as steady-state creep rate. The presence of Ni in Ti-6-2-4-2 leads to an acceleration in steady-state creep rate in the same manner as it leads to enhanced rates of Ti self and Al substitutional diffusion within the alpha phase of Ti thus suggesting a strong link between creep and diffusion behavior in the presence of certain trace impurities. Our previous results were obtained from creep studies of two heats of Ti-6-2-4-2 having low (0.005 wt.%Ni) and high (0.035 wt.%Ni) Ni concentrations. Based upon our previous findings, it was decided to extend this work using a third heat of material having an intermediate (0.014 wt.%Ni) Ni concentration. We find that the third heat of material behaves as expected reinforcing what is now realized regarding the influence of trace levels of Ni on diffusion and creep behavior in titanium. Details of the deformation structures are presented along with creep data and the creep mechanism pertaining to the temperature and stress levels investigated is discussed.

4:40 PM

Processing, Microstructure, and Mechanical Behaviors of the Cr-Ta Alloy Composites Reinforced by the Cr2Ta Laves Phase: Yuehui He[1]; *Daifeng Wang*[1]; Peter K. Liaw[1]; Chain T. Liu[2]; Lee Heatherly[2]; Easo P. George[2]; [1]University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., Oak Ridge, TN 37831 USA

The process of directional solidification (DS) using an optical high-temperature floating zone furnace to obtain an aligned eutectic microstructure of a binary eutectic Cr-Ta alloy was studied in order to improve the ductility and fracture toughness. The effects of DS conditions (growth speeds and rotation rates) on the microstructures of Cr-(9.0-9.8) at.% Ta alloys in pure argon were examined. It was found that a high growth speed of 100 mm/h was necessary for the formation of an aligned microstructure of the Cr-Ta alloy. The influence of DS conditions and heat-treatments on the hardness at room temperature and tensile properties at room and elevated temperatures (800ºC and 1,200ºC) was investigated. It was observed that the tensile properties of the Cr-Ta alloys at room and elevated temperatures could be improved by obtaining aligned microstructures, and that the fracture toughness at room temperature of the directionally solidified alloy was increased. This trend is mainly associated with crack deflection and the formation of shear ribs in the samples with aligned microstructures.

General Abstracts: Industrial Innovations, Corrosion & Environmental Effects, and Electronic Packaging & Interconnection Materials

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Monday PM Room: 117
November 5, 2001 Location: Indiana Convention Center

Session Chair: Michael McNallan, University of Illinois at Chicago, Matls. Sci., 842 W. Taylor St., MC 246, Chicago, IL 60607-7021 USA

2:00 PM
Heat Transfer, Morphologies and Microstructures of Copper Shells Solidified on Substrates: *Dominique Bouchard*[1]; *François G. Hamel*[1]; *Serge F. Turcotte*[1]; *Jean-Paul Nadeau*[1]; *Daniel Simard*[1]; [1]Industrial Materials Institute, 75 de Mortagne, Boucherville, Quebec J4B 6Y4 Canada

Solidification aspects of copper have been studied by a series of immersion tests. Copper blocks were dipped into the molten metal for short time periods during which a thin shell solidified. The heat transfer between the blocks and the shells was characterized using an inverse method. The morphologies and microstructures of the shells were also examined. They were significantly affected by the various substrate surface conditions that were prepared. The implication of these results for the twin roll casting process of copper strips will be discussed.

2:20 PM
The Effect of Casting Speed on the Solidification Uniformity in the Twin-Roll Strip Casting Process: *François G. Hamel*[1]; Dominique Bouchard[1]; Jean-Paul Nadeau[1]; Daniel Simard[1]; Serge F. Turcotte[1]; [1]National Research Council, Indl. Matls. Inst., 75 Blvd. de Mortagne, Boucherville, Quebec J4B 6Y4 Canada

Non-uniform temperature patterns have been observed during the formation of strips in the twin-roll casting process for steel and copper alloys. Zones of different temperatures are responsible for thermal stresses and cracks. Previous investigations have shown a correlation between the size of the temperature pattern and the casting speed. The proposed mechanisms were related to the influence of the roll velocity on the curvature of the meniscus and the depth of the roll-melt contact point. Recent results obtained with copper substrates dipped at constant velocity into molten copper have shown that the solidification time is a key factor which determines the size of the zones of uneven solidification. The proposed mechanism is that a metal solidifying on a substrate behaves like a series of barely connected droplets which form bumps that later coalesce and grow in size at the detriment of their neighbors.

2:40 PM
Solidification of Hot-Dip Galvanized Layer by Electrostatically Charged Aerosol Particles: *Sangheon Kim*[1]; [1]Pohang Iron and Steel Company, Kwangyang Rolling Prod. Rsrch. Grp., Tech. Rsrch. Labs, Kumho-Dong, 699, Kwangyang-Shi, Cheonnam 544-090 S. Korea

A novel electrostatic spraying method for solidifying a galvanized coating layer was studied. Our experimental results and computer simulations showed that electric field could assist the fine droplets to attach on the steel surface and change the sprayed droplets trajectory especially in the space near the steel surface. It was necessary to apply the electric voltage higher than 20 kV to obtain the enough electrostatic attraction force between droplets and the steel sheet. Owing to the phenomena so called electro-hydrodynamic atomization, the large particle break down into small ones with the applied high voltage such as 40 kV. Thus, by using the electrostatically charged aerosol droplets, we could easily produce the spangle free galvanizing coating layer.

3:00 PM Break

3:20 PM
Modes of Oxidation and Nitridation in Two-Phase Cr(Pt)-Cr3Pt Alloys with Potential Application to Nanocomposite Synthesis: *Sarah Kate Baham*[1]; Michael P. Brady[2]; Peter K. Liaw[1]; Raymond A. Buchanan[1]; Thomas A. Lograsso[3]; [1]University of Tennessee, Matls. Sci. & Eng., 434 Dougherty Engineering, Knoxville, TN, USA; [2]Oak Ridge National Laboratory, Corrosion Sci. & Tech. Grp. & Alloying Behavior & Design Grp., PO Box 2008, MS 6115, Oak Ridge, TN 37831 USA; [3]Ames Laboratory, Iowa State University, 111A Metals Development, Ames, IA 50011 USA

Oxidation reactions in multi-phase alloys yield an incredibly wide variety of surface reaction products. Such reactions are of great relevance to the design of heat-resistant alloys, but are also potentially useful for the controlled synthesis of functional surfaces. In support of these applications, the oxidation and nitridation behavior of model single-phase Cr(Pt) and Cr3Pt alloys were studied individually and in combination at 800°C and 1000°C. Preliminary results suggest that that nitridation exactly followed the underlying two-phase microstructure, which led to the formation of a dispersion of the Cr3PtN perovskite phase, derived from Cr3Pt metal precipitates. Interestingly, the nitridation behavior of the Cr3Pt phase differed when present as the matrix phase as opposed to as a second phase precipitate. Details of the modes of oxidation, with an emphasis on the role of the interphase interfaces, will be presented. This work is funded by the National Science Foundation (NSF) through an Integrative Graduate Education and Research Training (IGERT) Program (DGE-9987548) with Dr. W. Jennings and Dr. L. Goldberg as contract monitors, and is also supported by a Process Science Initiative grant in collaboration with the Materials Preparation Center at Ames Laboratory.

3:40 PM
Comparison of the Corrosion Behavior and Properties of a Zirconium Based Bulk Amorphous Metal with its Crystalline State: *W. H. Peter*[1]; R. Buchanan[1]; C. T. Liu[2]; P. Liaw[1]; J. Horton[2]; C. Carmichael[2]; J. Wright[2]; [1]University of Tennessee, Matls. Sci. & Eng. Dept., Dougherty Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, MS6115, Oak Ridge, TN 37831-6115 USA

Exciting recent advances have led to “bulk amorphous metals” (BAMs) with fabrication diameters as large as 20 mm. Although considerable mechanical testing has been conducted, little corrosion testing has been performed. Early fatigue testing has resulted in fatigue strengths lower than anticipated. It is suspected that environmental effects degrade the fatigue life. A first step toward understanding the corrosion-fatigue phenomenon is to compare the corrosion behaviors of an amorphous metal and its crystalline counterpart. Amorphous samples of BAM-11, Zr52.5Cu17.9Ni14.6Ti5Al10 (at %) prepared by arc melting, were then heat treated to create a completely crystalline state. The samples were then subjected to cyclic anodic polarization tests in a 3.5% NaCl electrolyte at room temperature. The test results yielded corrosion potentials, pitting potentials, corrosion current densities, and corrosion rates. The modes of corrosion for both the amorphous and crystalline samples were identified microscopically. Preliminary results indicate a large difference in pitting potentials between the crystalline and amorphous samples. This research effort would not be made possible without the funding of the National Science Foundation Integrative Graduate Education and Research Training Program (DGE 9987548).

4:00 PM
Kinetics of Diffusionally Accommodated Sliding at Model: *Keith Andrew Peterson*[1]; Indranath Dutta[2]; [1]Naval Postgraduate School, Ctr. for Matls. Sci. & Eng., 700 Dyer Rd., Bldg. 245, Rm. 131B, Monterey, CA 93943 USA; [2]Naval Postgraduate School, Ctr. Matls. Sci. & Eng., 700 Dyer Rd., Bldg. 245, Rm. 317, Monterey, CA 93943 USA

An experimental approach was developed to quantify the kinetics of creep-sliding at planar metal-semiconductor interfaces in order to address potential reliability concerns related to back-end structures in microelectronic devices. Large interfacial shear stresses may develop near the edges of thin film interconnect lines during thermal excursions due to CTE mismatch between the metal line and the die/inter-layer dielectric (ILD). This can drive interfacial creep at high homologous temperatures, the effect becoming more prominent with decreasing line dimensions. In the present work, the steady state creep rate at planar interfaces was studied using a double-shear specimen geometry, using diffusion-bonded Al-Si as a model system, under applied loads which were much smaller than that required to cause interfacial fracture. A creep law applicable to diffusional sliding at interfaces between dissimilar materials is proposed, and the role of interfacial morphology on the kinetics of interfacial creep is assessed. The law describes diffusionally accommodated interfacial sliding, which has been observed in a several multi-component materials systems. This research was supported by National Science Foundation grant # DMR 0075281 and an NCCOSC Graduate Student Fellowship.

General Abstracts: Refractory Metals

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Monday PM Room: 212
November 5, 2001 Location: Indiana Convention Center

Session Chairs: David M. Stepp, US Army, Matls. Sci. Div., Research Triangle Park, NC 27709-2211 USA; John J. Stephens, Sandia National Labs, Albuquerque, NM 87185-0367 USA

2:00 PM
Effect of Target Structure on Film Properties in W-Ti Alloy Sputtering: *Paul S. Gilman*[1]; Chi-Fung Lo[1]; [1]Praxair-MRC, 542 Rt. 303, Orangeburd, NY 10962 USA

The effect of sputtering target microstructure, density, existing phase and configuration on the film properties of W with 10wt% Ti was investigated. Various planar targets with diameters ranging from 10.16cm to 29.64cm and 0.635cm thick plus one 20.32cm diameter conical target were sputtered to the end of target life. By controlling the extent of interdiffusion between W and Ti, two different microstructures, namely multiple-phase and single-phase, were prepared. At various kilo-watt-hours of target life, sputtered films were deposited on the silicon wafers for microstructural, compositional and mechanical property evaluations. The surface morphology of the 10.16cm diameter sputtered targets were also intermittently observed after various amount of sputterings. One observation correlated the existence of W-rich lamellar microstructure with the potential generation of particles. The surface pores can be another source of particles. Although, preferred sputtering of Ti was observed in the multiple-phase targets, no significant difference in film composition between planar multiple-phase and single-phase target sputterings were detected. However, higher Ti content was measured in the planar targets than that from the conical target deposition using the same sputtering parameters. In addition, higher compressive stresses were measured in the films deposited from the single-phase target than those deposited from the multiple-phase target. It was concluded that target properties, including microstructure, density, existing phase and configuration significantly effects the properties of the W-Ti sputtered films.

2:20 PM
A Kinetic Study of the Carbochlorination of Ta_2O_5 and Sucrose Carbon Mixture: *J. A. González*[1]; D. M. Pasquevich[2]; J. B. Rivarola[1]; M. del C. Ruiz[1]; [1]Universidad Nacional de San Luis–CONICET, Instituto de Investigaciones en Tecnología Química, Chacabuco y Pedernera, C.C. 290, 5700 San Luis, Argentina; [2]Centro Atómico Bariloche, CNEA, Argentina

A kinetic study by thermogravimetry of the chlorination of a ß-Ta_2O_5-carbon mixture was performed, using chlorine gas as the chlorinating agent. The studied variables were: reaction time and temperature, Cl_2 partial pressure and composition of the Ta_2O_5-sucrose carbon mixture. The variation of the system reactivity with the experimental methodology employed was also investigated. The solids involved in the reacting system were characterized by X-ray diffraction, X-ray fluorescence and scanning electronic microscopy, among other techniques. The results obtained showed that ß-Ta_2O_5 carbochlorination is strongly influenced by the carbon percentage and by the previous treatments of the solid mixture. The variation of the system reactivity with the percentage and previous treatment of the carbon indicate that the reaction global rate might be controlled by carbon oxidation. The apparent value of activation energy found between 500 and 600°C was 34.5 kcal/mol and the order of reaction is close to zero at high partial pressures Of Cl_2.

2:40 PM
A Preliminary Study of the Carbochlorination of Fe203, Ta205 and the Ta205-Fe2O3 Mixture: *J. A. González*[1]; J. B. Rivarola[1]; M. del C. Ruiz[1]; [1]Universidad Nacional de San Luis–CONICET, Instituto de Investigaciones en Tecnología Química, Chacabuco y Pedernera, C.C. 290, 5700 San Luis, Argentina

A thermogravimetric study was performed of the carbochlorination of the following mixtures: 50% w/w hematite-Fe_2O_3-sucrose carbon, 50% w/w ß-Ta_2O_5-sucrose carbon and 1:1 (w/w) ß-Ta_2O_5-Fe_2O_3, plus 50% sucrose carbon (w/w). Pure Cl_2 was used as the chlorinating agent. The reacting solids and the residues were characterized by techniques such as X-ray diffraction, X-ray fluorescence and scanning electronic microscopy. The results obtained showed that, during Fe_2O_3 carbochlorination, $FeCl_2$ is formed at an initial stage and then becomes $FeCl_3$ and/or Fe_2Cl_6. This behavior is influenced by the temperature, the presence of carbon and the Cl_2 partial pressure. The thermodynamic calculations match this observed experimental behavior. Ta_2O_5 is less reactive to carbochlorination than Fe_2O_3, giving Ta/Cl_5 as product. The carbochlorination reaction of the Ta_2O_5-Fe_2O_3 mixture showed that the presence of Ta_2O_5 favors the formation of $FeCl_3$ and/or Fe_2Cl_6.

3:00 PM
Shock-Induced Shear Transformations in Tantalum: *Luke L. Hsiung*[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., L-352, PO Box 808, Livermore, CA 94551-9900 USA

Effects of high strain-rate and high plastic-strain deformation on the development of deformation substructures in tantalum and tantalum-tungsten alloys (Ta-2.5 wt.% W and Ta-10 wt.% W) shocked at 15 and 45 GPa have been investigated. In addition to a high-density dislocation substructure and {112}<111>-type deformation twinning, a shock-induced omega phase (hexagonal) is discovered within polycrystalline tantalum shocked at 45 GPa. Since both deformation twinning and omega transformation occur preferably in the {211} planes with high resolved shear stresses, it is suggested that both can be considered as alternative paths for shear transformations in shock-deformed tantalum. A greater volume fraction of twin and omega phase formed in Ta-W than in pure Ta reveals that shock-induced shear transformations can be promoted by solid solution alloying. While deformation twinning is resulted from a/6<111> homogeneous shear in consecutive {211} planes, omega transformation can be attributed to the a/12<111>, a/3<111>, and a/12<111> inhomogeneous shear in consecutive {211} planes. Dislocation mechanisms for shock-induced twinning and omega transformation will be proposed and critically discussed. This work was performed under the auspices of the US Department of Energy by University of California, Lawrence Livermore National Laboratory under cotract No. W-7405Eng-48. The author would like to thank Dr. D. H. Lassila for providing shock-recovered tantalum samples for this study.

3:20 PM Break

3:40 PM
Recrystallization Behavior of VAR Tantalum: *S. N. Mathaudhu*[1]; K. T. Hartwig[1]; [1]Texas A&M University, Dept. of Mechl. Eng., College Station, TX 77843-3123 USA

The objective of this work is to correlate the recrystallization temperature and recrystallized grain size with level of cold work and prior grain size for vacuum arc remelted pure tantalum. Bulk as-cast or coarse grained material was deformed to plastic strains of 1.16, 2.32, and 4.64, by equal channel angular extrusion (ECAE) at room temperature. Recrystallization heat treatments were done in vacuum to 1200°C. Results of hardness measurements and metallography are reported.

4:00 PM
A Study on the Effect of Carbon Addition on the Chlorination of M003: *Manuel W. Ojeda*[1]; Juan B. Rivarola[1]; Oscar D. Quiroga[2]; [1]Universidad Nacional de San Luis, Instituto de lnvestigación en Tecnología Quírnica (INTEQUI), Casilla de Correo 290, 5700 San Luis, Argentina; [2]Universidad Nacional de Salta–CONICET, Instituto de Investigaciones para la Industria Quírnica (INIQUI), Buenos Aires 177, Salta, Argentina

The effect of different working variables on the chlorination of M003 in the presence of carbon was studied at atmospheric pressure in an isothermal fixed bed reactor at laboratory scale, with continuous gas flow. A thermodynamic analysis performed on a set of probable chlorination and carbochlorination reactions predicts the formation of oxichlorinated compounds during direct chlorination and of chlorinated compounds during carbochlorination. The experimental results showed that: a) M003 conversion is markedly affected by temperature and reaction time, and slightly affected by chlorine partial pressure and flow; b) the identification of all the products obtained allows to propose the following carbochlorination reaction: 2 M003(1) + C(s) + 2 C12(g) —> 2 M00202(g) + C02(,); c) the presence of an active chlorinating species that would generate on the carbon surface; d) the partial pressure of 02 on the oxide surface is enough to achieve the formation of an oxichlorinated species. We conclude by proposing a mechanism based on the formation of an active species, that may account for the higher reactivity observed in carbochlorination as compared to direct chlorination.

4:20 PM
Direct Element Characterization of Refractory Metal Films using High Resolution Glow-Discharge Mass Spectrometry: *Karol Putyera*[1]; Martin Kasik[1]; Alexandre Efimov[1]; [1]Shiva Technologies, Inc., 6707 Brooklawn Pkwy., Syracuse, NY 13211 USA

The variety and concentrations of impurities, together with the elemental composition or stoichiometry, in refractory metal films have a profound effect on their physical and electrical properties. Generally, Auger electron spectroscopy (AES) and secondary ion mass spectrometry (SIMS) are the most frequently used analytical techniques to assay impurities in these films, having the capability of quantifying elements at different depths. However, these methods sustain time-consuming sample preparation and require accurate calibration using expensive standard reference materials. High-resolution glow-discharge mass spectrometry (HR GDMS) equipped with a low-pressure glow discharge ion source, can offer an alternative or complementary solution for these types of analyses. Since material atomization and subsequent ionization are separated in space and time, calibration of HR GDMS is much less matrix dependent. With higher atomization and ionization yields, HR GDMS is capable of extending applications to ultra-trace levels of detections. Analytical procedures were therefore developed to allow direct analysis of refractory metal films.

Materials Design Approaches and Experiences - I

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: Ji-Cheng Zhao, GE Corporate Research & Development, Physical Metallurgy Laboratory, Schenectady, NY 12301 USA; Michael G. Fahrmann, Special Metals Corporation, Technology Department, Huntington, WV 25705-1771 USA; Tresa M. Pollock, University of Michigan, Materials Science and Engineering Department, Ann Arbor, MI 48109-2136 USA

Monday PM Room: 204
November 5, 2001 Location: Indiana Convention Center

Session Chair: J.-C. Zhao, General Electric Company, Physl. Metall. Lab., Schenectady, NY 12301 USA

2:00 PM Opening Remarks

2:05 PM Invited

Design of Superalloys for Single Crystal Blade Applications: A 20-Year Experience: *Pierre Caron*[1]; Tasadduq Khan[1]; [1]ONERA, DMMP, 29, Ave. de la Division Leclerc, Chatillon 92322 France

For the past 20 years ONERA has developed new superalloys for single crystal turbine blade applications in aeronautical, industrial or aerospace gas turbine engines. During the 80's, the effort was made to identify chemistries promoting a good castability, a full solutioning of the γ' phase, and a good balance between the creep strength and the density. The alloys AM1, AM3 and MC2 are thus used today by SNECMA and TURBOMECA. More recently, the development of new generation superalloys MC-NG containing both rhenium and ruthenium was driven by the need for improved temperature capability and increased life of the blades. The use of predictive methods allowed to estimate some pertinent characteristics such as density, γ' solvus temperature, γ' fraction, γ and γ' phase compositions, γ/γ' mismatch and microstructural stability, for a given alloy chemistry. These predictive methodologies were particularly useful for designing appropriate alloy chemistries for tailored applications.

2:35 PM Invited

Turbine Airfoil Alloy Development in the Commercial World: *Kevin S. O'Hara*[1]; [1]General Electric Aircraft Engines, Matls. & Proc. Eng. Dept., 1000 Western Ave., MD 36802, Lynn, MA 01910 USA

Turbine airfoil alloy development is undertaken for a variety of reasons including economics, desperation and the search for knowledge; of these three reasons, one is almost inconsequential. A variety of methodologies have been followed in alloy development, but one seems to match the pace of the turbine business. Some find it surprising that alloy development is proceeding. The promise of property improvements still exists in the Ni-base system despite the large body of prior work. Indeed, this history allows specific matching of the alloy to the component mission or provides guidelines for new alloy subsystems.

3:05 PM Invited

Recent Progress in the New PHACOMP Approach: *Masahiko Morinaga*[1]; Yoshinori Murata[1]; Hiroshi Yukawa[1]; [1]Nagoya University, Dept. Matl. Sci. Eng., Nagoya 464-8603 Japan

The NEW PHACOMP approach is reviewed in regard to the quantitative analysis of the phase stability of Ni-base superalloys. The precipitation of topologically closed-packed (TCP) phases (e.g., the sigma phase) in the alloys deteriorates markedly their mechanical properties. The PHACOMP method proposed in 1964 has been widely used for predicting the compositions of those alloys which are free from such precipitates. Its predictions are, however, very poor. The NEW PHACOMP proposed by us in 1984 provides a better tool for the prediction of phase stability. The new parameter used in this approach is the d-orbital energy level of transition metal alloying elements, Md, which is determined theoretically from the molecular orbital calculations of alloyed clusters. Also, the Md number accounts for the electronegativity and the atomic radius of the various alloying elements. This NEW PHACOMP with the Md parameter has been applied successfully to predicting the occurrence of TCP phases in various multi-component superalloys. It has not only been used for alloy design and development, but also for the prediction of solidification microstructures in weld metals. Recently, the calculations have been further extended to alloying elements such as Ru and Ir.

3:35 PM Invited

Design of Superalloys: *Hiroshi Harada*[1]; [1]National Research Institute for Metals, High Temp. Matls. 21 Proj., 1-2-1 Sengen, Tsukuba Science City, Ibaraki-ken 305-0047 Japan

Ni-base single crystal superalloys with higher temperature capabilities have been successfully designed by using our alloy design computer program established on a database by means of regression analysis. The microstructural parameters including gamma/gamma' equilibrium compositions, volume fractions, lattice parameters and misfit values, etc, are calculated and used for estimation of creep strengths, etc. This alloy design program is now being combined with gas turbine simulation programs to make a virtual gas turbine, in which new superalloys can be virtually tested in terms of creep properties. A part of prototype virtual gas turbine is to be open for public to use on our WWW site.

4:05 PM Break

4:20 PM

The Microstructural Instability of Single Crystal Superalloys: Mechanisms and Modelling: *Catherine M.F. Rae*[1]; Roger C. Reed[1]; [1]University of Cambridge/Rolls-Royce University Technology Centre, Dept. of Matls. Sci., Pembroke St., Cambridge CB2 3QZ UK

Most modern single crystal alloys are thermodynamically unstable with respect to the formation of topologically close packed (TCP) phases. In the alloy design process it is becoming increasingly important to acknowledge this, particularly for land-based turbine applications. Ideally, one would like a predictive capability for the onset of TCP formation. Traditionally, procedures such as PHACOMP have been used but nowadays the CALPHAD-type method is being employed. In this presentation, the authors' experiences will be discussed. The extent to which it is possible to use thermodynamics modelling alone to predict the stability of alloys will be described together with the role of the sigma phase in the accelerated nucleation of other TCP phases.

4:40 PM Invited

Reliability of Thermodynamic Data in Predicting Phase Stability of Superalloys: *J.-C. Zhao*[1]; [1]General Electric Company, Corp. R&D, PO Box 8, K1-MB231, Schenectady, NY 12301 USA

The time-temperature-transformation (TTT) diagrams of several superalloys such as Nimonic 263 and Hastelloy X are constructed based on careful experimental investigations. Since the upper arm of a C-curve should be asymptotic to the equilibrium solvus of the corresponding phase, the TTT diagrams (especially the very long-term exposure data) provide equilibrium information for the complex multicomponent superalloys. The experimental solvus of various phases (the gamma prime phase, the topologically close packed (TCP) phases, and carbides) is used to check the thermodynamic calculation results. The comparison shows that the existing thermodynamic data generally do a very good job in predicting the stability of the gamma prime phase and the carbides, but a less satisfactory job for the TCP phases. A method is suggested to provide critical experimental data needed to improve the thermodynamic parameters of the TCP phases. In addition, an example is presented to illustrate the importance of long-term exposure data in judging the real equilibrium phases of multicomponent superalloys.

5:00 PM Invited

Development of Computational Tools for Microstructural Engineering of Ni-Based Superalloys by Means of Phase Field Method: Chen Shen[1]; Jeff P. Simmons[2]; *Yunzhi Wang*[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboratory, AFRL/MLLM, Wright-Patterson AFB, Dayton, OH 45433 USA

To develop computational design tools for microstructural evolution in Ni-based superalloys, the phase field method could offer several advantages over other existing methods. For example, it is able to simulate realistic microstructures and their evolution under conditions of high volume fraction, elastically interacting particles, and concurrent nucleation, growth and coarsening of multiple ordered domains in a single consistent method. From the simulation, detailed information can be obtained on sizes and shapes of individual particles and domains and their spatial arrangement at each time moment during the microstructural evolution. However, the adoption of the phase field method in practical applications has been slow because the current phase field modeling is qualitative in nature and is limited to isothermal conditions. In addition, the method is computationally intensive and most of the work was done on supercomputers. In this presentation, new development intended to take the phase field method beyond the research stage towards practical applications will be discussed. This includes extension of the phase field method to non-isothermal conditions and development of constitutive relations from quantitative analyses of the simulation results. The latter could be easily incorporated into PC-based computational design tools for engineering applications. The general formulation of the method and basic parameters that must be determined externally will be discussed. The models developed will be validated against existing analytical and numerical models for simple systems and predictions for Ni-based superalloys with the effects of soft impingement, elastic interactions, particle coalescence will be compared with available experimental data.

Microstructure Modeling and Prediction During Thermomechanical Processing - I

Sponsored by: Structural Materials Division, Materials Processing & Manufacturing Division, Shaping and Forming Committee, Titanium Committee
Program Organizers: Raghavan Srinivasan, Wright State University, Department of Mechanical & Materials Engineering, Dayton, OH 45435 USA; Armand J. Beaudoin, University of Illinois at Urbana–Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Steve P. Fox, Timet Corporation, Timet Henderson Technical Laboratory, Henderson, NV 89009 USA; Zhe Jin, Reynolds Metals Company, Metallurgy Department, Chester, VA 23836-3122 USA; S. L. Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Monday PM — Room: 208
November 5, 2001 — Location: Indiana Convention Center

Session Chairs: Raghavan Srinivasan, Wright State University, Dept. of Mechl. & Matls. Eng., Dayton, OH 45435 USA; S. Lee Semiatin, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA

2:00 PM Introduction

2:05 PM
Accelerated Insertion of Materials for Aircraft Engine Superalloy Applications: *Daniel G. Backman*[1]; Xavier Pierron[1]; Daniel Y. Wei[1]; Michael F. Henry[2]; Canan U. Hardwicke[2]; [1]GE Aircraft Engines, Eng. Dept., 1000 Western Ave., MZ 36807, Lynn, MA 01910 USA; [2]GE Corporate Research & Development, Physl. Metall., 1 River Rd., K1-MB229, Schenectady, NY 12301 USA

Material and process development for aircraft engines is a long and costly undertaking that often extends upward to fifteen years–imposing a significant barrier to the insertion and exploitation of new materials. While product design has benefited from the application of computer-aided methods to increase design productivity, the industrial materials community has only recently begun to embrace computer modeling and simulation. This paper will provide an overview of an approach for accelerating the insertion of materials (AIM). The AIM approach involves the integration of materials models, database, and analysis tools that together inform design engineers about material performance, producibility, and cost. Acceleration from such an integrated system depends on models with sufficient fidelity and robustness to confidently predict microstructure and mechanical properties. The role of microstructural modeling and current progress in predicting the grain and precipitate microstructures of nickel-base superalloys will be presented.

2:35 PM
Micro Forming Analysis by Finite Element Method using Strain Gradient Crystal Plasticity: *Heung Kyu Kim*[1]; Soo Ik Oh[1]; [1]Seoul National University, Sch. of Mechl. & Aeros. Eng., Micro Forming Lab., San 56-1, Shilim Dong, Kwanak Gu, Seoul 151-742 Korea

Rate-dependent rigid plastic model of face-centered cubic crystalline material has been formulated within the framework of 2-dimensional finite element method to predict the forming behaviors of small scale polycrystalline aggregates. The formulation is based on the single crystal model and each grain of polycrystalline aggregates is divided into finite elements to capture the deformation behavior at the sub-grain level. However, from the plane strain compression test, it is observed that the deformation pattern changes as the mesh refines for some initial grain orientations when the isoparametric 4-node linear rectangular element is used. As a tool to get a consistent prediction as the mesh refines with such simple element, the concept of strain gradient has been introduced into the variational functional based on the rigid-plastic analogy. The developed formulation introduces an additional boundary energy term to compensate the strain gradient energy jump across the linear element boundary. It is verified by numerical tests that a mesh-independent prediction can be obtained by the suggested strain gradient formulation. It is also shown that the specimen size effect observed in micro forming processes can be predicted by the formulation.

3:00 PM
Evolution of Lattice Orientations and Misorientations in Titanium Alloys: *Paul Richard Dawson*[1]; Nathan Rhodes Barton[1]; [1]Cornell University, Mechl. & Aeros. Eng., 196 Rhodes Hall, Ithaca, NY 14853 USA

The development of accurate predictive models for alloys titanium has been hindered by the wide variety of microstructural forms that these materials can take and by the interactions between the hexagonal and cubic phases, given that the phases can differ substantially in strength and that the hexagonal phase displays strong anisotropy. We will discuss our efforts to model single and two-phase titanium systems using finite element formulations in which individual crystals are discretized with elements. Polycrystalline aggregates constructed using the discretized crystals are subjected to deformation histories corresponding to idealized forming events. The simulations deliver predictions of the lattice reorientations over the course of the deformation history. We will focus on the influence of the grain interactions on the heterogeneity of deformation within the crystals as well as the impact this has on the overall evolution of texture. Of interest is the influence of the grain morphology on the lattice reorientations, which will be discussed in terms of equiaxed versus lamellar microstructures. Results show a relationship between spread of individual grains in orientation space, the anisotropy of the single crystal response, and the volume fraction of each phase.

3:25 PM
Analysis of Primary Hot Working of Ti-6Al-4V using Orientation Imaging: *Thomas R. Bieler*[1]; S. L. Semiatin[2]; [1]Michigan State University, Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824-1226 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433-7817 USA

Orientation Imaging provides a combination of microstructural and crystallographic information that is valuable for examining coupled changes in texture and microstructure. This capability has been used to examine how large grain colony microstructures in Ti-6Al-4V are altered by primary hot working to develop a fine-grain microstructure that is technologically desirable for secondary hot working processes such as rolling and superplastic forming. With this technique, the reasons for retaining large clusters of grains with similar grain orientation, cavity nucleation and growth, and heterogeneous deformation phenomena such as shear banding can be elucidated. The impact of this new understanding on developing physically based constitutive models and new forming strategies that improve the homogeneity of the microstructure will be described.

3:50 PM Break

4:05 PM
Mesoscale Simulations of Microstructure and Texture Evolution during Deformation of Columnar Grains: *Gorti B. Sarma*[1]; Bala Radhakrishnan[1]; Thomas Zacharia[1]; [1]Oak Ridge National Laboratory, Comp. Sci. & Math. Div., PO Box 2008, MS-6359, Oak Ridge, TN 37831-6359 USA

In recent years, microstructure evolution in metals during deformation processing has been modeled at the mesoscale by combining the finite element method to discretize the individual grains with crystal plasticity to provide the constitutive relations. This approach allows the simulations to capture the heterogeneous nature of grain deformations due to interactions with neighboring grains. The application of this approach to study the deformations of columnar grains from solidification microstructures is described. The microstructures are deformed in simple compression, assuming the easy growth direction of the columnar grains to be parallel in one case, and perpendicular in another case, to the compression direction. These deformations are similar to those experienced by different regions of a large billet obtained by solidification when subjected to upsetting. The differences in the initial microstructures relative to the loading axis on the resulting changes in grain shape and orientation are investigated.

4:30 PM
Mesoscale Modeling of Abnormal Subgrain Growth: *Bala Radhakrishnan*[1]; Gorti B. Sarma[1]; Thomas Zacharia[1]; A. Kazaryan[2]; Yunzhi Wang[2]; [1]Oak Ridge National Laboratory, Comp. Sci. & Math., Bldg. 6025,

MS 6359, Oak Ridge, TN 37831 USA; [2]Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Abnormal subgrain growth plays an important role in the thermally activated evolution of deformation substructures. The origin of abnormal subgrain growth is the heterogeneity of deformation at the microstructural lengthscale that gives rise to non-random variability in the energies and mobilities of subgrain boundaries in the structure. The conditions under which abnormal subgrain growth initiates and the maximum size to which the abnormal subgrain will grow under given conditions can be obtained through an analytical approach. However, mesoscale simulations of abnormal subgrain growth under identical conditions using the Potts model do not reproduce the analytical results. The reasons for the discrepancies are discussed. Mesoscale simulation results obtained by the Potts model are also compared with those obtained by the Phase Field model in order to determine whether such differences are due to topological effects or due to inherent limitations in the mesoscale models.

4:55 PM

Realistic Simulation of Microstructure Evolution during Recovery: *Claire Maurice*[1]; Hasso Weiland[2]; [1]Ecole des Mines de Saint-Etienne, CNRS PECM Lab., SMS Ctr., 158 cours Fauriel, Saint-Etienne 42100 France; [2]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069 USA

It is commonly accepted that nucleation of recrystallization in cell forming materials takes place at various heterogeneities of deformed microstructures by abnormal growth of particular subgrains. A 3D-network simulation is used to examine the conditions required for nucleation in deformed microstructures. The spatial positions of interfaces, such as grain or subgrain boundaries, are tracked by means of a limited number of vertices. The interfacial energy contained in the sub-boundaries provides the driving force for their motion. Equations of motion for the vertices are derived from an energy balance principle between the free energy decrease due to boundary area reduction and the energy dissipated by the "viscous" motion of interfaces. Such a model adequately reproduces 3D normal grain growth with isotropic boundary properties. Here, anisotropic grain boundary properties are used to investigate the annealing behaviour of deformation substructures, characterized by the spatial arrangement of subgrains together with their crystallographic orientation.

Modeling the Performance of Engineering Structural Materials -I: Deformation Behavior

Sponsored by: Structural Materials Division, Structural Materials Committee
Program Organizers: Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA

Monday PM — Room: 140
November 5, 2001 — Location: Indiana Convention Center

Session Chairs: T. R. McNelley, US Naval Postgraduate School, Dept. of Mechl. Eng., Monterey, CA 93943-5145 USA; W. C. Johnson, University of Virginia, Matls. Sci. & Eng. Dept., Charlottesville, VA 22904-4745 USA

2:00 PM Invited

Numerical Modeling of Solute-Drag Creep: *Eric M. Taleff*[1]; Sean J. Yoon[1]; [1]The University of Texas at Austin, Matls. Sci. & Eng., Dept. of Mechl. Eng., Austin, TX 78712-1063 USA

The solute-drag creep process controls deformation at elevated temperatures and relatively slow strain rates in a variety of technologically important materials. The solute-drag creep process gives rise to two beneficial behaviors which are taken advantage of in engineering alloys. First, solute-drag creep increases creep strength by making dislocation glide more difficult than dislocation climb. Second, solute-drag creep increases tensile ductility by increasing the strain-rate sensitivity to a value near 1/3. The original model of Weertman for solute-drag creep is one of the most successful of creep theories. Recent work has aimed at extending this basic framework to better predict the behavior of complex engineering alloys. The results of modeling efforts for ternary and more complex alloys are discussed. Model predictions are compared to a variety of experimental data.

2:30 PM Invited

Formulation of a Substructure-Based Creep Model: *S. V. Raj*[1]; [1]NASA Glenn Research Center, Matls. Div., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

A large body of experimental data accumulated in the power-law creep regime over the last several decades has revealed a remarkable similarity in the creep behavior of many materials varying from metals, intermetallics, ionic salts, ceramics to geological materials. Recent studies have revealed that several creep processes may be dominant depending on stress and temperature. Correspondingly, detailed microstructural studies on NaCl single crystals have revealed that several types of creep substructures other than equiaxed subgrains may also evolve during deformation, which cannot be rationalized in terms of conventional dislocation climb-controlled creep models. A universal approach to creep modeling is proposed in this paper with a long term objective of impacting engineering design. First, the microstructural observations are qualitatively rationalized in terms of a bifurcation diagram. Next, the use of nonlinear dislocation dynamics in creep modeling is advocated to rationalize the observed diversity in the creep substructures, and a simple technique for formulating these equations is discussed. A method is proposed for scaling-up the dislocation substructure evolution models by coupling them to a viscoplastic model through the volume fractions of the "hard" and "soft" phases. This coupling is shown to lead to the stress-subgrain size relationship in a simple and natural way.

3:00 PM Invited

Predicting the Mechanical Response of Cu-Based Micro- and Macrocomposites: *Martin Heilmaier*[1]; Wolfgang Grünberger[1]; Jeffery C. Gibeling[2]; Ludwig Schultz[1]; [1]IFW Dresden, Inst. for Metallic Matls., PO Box 27 00 16, Dresden D-01171 Germany; [2]University of California–Davis, Dept. of Cheml. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616-5294 USA

Cu-based composites are frequently used in applications where a combination of high electrical and thermal conductivity and high strength is required. Candidates are Cu-core/steel-jacket macrocomposites and oxide dispersion strengthened (ODS) Cu-Al2O3 microcomposites. The prediction of the mechanical response in such composites is exemplified with the following two case studies: (i) the evolution of strength as a function of drawing strain at room temperature in pearlitic steel-copper macrocomposites is described applying the model of Embury and Fisher[1] and (ii) a microstructurally-based model is proposed that rests on the essential idea that internal back stresses develop during creep deformation due to dislocation/dislocation and dislocation/dispersoid interaction[2]. This effective stress model is applied to explain constant structure as well as steady-state creep in ODS Cu microcomposites at elevated temperatures in a unified way. [1]J. D. Embury, R. M. Fisher, Acta Metall. 14 (1966), 147. [2]M. Heilmaier, J. C. Gibeling, submitted to Scripta Mater.

3:30 PM

Mechanical Properties of AZ91D Magnesium Alloy: *Asim Bag*[1]; W. Zhou[1]; [1]Det Norske Veritas Pte, Ltd., 10 Science Park Dr., DNV Technology Ctr., Singapore 118 224 China

Increasing demand for lighter components and light weight design have led to magnesium alloys being considered as possible engineering materials in the last few years. In particular, magnesium matrix composites are attracting a lot of attention because the addition of a reinforcing phase, such as ceramic particles or fibers may produce a remarkable improvement in the property profile. Magnesium silicon carbide composite has found extensive use in automotive components such as pistons and wheels and aerospace components, which are subjected to a cyclic loading. To the authors' knowledge, there is no detailed report on the tensile fracture and fatigue behavior in the open literature. Therefore the present investigation examines the tensile and fatigue behavior of AZ91D alloy and its composite with 10% SiC in three different microstructural states to understand how the matrix and ceramic particle interactions influence the tensile and fatigue crack growth and in turn scar threshold behavior.

3:55 PM Break

4:15 PM Invited

Computer Modeling and Validation of Ballistic and Hypervelocity Impact and Penetration Phenomena: Deformation Microstructures, Mechanical Performance, and Geometrical Issues: *L. E. Murr*[1]; [1]University of Texas at El Paso, Metlgcl. & Matls. Eng., 500 W. University Ave., M-201, El Paso, TX 79968-0520 USA

This study compares a wide range of crater and penetration channel phenomena involving soda-lime glass, stainless steel, and tungsten carbide projectiles impacting copper and aluminum targets at velocities ranging from 0.5 to 5 km/s. Dense WC projectiles create anomalously elongated craters in aluminum at impact velocities up to 2 k/s which do not allow effective projectile fragmentation/ejection. A comparison of elongated craters and tungsten-alloy rod penetration channel microstructures in copper targets shows a mixing of deformation twins and microbands except for hemispherical craters characteristic of spherical shock phenomena where only microbands occur. Dynamic recrystallization at the crater of penetration channel walls facilitates solid-state material flow, and this microstructural feature is modeled in computer simulations and extrapolations beyond the laboratory test environment. Microhardness maps surrounding the craters and penetration channels are compared with computer-generated, residual stress maps. These features, together with TEM microstructural observations and impact-related geometries, can provide dependable validations for extrapolated simulations into the hypervelocity regime. Supported by NASA MURED Grant NAG-9-1171, NASA Johnson Space Center Grant NAG-9-1100, and ARO Defense Augmentation Award (DAAG55-97-1-0238).

4:45 PM

Modeling the Ballistic Impact Performance of Two Aluminum Alloys: *Kathryn A. Dannemann*[1]; Charles E. Anderson[1]; [1]Southwest Research Institute, Mechl. & Matls. Eng. Dept., PO Drawer 28510, San Antonio, TX 78228-0510 USA

Thin plates (4.75 mm) of two aluminum alloys, 6061-T6 and 7075-T6, were impacted with a Sierra 165 lead-filled bullet over a range of velocities. The impact velocities ranged from approximately 260 to 370 m/s, with the higher velocities approaching the ballistic limit of the aluminum plates. The 7075-T6 aluminum plates exhibited less deformation than the 6061-T6 plates at the same impact velocity. The in-plane back surface strains were determined by post-test analysis of a grid pattern, placed on the non-impacted surface of the plates prior to testing. The deformation behavior of the plates was further investigated using the CTH hydrocode. CTH is an Eulerian code used to model the performance of materials at high strain rates. The modeling results, in conjunction with microstructural evaluation of the damage, are used to explain the observed difference in deformation behavior of the impacted 6061-T6 and 7075-T6 plates.

5:10 PM

Modeling Large Strain, High Rate Deformation in Metals: *Donald Ritchie Lesuer*[1]; Greg J. Kay[1]; Mary M. LeBlanc[1]; [1]Lawrence Livermore National Laboratory, L-342, Livermore, CA 94551 USA

Many modeling problems require accurate representation of the high-rate, large-strain deformation response of metals. Examples include processing operations (such as metal cutting, rolling and forging) as well as the in-service performance of metals (such as ballistic penetration and perforation, munition performance and explosive fragmentation). In this paper, we examine the large strain deformation response of 6061-T6 and Ti-6Al-4V over a range in strain rates from 10-4 s-1 to over 104 s-1. The results have been used to critically evaluate material models used for simulations involving large strains and high deformation rates, including the Johnson-Cook material model. Two new models that address the shortcomings of the Johnson-Cook model were then developed and evaluated. One of the models is derived from the rate equations that represent deformation mechanisms active during moderate and high rate loading while the other model accounts for the influence of void formation on yield and flow behavior of a ductile metal (the Gurson model). The characteristics and predictive capabilities of these models are then reviewed. Work performed under the auspices of the US Department of Energy by the Lawrence Livermore National Laboratory under contract W-7405-ENG-48.

Polymer Plenary Session

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee, SPE Polymer Analysis Division

Program Organizers: George T. Gray III, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Linda Schadler, RPI, Materials Science & Engineering Department, Troy, NY 12180-3590 USA; Carl Schultheisz, National Institute of Standards and Technology, Polymers Division, Gaithersburg, MD 20899 USA

Monday PM — Room: 500 Ballroom
November 5, 2001 — Location: Indiana Convention Center

2:00 PM Keynote

A Current Overview of Clay-Polymer Nanocomposites: *Emmanuel P. Giannelis*[1]; [1]Cornell University, Dept. of Matls. Sci. & Eng., 326 Bard Hall, Ithaca, NY 14853-1501 USA

Work in clay-polymer nanocomposites has exploded over the last few years. The prospect of a new materials technology, which can function as low-cost alternative to high-performace composites for applications ranging from automotive to food packaging to microlectronics has become irresistible to researchers around the world. In this talk I will review our current efforts in nanocomposites and discuss how the nanostructure can influence dynamics and properties.

2:30 PM

New Paradigms for Low Dielectric Constant Films: *Karen K. Gleason*[1]; [1]Massachusetts Institute of Technology, Cheml. Eng., 77 Massachusetts Ave., 56-469, Cambridge, MA 02139 USA

As microelectronic feature sizes decrease to 100-nm and below, new paradigms are required for both interconnect and lithography. Novel low-k dielectric candidates being include both fluorocarbon and organosilicon films. Pulse plasma and hot filament chemical vapor deposition (CVD) have the ability to control the composition and properties of these two classes of materials. Fluorocarbon and siloxane polymers are also ideal resist candidates for 157-nm photolithography, as conventional photoresists are opaque at this wavelength. This motivates the idea of direct dielectric patterning process in which a low-k dielectric film is deposited, exposed, and developed using no sacrificial imaging layer. This technology would greatly simplify future device manufacture by reducing the number of steps involved in patterning. Additionally, in order to achieve high aspect ratio features, the development step uses supercritical CO2, offering environmental, safety and health advantages over conventional aqueous base development. Positive-tone contrast at 0.25 mm resolution has been demonstrated in fluorocarbon hot filament CVD films.

3:00 PM

A Comprehensive Nonlinear Viscoelastic Constitutive Model: Theory, Numerical Implementation and Experimental Validation: *James M. Caruthers*[1]; [1]Purdue University, Dept. of Cheml. Eng., W. Lafayette, IN, USA

A comprehensive nonlinear thermoviscoelastic constitutive model has been developed for amorphous polymers. The model is based upon the fundamental requirements for a continuum description of material behavior, including the conservation of mass, energy and linear and angular momentum; the second law of thermodynamics; objectivity; determinism; equipresence; and applicable material symmetries. The model has been formulated to include generalized deformation tensors, and it incorporates a relaxation time that depends on the thermodynamic state of the material. In collaboration with researchers at Sandia National Laboratories, the constitutive model has been implemented in a three-dimensional finite element code. A wide range of experiments with uniaxial and multiaxial deformations using single and multi-step thermal and/or mechanical histories have been compared to finite element simulations to validate the predictive capability of the constitutive model.

3:30 PM

Aging Measurements, Degradation Mechanisms, and Lifetime-Prediction in Elastomers: *Roger L. Clough*[1]; Kenneth Gillen[1]; Mathias Celina[1]; Johnathan Wise[1]; Michael Malone[1]; Todd Alam[1]; Roger Assink[1]; [1]Sandia National Laboratories, Organic Matls. Dept., MS 0888, Albuquerque, NM 87185 USA

We have studied polymer degradation for two decades, focusing on understanding aging mechanisms, and utilizing insights obtained, to develop better lifetime prediction methods. Aging simulations often predict trends and rates dramatically at odds with results from actual applications. Examples include: 1) “straight-line” extrapolations which greatly overestimate lifetimes; 2) an elastomer which becomes hard and brittle in short-term tests, but stretchable and weak under lower-level aging conditions; 3) a polyolefin which exhibits more rapid degradation when irradiated at 22C than at 60C; 4) cases where rank-ordering of stability for a group of materials is different under varying degrees of acceleration. We have identified time-dependent effects involving phenomena such as oxygen diffusion and metastable reactive species in the oxidation chemistry, as causes of complex aging behaviors. Specialized analytical techniques which have been instrumental in understanding aging mechanisms will be reviewed, and a lifetime assessment technique which directly correlates accelerated aging results with measurements obtained under application conditions will be discussed. Sandia is a multiprogram laboratory operated by Sandia corporation, a Lockheed Martin company, for the United States Department of Energy under contract DE-AC04-94AL85000.

Polymer Poster Session

Powder Materials: Current Research & Industrial Practices: Powder Making, Processing and Consolidation

Sponsored by: Materials Processing & Manufacturing Division, Powder Materials Committee
Program Organizers: Fernand D.S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgical Engineering, Rapid City, SD 57701-3995 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Monday PM Room: 209
November 5, 2001 Location: Indiana Convention Center

Session Chairs: Enrique V. Barrera, Rice University, Mechl. Eng. & Matl. Sci. Dept., Houston, TX 77251 USA; Iver E. Anderson, Iowa State University, Ames Laboratory, Ames, IA 5001 USA

2:00 PM Opening Remarks and Introductions: Fernand Marquis

2:10 PM Keynote
Enhanced Understanding of High Pressure Gas Atomization Processing: *Iver E. Anderson*[1]; Robert L. Terpstra[1]; [1]Iowa State University, Ames Lab. (USDOE), 122 Metals Dvlp. Bldg., Ames, IA 50011-3020 USA
Gas atomization is the most versatile process for mass production of metal and alloy powders with a wide variety of melting temperatures and compositions. Many advanced technologies based on particulate materials of all types demand the availability of powders with a controlled size distribution, often finer than the limit of typical sieve classification, i.e., 400 mesh (dia.=38 µm). High pressure gas atomization (HPGA), a close coupled discrete jet atomization method, has been proven very effective for producing rapidly solidified metal and alloy powders with high yields in the ultrafine size class (dia.< 20 µm). Development of HPGA jets with convergent-divergent (C-D) rocket nozzle designs that have high total kinetic energy was performed to increase atomization efficiency and uniformity. Results of experiments with a low pressure C-D jet design (Pideal<3 MPa) for the most recent "HPGA-III" nozzle and with a modified melt pour tube configuration will be compared with earlier results and with new results on a low pressure version of the original HPGA-I nozzle. Visualization of the distinctions in these atomization processes was facilitated by high-speed cinematography with Cu vapor laser illumination and with gas-only Schlieren imaging. Different aspects of this work were supported by USDOE-BES (W-7405-Eng-82), USDOC (ITA81-02), and the ISU Research Foundation.

2:45 PM
Developments in Titanium Powder Metallurgy: *F. H. (Sam) Froes*[1]; V. S. Moxson[2]; [1]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]ADMA Products, Inc., 8180 Boyle Pkwy., Twinsburg, OH 44087 USA
Titanium powder metallurgy is reviewed dividing the technology into the categories of laserforming, powder injection molding, spraying, near net shapes (blended elemental and prealloyed) and far from equilibrium processing (rapid solidification, mechanical alloying and vapor deposition). A number of good quality, reasonably priced powders are now available and this should spur growth with powder injection molding leading the way.

3:15 PM
Analysis of Ultrasonic Gas Atomized 5083 Al using Phase Doppler Interferometer: *Yaojun Lin*[1]; Yizhang Zhou[1]; Enrique J. Lavernia[1]; [1]University of California–Irvine, Dept. of Cheml. & Biocheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA
Aluminum alloy 5083 Al is atomized into a dispersion of droplets using inert gas at various pressures. A Phase Doppler interferometer and a laser-based particle analyzer are utilized to measure in-flight droplet velocities in the spray-cone centerline during atomization and the droplet size distribution after atomization, respectively. A methodology is formulated to determine the atomization gas velocity field along the centerline by fitting the measured droplet velocities. The gas velocities at the atomization point, obtained through the above regression analysis, are used to determine the constants in Lubanskas equation for the mass median diameter, as well as in the equation for geometric standard deviation. The equations are then used to predict the powder size, and associated size distribution in the case of 5083 Al, under various processing parameters.

3:45 PM Break

4:00 PM
Pd Alloy Oxides and their Behavior under Thermal Exposure in Gaseous: *Heng Zhang*[1]; Jack Gromek[1]; Matthew Augustine[1]; Gayanath Fernando[1]; Marwan Rasamny[2]; Samuel R. Boorse[3]; *Harris Marcus*[1]; [1]University of Connecticut, Inst. of Matls. Sci., Storrs, CT 06269-3136 USA; [2]Delaware State University, Dept. of Comp. & Info. Sci., Dover, DE 19901 USA; [3]Precision Combustion, Inc., North Haven, CT 06473 USA
Novel Pd-transition metal oxides have been synthesized using chemical processing. The composition, structure and surface features have been investigated using X-ray diffraction, SEM, EDS, TEM, AES and XPS. The phase stability, thermal behavior and catalytic properties under methane atmosphere have been examined by using thermogravimetric analysis, gas environment high temperature X-ray (GEHTX) diffraction in-situ experiments and gas phase mass spectroscopy. The study indicated that the Pd-based oxide alloy powders have nano-meter structure. The alloying of transition metals can significantly affect the thermal stability and heterogeneous characteristic of the palladium oxide during the thermal cycling. The surface feature and its effect on the catalytic performance of these systems are discussed.

4:30 PM
Accuracy of Fractal Dimension Measured from Single Particle's Contour Image: *Teruyuki Satou*[1]; *Yoshihiko Nomura*[1]; [1]National Institute of Advanced Industrial Science and Technology, Indl. Measurement Standards Sec., 1-1-4,Umezono, Tsukuba, Ibaraki 305-8563 Japan
The fractal dimension of single particle shape is expected to be an effective measure for surface roughness of the particle. The reason, why the fractal dimensions are not generally used yet, is that there is not clarified the reliability of accuracy. To improve the reliability of accuracy, the relationships between the contour types and the fractal dimensions were, first, examined with respect to the sharpness of image resolution: the fractal dimensions are measured by the box-counting method. As a result, some noteworthy facts are known to achieve highly accurate fractal dimension values. In addition to that, it was found out that the focus of the field images also influences the fractal dimension values markedly. Considering the characteristics with focus, the authors propose a method to reduce the focus caused error.

5:00 PM
Methodology for the Development of a Multicomponent Plasma Jet: *Medgar Gelenidze*[1]; Fernand D.S. Marquis[2]; Akaki B. Peikrishvili[1]; Nikoloz Chikhradze[1]; [1]Institute of Mining Mechanics, Tbilisi 380086 Georgia; [2]South Dakota School of Mines and Technology, Dept. of Matls. & Metlgcl. Eng., Col. of Matls. Sci. & Eng., Rapid City, SD 57701 USA
The application of high temperatures and large heat capacity permit the use the plasma jet for treating refractory and ceramic materials as well as those with a high thermal conductance. For the complete realization of these possibilities it is advisable in many cases to obtain the multicomponent plasma jet (manufacturing of new composite materials, deposition of coatings upon various articles by welding and sputtering, formation of new phases etc.) However the development of the multicomponent plasma jet is linked with some scientific and technical challenges. Some of these challenges are described in presentations developed by some authors of the methodology for obtaining the multicomponent plasma jet (USSR patents: 434893, 1122204, 539413, 560396). These technical inovations make possible the introduction of the plasmatron and to obtain a flame jet from different gases, liquid and powder materials. This is developed as combination of two to three components while the formation of plasma jet is stable and at the same time its heat power is increased. For the undisrupted and stable formation of the plasma jet it is recommended to build the plasmatron's power source developed by authors from regulated inductance-capacitance converters of current source (USA patent: 4378522).

Affordable Metal-Matrix Composites for High Performance Applications - II: Processing of MMCs - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee

Program Organizers: Awadh B. Pandey, Pratt & Whitney, Liquid Space Propulsion, West Palm Beach, FL 33410-9600 USA; Kevin L. Kendig, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Watson, Pratt & Whitney, East Hartford, CT 06108 USA

Tuesday AM Room: 139
November 6, 2001 Location: Indiana Convention Center

Session Chair: Kevin L. Kendig, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA

8:30 AM Invited

Spray Formed Al MMCs via In-Situ Reactions: Yaojun Lin[1]; Yizhang Zhou[1]; *Enrique J. Lavernia*[1]; [1]University of California–Irvine, Dept. of Cheml. & Biocheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

In order to achieve grain sizes that are smaller than 10m, as required for super-plasticity, in-situ oxidation reactions are incorporated into conventional spray forming processing to synthesize metal matrix composites with fine dispersoids. In the present work, 5083 Al/Al2O3 are synthesized via in-situ oxidation reactions between the atomized droplets and atomization N2-O2 gas mixture containing 5-35 vol.% O2. The volume fraction, size and distribution of oxides dispersoids in as-sprayed material are investigated by experimental (chemical analysis, TEM observation and SAD analysis) and simulation approaches. The following results are obtained: (1) Al2O3 is the only oxide present, (2) the volume fraction of Al2O3 is approximately 0.05-0.2 vol.%, (3) the size of Al2O3 dispersoids is approximately 20-50 nm; (4) most of the Al2O3 is located at the grain boundaries. On the basis of the grain size measurement from SEM micrographs, the influence of Al2O3 dispersoids on grain growth of as-sprayed material during the solid phase cooling stage and during annealing stage is investigated in detail. The grain growth exponent n and grain growth activation energy Q in the Arrhenius type of empirical equation are determined. Finally, the tensile strength and elongation behavior of the extruded materials is reported.

9:00 AM

Fabrication and Evaluation of Carbon Fiber Reinforced Aluminum by Thermal Management of Fibers: *Pradeep K. Rohatgi*[1]; Gaurang Narasimhan[1]; Atef Daoud[2]; R. S. Amano[3]; Purnendu Shukla[1]; Vindhya Tiwari[1]; Kevin Lee[3]; [1]University of Wisconsin–Milwaukee, Matls. Eng., PO Box 784, EMS 574 CEAS, Milwaukee, WI 53201 USA; [2]Central Metallurgical Research & Development Institute, Helwan, Cairo, Egypt; [3]University of Wisconsin–Milwaukee, Mechl. Eng., PO Box 784, Milwaukee, WI 53201 USA

Attempts have been made to create novel microstructures at the interface between the matrix and the fibers by thermal management of the fibers. In the present research the ends of the carbon fiber extend outside the matrix envelope and are placed in low temperature environments during the solidification of the composite to enhance the cooling rate of the liquid metal. Limited work has been done on modeling, including the observation of the solidification microstructures around graphite rods, which were cooled. Numerical methods have also been used to compute the rate of solidification of liquid aluminum around high conductivity fibers using a finite volume two-dimensional axi-symmetrical model. The extraction of heat through the high-conductivity carbon fibers changes the solidification pattern. Selected composites containing nickel coated carbon fibers in 2014 Aluminum alloy were fabricated by liquid metal infiltration. Solidification microstructures observed around the fibers when the ends of the fibers were extending outside the matrix envelope to provide cooling will be reported.

9:20 AM

Laser Deposition of In Situ Ti-TiB Composites: *Rajarshi Banerjee*[1]; Peter C. Collins[1]; Craig Brice[2]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Lockheed Martin, Dallas, TX, USA

Due to their wide applicability, there is considerable interest in the development of metal-matrix composites consisting of hard precipitates, such as transition-metal borides, dispersed in a metallic/alloy matrix. One such system that has generated considerable interest in recent years is the titanium boride in titanium alloy matrix system. Despite the development of a variety of different processing routes for these composites, including some in situ ones, there are relatively few technologies capable of processing a fully dense, near-net shape component. A recent advancement in the field of near-net shape manufacturing techniques is laser engineered net-shaping (LENS) which falls in the class of direct laser deposition processes from powder feedstock. This paper will discuss the direct laser deposition of an in situ Ti-TiB composite using the LENS technology from a blend of elemental titanium and boron powders. A detailed characterization of the composite using transmission electron microscopy (TEM) will be presented. In addition to the significantly large savings in terms of raw material costs, the use of elemental powders for direct laser deposition allows for the exploitation of the thermodynamic enthalpy of mixing of these powders. Since the mixing of titanium and boron powders is a highly exothermic process, the thermodynamic enthalpy of mixing is expected to play a significant role in the microstructural development process of the composite during solidification and will be discussed in this paper.

9:40 AM Invited

A Neutron Diffraction Study of Phase Stress Evolution in an Al-7093/SiC Particulate MMC: *Partha Rangaswamy*[1]; Hahn Choo[1]; Mark A.M. Bourke[1]; Bhaskar S. Majumdar[2]; [1]Los Alamos National Laboratory, MST-8, Lujan Ctr., Los Alamos, NM 87545 USA; [2]New Mexico Tech, Dept. of Matls. & Metall., Socorro, NM 87801 USA

A pulsed neutron diffraction source was used to monitor the evolution of elastic strains in the reinforcement and matrix phases of an Al-7093/SiC particulate metal matrix composite (MMC). The MMC had 15 volume percent particles, and was loaded in-situ in the diffractometer. Analysis of the data showed that the SiC contained both cubic and hexagonal polytypes, in contrast to either an alpha or beta SiC, as has generally been presumed in the literature. The proximity of the aluminum diffraction peaks with the two-polytype constituents of the SiC, made the evaluation of stresses more difficult. On the other hand, they point out important issues that must be resolved before stresses in constituent phases can be determined accurately using either X-ray or neutron diffraction. The neutron diffraction data were also used to extract strength statistics for the SiC particulates, since the MMC showed particle cracking as the predominant mode of damage as observed in a separate study. This is the first direct attempt to extract such strength statistics from neutron diffraction data, and initial results will be presented.

10:10 AM Break

10:25 AM Invited

Interfacial Aspects to Produce Particulate Reinforced Metal Matrix Composites: *George Kaptay*[1]; [1]University of Miskolc, Dept. of Physl. Chem., Egyetemvaros, Miskolc 3515 Hungary

The successful and affordable production of particulate reinforce metal matrix composites requires the understanding of interfacial phenomena taking place during the process. In this presentation the following three phenomena will be addressed: i. introduction of particles into liquid metals (technical goal: to ensure spontaneous introduction of particles without stirring), ii. interaction of particles at the surface and in the bulk of the liquid metal (technical goal: to avoid clustering of the particles), iii. interaction of particles with the approaching solidification front in one-component and multi-component liquid metals, including the effect of interface active components at the liquid metal/particle interface (technical goal: to ensure attractive surface forces between the growing crystal and the particles, or at least to ensure low critical solidification front velocities of the pushing/engulfment transition). Fortunately all the above mentioned three technical requirements are met better with increased adhesion between the liquid matrix and the particles. This is in accordance with the fact that the performance of MMCs is improved as the adhesion between the solidified matrix and the particles is increased. Possibilities and limitations to this requirement of high adhesion energy in liquid metal/ceramic systems will be discussed.

10:55 AM

Low-Cost, In Situ Synthesis of Heat Resistance Alloys Reinforced with Alumina Nanofilaments: *Pragati Kumar*[1]; Nahum Travitzky[1]; Ken H. Sandhage[2]; Nils Claussen[1]; [1]Technical University of Hamburg–Harburg, Adv. Cer. Grp., Denickestrasse 15, Hamburg 21073 Germany; [2]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Fe- and Ni- based heat resistant alloys (HRAs) are used in a number of high temperature applications. HRAs with ceramic reinforcements can exhibit enhanced creep and flexure strength. A novel approach for the fabrication of dense, alumina-nanofilament reinforced HRAs is demonstrated: the in-situ infiltration route to metal-matrix composites (isi-MMC). In this process, an intimately-mixed powder compact comprised of Al, reactive oxides and additional constituents of a metal matrix composite is prepared. This compact is then subjected to mild pressure and modest temperature so as to cause a thermite-type reaction resulting in rapid formation of a dense, oxide-reinforced metal matrix composite. Dense, alumina-nanofilament reinforced Fe- and Ni-based heat resistant alloys were fabricated by subjecting Fe2O3/Fe/Ni/Cr/Al and Cr2O3/CoO/Ni/Al containing powder compacts, respectively, to 900°C and 20 MPa pressure for < 5 min. Such composites had novel micro- and nanostructures along with superior mechanical properties. The processing route, microstructure and mechanical properties of the as-fabricated composites will be presented.

11:15 AM
Dynamic Simulation of the Movement of a Ceramic Particle in Front of a Solidifying Interface: *Akos Borsik*[1]; Katalin K. Kelemen[2]; George Kaptay[1]; [1]University of Miskolc, Dept. of Physl. Chem., Egyetemvaros, Miskolc 3515 Hungary; [2]University of Miskolc, Dept. of Mech., Egyetemvaros, Miskolc 3515 Hungary

The computer aided simulation of the movement of a ceramic particle in liquid metals has been performed, while an originally planar solid/liquid interface approaches the particle. Only the repulsive interfacial force and the drag force have been taken into account. Also, the effect of the particle on the local shape of the interface has been considered from the equality of the Laplace pressure and Derjaugin's disjoining pressure. It has been found that the particle accelerates gradually under the influence of the interfacial force, and reaches the steady state of pushing (without a minimum or maximum). The conditions of the critical interface velocity and critical separation have been established. The dependence of the critical velocity on different physical constants (size of the particle, interfacial energies, viscosity and density of the melt) has been established numerically. The numerical results are close to those obtained recently by a simplified analytical solution.

11:35 AM Invited
Tensile and Fatigue Properties of Permanent Mold Cast A359-SiCp Aluminum Alloys: *Pradeep Rohatgi*[1]; *Purnendu Shukla*[1]; David Weiss[2]; Atef Daoud[3]; [1]University of Wisconsin–Milwaukee, Matls. Eng., PO Box 784, EMS 574 CEAS, Milwaukee, WI 53201 USA; [2]Eck Industries–Wisconsin, Manitowoc, WI, USA; [3]Central Metallurgical Research, Helwan, Cairo, Egypt

The current investigation was undertaken to study the influence of foundry, alloy supplier, testing laboratory and % SiCp on mechanical properties of SiCp reinforced Al-matrix composites with the intent to develop standard handbook grade data to facilitate design in these materials. Ingots of 20% and 30% SiCp reinforced aluminum based alloy composites were remelted and permanent mold cast into tensile bars at three different foundries. All the bars were T6 heat treated at one location. Tensile properties were measured at three different laboratories. Samples from one foundry were also tested under strain controlled mode for their fatigue properties. The variations in tensile properties from different testing labs, for different % SiCp, and for different foundries were analyzed. The microstructure and fractographs of the fracture surface of tensile and fatigue tested bars were analyzed using optical stereo microscope and SEM-EDX and related to properties. Significant differences were observed in the tensile properties of the bars cast at different foundries and tested at different labs. The modulus of 30% SiC tensile bars was found to be greater than modulus of 20% SiCp, however the tensile strength of 20% SiCp bar was higher. Factors associated with low tensile properties included casting defects such as shrinkage porosity, gas porosity and oxide inclusions.

Applications of Computational Thermodynamics in Materials Processing - II

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee
Program Organizers: Qingyou Han, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; Ravi Vijayaraghavan, Ford Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Tuesday AM — Room: 111
November 6, 2001 — Location: Indiana Convention Center

Session Chairs: Sudarsanam Suresh Babu, Oak Ridge National Laboratory, Metals & Cer. Div., Oak Ridge, TN 37831-6096 USA; Florian Kongoli, Flogen Technologies, Metall., Montreal, QC H3S 2C3 Canada

8:30 AM
Applicability of Some Thermodynamic Models for Liquid Phases in Metallurgy and Materials Processing: *Florian Kongoli*[1]; Ian McBow[1]; [1]FLOGEN Technologies, Inc., Metall., 5757 Decelles Ave., Ste. 511, Montreal, Quebec H3S 2C3 Canada

Several attempts have been made to model the liquid phases pertaining to several chemical or metallurgical processes. Some models have been used in this regard with a certain degree of success. Each model is based on a particular formalism such as the sub-lattice, quasichemical and association formalisms or on simple polynomial expansions of the excess Gibbs energy. Due to the physical assumptions they employ as well as the mathematical formulations they use, they all have several strong and weak points. In this work the applicability of some models for liquid phases is critically evaluated in terms of their physical and chemical significance, their generality and simplicity and the number of mathematical parameters used. It is found that their application cannot be general but only restricted to particular processes.

8:55 AM
Thermodynamic Investigations of Yttriumsilicate Coatings: *Sigrid Wagner*[1]; Hans Jürgen Seifert[1]; Fritz Aldinger[1]; [1]Max-Planck-Institute, PML, INAM, Heisenbergstr. 5, Stuttgart 70569 Germany

Carbon fibre-reinforced ceramic matrix composites are promising materials for high-temperature structural applications. Coatings are required because of the oxidation of carbon. A common coating is SiC, which is oxidation-resistant due to the formation of a silica scale during oxidation. Problems arise due to the active oxidation of SiC at low oxygen partial pressures, as occurring during re-entry of aerospace vehicles. Therefore, the development of protective coatings based on a SiC bonding layer combined with an outer Yttriumsilicate layer is under investigation. Thermodynamic calculations show chemical compatibility of this multi-layer arrangement. Calculated vapor pressure diagrams indicate the possibility of plasma-spraying of Y-Silicates for production. Potential phase-diagrams calculated for different temperatures explain observed bubble formation at the interface Yttriumsilicate/SiC. This causes rapid degradation of the underlying ceramic. The exposure of the coating to oxygen has been simulated and results were experimentally verified.

9:20 AM
Optimization of 357 Aluminum Alloy for Semi-Solid Metal Casting: *Qingyou Han*[1]; Srinath Viswanathan[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831-6083 USA

Currently, established casting alloys such as aluminum A356 and 357 alloy are also used for semi-solid metal casting (SSM). Process inconsistencies arising from the use of these alloys are partly due to variations in the solid fraction due to temperature variations. Consequently, the process would be more robust if the alloys were tailored such that the solid fraction variation in the temperature range of operation were minimized. For this purpose, thermodynamic simulations have been carried out to determine the solid fraction of 357 alloy as a function of temperature and composition. The solid fraction variation for a given temperature variation was then calculated and optimized over the composition range of 357 alloy. A criteria function for determining the temperature at which the solid fraction variation is minimum is proposed. It is shown that computational thermodynamics can be successfully used for the development of processing friendly SSM alloys.

9:45 AM
Thermodynamics, Phase Equilibria and Crystallization Behavior of Al-Base Metallic Glasses: *Michael C. Gao*[1]; Robert E. Hackenberg[1]; Gary J. Shiflet[1]; [1]University of Virginia, Matls. Sci. & Eng., 116 Engineer's Way, Charlottesville, VA 22904 USA

The thermodynamics, solid phase equilibria and crystallization behavior of Al-Ni-Gd and Al-Fe-Gd metallic glasses were investigated in detail. Up to 30 alloy compositions in each system on the Al-rich corner were chosen for this study. The solid phase equilibria and the crystal structures of the ternary compounds were determined using X-ray diffraction, TEM, quantitative TEM, and high resolution TEM simulation. The solidus and liquidus temperatures of these alloys were measured using DTA. Using these data combined with other published data, the Al-rich corners of these systems were thermodynamically optimized using the CALPHAD approach. The driving forces for nucleation of crystalline phases were calculated. The crystallization behavior of these systems was studied using DSC, X-ray diffraction and TEM. The relationship between thermodynamics and crystallization behavior, glass forming ability and structural evolution during devitrication in Al-base metallic glasses will be discussed.

10:10 AM Break

10:20 AM
Modeling Microstructure Development in Welds using Computational Thermodynamics and Kinetic Models: *Sudarsanam S. Babu*[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., 1 Bethel Valley Rd., Bldg. 4508, MS 6096, Oak Ridge, TN 37831 USA

The weld microstructure evolutions in structural alloys are complex due to interactions of transient thermal cycles and phase stability. To achieve predictive modeling of weld microstructure and relate the same to the performance of welded structures, we need comprehensive thermodynamic and kinetic models. These models must be capable of predicting phase stability in multicomponent alloys, as well as, kinetics of the phase transformations. The talk will present recent results from application of such models to describe the solidification and solid-state transformation in Fe-Al-C-Mn and nickel base superalloys. In addition, the need for concurrent experimental characterization including high-resolution characterization tools and in-situ neutron and synchrotron diffraction tools will be highlighted.

10:45 AM
The Equilibria in the AlN-Al2O3-Y2O3 Ternary System-Thermodynamic and Neutron Diffraction: *M. Medraj*[1]; R. Hammond[2]; W. T. Thompson[3]; R. A.L. Drew[1]; [1]McGill University, Metlgcl. Eng. Dept., 3610 University St., M. H. Wong Bldg., Montreal, Canada; [2]National Research Centre, Neutron Prog. for Matls. Rsrch., Chalk River, Ontario, Canada; [3]Royal Military College, Kingston, Ontario, Canada

Aluminum nitride (AlN) is currently used in electronic packaging and engineering ceramics. It offers both higher thermal conductivity and superior electrical insulating properties compared with Al2O3. Yttria (Y2O3) is the best additive for AlN sintering, and it has been shown that AlN densifies by a liquid phase mechanism, where the surface oxide, Al2O3, reacts with the oxide additive, Y2O3, to form a Y-Al-O-N liquid that promotes particle rearrangement and densification. Construction of the phase relations in this multicomponent system is becoming essential for further development of the AlN. Binary diagrams of Al2O3-Y2O3, AlN-Al2O3, and AlN-Y2O3 were thermodynamically modeled. The obtained Gibbs free energies of components, stoichiometric phases and solution parameters were used for the calculation of isothermal sections and liquidus surface of AlN-Al2O3-Y2O3 system. The predicted ternary phase diagram was verified experimentally using in situ high temperature neutron diffractometry. The ternary phase diagram AlN-Al2O3-Y2O3 has been constructed for the first time in this work.

11:10 AM
Calculating Liquid Metal Transport Properties via Molecular Dynamics: *Frank Joseph Cherne*[1]; Michael I. Baskes[1]; Pierre A. Deymier[2]; [1]Los Alamos National Laboratory, MST-8, PO Box 1663, MS G755, Los Alamos, NM 87544 USA; [2]The University of Arizona, Dept. of Matls. Sci. & Eng., PO Box 210012, Tucson, AZ 85721-0021 USA

The advanced casting modeler requires accurate viscosity and diffusivity data of liquid metals and their alloys. The general scarcity of this data combined with the experimental difficulties in obtaining this data motivates this work. Utilizing the equilibrium and non-equilibrium molecular dynamics techniques, we calculate the diffusivity and viscosity of several technologically important metals and their alloys (i.e. aluminum, nickel, and aluminum-nickel alloys). We compare the transport properties and melting points obtained from both embedded atom method (EAM) and modified EAM (MEAM) potentials to the available experimental data. The MEAM potentials are shorter range than the EAM potentials, but include angular forces. The results from this work indicate that equilibrium and non-equilibrium molecular dynamics can provide reliable and accurate data required by the advanced casting modeler.

11:35 AM
Simulation of Microstructures and Mechanical Properties of a Cast Aluminum (319 Alloy) Engine Block: *Mei Li*[1]; *Ravi Vijayaraghavan*[1]; John E. Allison[1]; Xinyan Yan[2]; [1]Ford Motor Company, Scientific Rsrch. Lab., MD 3182/SRL, PO Box 2053, Dearborn, MI 48121-2053 USA; [2]University of Wisconsin–Madison, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53705 USA

Cast aluminum alloys, particularly 319-type, are increasingly being utilized by automotive industry for manufacturing engine blocks and cylinder heads to reduce vehicle weight and consequently increase fuel economy and reduce emissions. Predictions of microstructure and the corresponding mechanical properties are crucial for estimating in-service performance of these components. This talk describes the simulation of microstructure evolution in casting and subsequent heat treatment processes and yield strength after heat treatment in a 4.6L engine block. A micromodel is used to predict the fraction of as cast Al2Cu phase and secondary dendrite arm spacing (SDAS). A solution treatment model is developed to calculate the fraction of dissolved Al2Cu. This result is then used in conjunction with a precipitation model to predict amount of Al2Cu precipitated in the aging process and the corresponding yield strength. Finally, the predicted yield strength in a 4.6L engine block is compared with experimental data.

Friction Stir Welding and Processing - II: Microstructure and Properties - I

Sponsored by: Materials Processing and Manufacturing Division, Shaping and Forming Committee, Texture & Anisotropy Committee
Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; Shigeo Saimoto, Queens University, Ontario K1L 3NG Canada; S. L. Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Tuesday AM   Room: 115
November 6, 2001   Location: Indiana Convention Center

Session Chairs: Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; John A. Wagner, NASA Langley Research Center, Hampton, VA 23681-2199 USA

8:30 AM
Residual Stress Effects on Near-Threshold Fatigue Crack Growth in Friction Stir Welds: *Kumar V. Jata*[1]; Reji John[1]; [1]AFRL/MLLM, 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA

Near threshold fatigue crack growth in as-friction stir welded materials has been studied on a number of alloys as a function specimen geometry, size and stress ratio. Residual stresses were also measured on the specimens prior to testing. These studies show that, although friction stir welding process induces small residual stresses in the welds, they are large enough to affect the near-threshold fatigue crack growth behavior in the welds. This presentation will discuss the results of the experimental and analytical work performed to assess the influence of residual stresses on the effective crack driving force in the welds.

8:55 AM
Friction Stir Welding of D357 Castings and 2024 Wrought Plate: *R. J. Lederich*[1]; J. A. Baumann[1]; [1]Boeing, St Louis, MO, USA

Friction stir welding (FSW) was used to join castings-to-castings and castings-to-wrought aluminum alloys for potentially expanding the usage of economical castings in airframe and missile applications. Successfully joining of castings to plate enables more efficient design by locating wrought materials at high loading points to take advantage of their inherently superior bearing strength and damage tolerance. Such joining also permits other cost-effective fabrication sequences to be employed. However these two types of materials have different FSW characteristics because of their respective high temperature deformation characteristics. FSW butt joints were produced using ¼” thick D357-T6 castings and 2024-T3 plate. Joint morphologies and strengths were characterized by NDE, optical microscopy, and tensile testing. A post weld heat treatment study was performed to provide stability and improved properties.

9:20 AM
Texture Effects on Corrosion Behavior of Friction Stir Welded 7075 Aluminum: *David P. Field*[1]; Tracy W. Nelson[2]; David F. Bahr[1]; [1]Washington State University, Mechl. & Matls. Eng., PO Box 642920, Pullman, WA 99164-2920 USA; [2]Brigham Young University, Mechl. Eng., Provo, UT 84604 USA

Aluminum alloys are generally resistant to corrosion in industrial and marine environments, but suffer a modicum of pitting and galvanic corrosion depending upon environment, alloy and other factors. Al 7075 is susceptible to galvanic attack, particularly near precipitates of MgZn2 and other chemistries. This occurs preferentially on grain boundaries where the precipitates are most likely to form. Though a solid state joining technique, friction stir welding substantially alters the microstructure of the weld region thereby effecting a dramatic change in local physical and mechanical properties. Significant differences in texture and grain boundary structure exist in the weld nugget and surrounding region compared with those present in the original product. This work investigates local pitting and intergranular attack near friction stir welds in 7075 plate. The heterogeneity of corrosion behavior is correlated to local gradients in crystallographic texture and grain boundary structure.

9:45 AM Break

9:55 AM
Texture Analysis of Friction Stir Weld Ti-6Al-4V Alloy: *Gopal B. Viswanathan*[1]; Nikhil Karogal[1]; Hamish Fraser[1]; Mary Juhas[1]; [1]The Ohio State University, 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Friction stir welding of Ti-alloys is characterized by extreme plastic deformation in the solid state, high in the (alpha +beta) and/or beta phase field. In a preliminary study conducted on Ti-6Al-4V alloy, it was found the resultant microstructure in the weld region could have varying proportions of alpha and beta phase, depending on the temperature and the cooling rate from the weld temperatures. The morphology could vary from fully transformed microstructure consisting of (alpha+beta) colonies to bimodal microstructure consisting of equiaxed alpha and transformed beta grains. The mechanical properties of these weld region is strongly influenced not only by the microstructure, but also by the crystallographic texture of the hexagonal alpha and bcc beta phases. In this study, texture analysis of the weld, HAZ and the base material is undertaken. Texture measurements are obtained by pole figure analysis through traditional X-ray diffraction and by SEM electron back scattered diffraction (EBSD) techniques. The results will be presented and possible mechanisms for texture evolution in this alloy will be discussed.

10:20 AM
Static, Fatigue and Crack Growth Behavior of 7075-T6 and 2024-T3 Aluminum Alloys: Matt Tester[1]; *Steven G. Russell*[1]; Alan Cleaver[1]; Ed Nichols[1]; John Maynor[1]; [1]Vought Aircraft Industries, Inc., Struct. & Loads, PO Box 655907, MS 220-06, Dallas, TX 75265 USA

Vought has completed a program of static, fatigue crack initiation, and fatigue crack growth tests on 7075-T6 and 2024-T3 aluminum alloy specimens joined by the friction stir welding (FSW) process. The static tests evaluated the unnotched and open hole tension strength of FSW coupons, and the notch sensitivity to hole location at the weld centerline or in the heat affected zone (HAZ). The fatigue crack initiation and fatigue crack growth tests evaluated the fatigue performance of parent material and FSW coupons in laboratory and high humidity air, room temperature environments. This paper discusses in detail the results of these coupon tests and highlights the influence of the friction stir welding process on critical material properties. The paper concludes with a survey of additional testing required to supplement the existing database and elucidate trends observed in the tests conducted to date.

10:45 AM
On the Relationships Between Microstructural Variations and Properties of a Friction Stir Welded 6056 Alloy: *Anne Denquin*[1]; Gilles Lapasset[1]; Delphine Allehaux[2]; Marie-Helene Campagnac[2]; [1]ONERA, Dept. of Metallic Matls. & Proc., 29 Ave. de la Division Leclerc, Chatillon 92322 France; [2]EADS CCR, Matls. & Mfg. Proc. Dept., 12 bis rue Pasteur, BP 76, Suresnes 92152 France

The objective of this study is to provide a better knowledge on the relationships between microstructural variations and properties of a friction stir welded 6056 alloy, with a particular emphasis on the influence of initial microstructure. Two routes have been compared. In the first case, FSW has been performed on T4 condition and followed by a T78 post-weld aging treatment. In the second case, FSW has been performed directly on T78 condition. The microstructural study focuses on hardening as well as grain boundary precipitation and has been performed on the various zones which characterize friction stir welds (the weld nugget (WN), the heat and deformation affected zone (HDAZ) and the heat affected zone (HAZ)). For both cases, the formation of the mismatch zone is discussed in terms of microstructure and related to weld properties.

11:10 AM
Process-Structure-Property Study of Friction Stir Welding of Aluminum Alloys Employing Finite Element Analysis: *Woonsup Park*[1]; Michael J. O'Brien[1]; [1]The Aerospace Corporation, Space Matls. Lab., M2-242, PO Box 92957, Los Angeles, CA 90009-2957 USA

Microstructures and mechanical properties of FSW joints of 2219 and 2195 aluminum plates were investigated to understand the microstructural evolution under the thermomechanical condition of the weld process and its effects on the joint properties. Along with experimental characterization of the weld joints, a finite element analysis (FEA) model is being developed with the ultimate goal of using this model to optimize the process parameters minimizing the need for weld trial runs. Unlike previous FEA studies of FSW, our approach is to develop a fully coupled thermal-solid mechanics model of the process by considering the plastic flow of the material during stirring as well as temperature changes due to heat transfer. This model will account for important effects that have not been present in FEA models based on heat transfer approaches to date. Material flow pattern predicted by the model will be compared to the physical microstructure produced by FSW.

11:35 AM
Friction Stir Welding: A Study of Tool Wear Variation in Aluminum Alloy 6061 + 20% Al2O3: *Rafael A. Prado*[1]; *Lawrence E. Murr*[1]; *David J. Shindo*[1]; [1]University of Texas at El Paso, Metall. & Matls. Eng., M201, El Paso, TX 79968-0520 USA

The present report investigates and compares tool wear in the FSW of Al 6061-T6 and Al 6061-T6, with 20% Al2O3; a metal matrix composite (MMC). Differences in the final form of the worn tool were obtained for the two materials used in this research by photographing the tool after specific traverse lengths of butted plates. The rotational speeds of the tool pin were 500, 1000, 1500, 2000 and 2500 rpm for the MMC and 1000 rpm for the Al 6061 with a traverse speed of 60 mm/min. The cylindrical rotating tools were right-hand threaded and set at a positive angle of 1 degree in the welding direction (counter clockwise) at 60 mm/min. The weld tool did not exhibit any wear in the Al alloy 6061, however, the tool consumption geometry changed for the MMC. The data was plotted as an effective wear rate (% nib consumption/linear traverse in cm) verses tool rotation speed (rpm). The maximum wear rate was roughly 0.64 %/cm at 1000 rpm for the MMC. Research supported by GSA Grant PF-90-018, at a Murchison Endowed, and a Graduate Assistantship at UTEP.

12:00 PM
Friction Stir Welding Development at National Aeronautics and Space Administration-Marshall Space Flight Center (NASA-MSFC): *Biliyar N. Bhat*[1]; Robert W. Carter[1]; Robert J. Ding[1]; Kirby G. Lawless[1]; Arthur C. Nunes[1]; Carolyn K. Russell[1]; Sandeep R. Shah[1]; [1]NASA Marshall Space Flight Center, Eng. Direct., Huntsville, AL 35812 USA

This paper presents an overview of friction stir welding (FSW) process development at MSFC. Starting in 1994 with low melting aluminum alloys the FSW process is now being applied to higher temperature materials such as copper alloys and also to composite materials. Process modeling is done to better understand the process and to fine tune it for best results. Special tools are developed to enable welding variable thickness materials, including very thin and very thick materials. Special attention is paid to transferring the FSW technology to the shop floor. Issues such as scale-up, production, nondestructive examination and reliability are addressed.

Fundamentals of Solidification - I

Sponsored by: Materials Processing & Manufacturing Division, Solidification Committee
Program Organizers: John H. Perepezko, University of Wisconsin–Madison, Department of Materials Science & Engineering, Madison, WI 55706 USA; Christoph Beckermann, University of Iowa, Department of Mechanical Engineering, Iowa City, IA 52242-1527 USA; William J. Boettinger, National Institute of Standards & Technology, Gaithersburg, MD 20879 USA

Tuesday AM Room: White River Ballroom
November 6, 2001 Location: Indiana Convention Center

Session Chair: John H. Perepezko, University of Wisconsin, Dept. of Matls. Sci. & Eng., Madison, WI 55706 USA

8:30 AM Invited
The Role of Solute Atoms in Solidification: Atomistic Study of Solid/Liquid Interfaces: *Michael Baskes*[1]; Marius Stan[1]; [1]Los Alamos National Laboratory, Structl./Prop. Relations Grp., G755, Los Alamos, NM 87545 USA

A semi-empirical Lennard-Jones/Embedded Atom Method model is used to capture real materials behavior through the introduction of many-body forces. Using molecular dynamics (MD) calculations, the model is used to study the dependence of the solid/liquid interface velocity on temperature and composition. An asymmetry in velocity between solidification and melting is found. Slowing of the interface velocity by solutes during solidification is demonstrated. The results of MD calculations are used to get free energies of the solid and liquid phases, and thereby the phase diagrams. These calculations illustrate the consequences of differences in energy and size between the components on the phase diagrams. This work was supported by the US Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences.

9:10 AM Invited
Grain Refinement of Aluminium Alloys: *A. Lindsay Greer*[1]; [1]University of Cambridge, Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

Almost all aluminium is cast with added inoculants. The resulting grain refinement involves potent nucleation as well as growth restriction. Recent progress on understanding refinement will be reviewed. Transmission electron microscopy shows the nature of the nucleant particles and reasons for the poisoning of the refining action in the presence of some solutes. However, the microscopy is not useful in predicting grain size or other microstructural features. The uses of simple thermal modelling and of stochastic cellular-automaton modelling will be explored. Effective refinement requires inoculant particles of the correct crystallography and chemistry, but the modelling shows that their size distribution is also important in determining refining efficiency. These themes will be explored for both the major refiner types used for aluminium alloys, Al-Ti-B and Al-Ti-C. The latest advances in understanding permit the development of poison-resistant refiners and the quantitative prediction of grain size.

9:50 AM Invited
A Study of Micro-Segregation using a Novel Single-Pan Scanning Calorimeter: Hongbiao Dong[1]; Matthew Mo-yat Shin[1]; *John David Hunt*[1]; [1]University of Oxford, Dept. of Matls., Oxford, Oxfordshire OX1 3PH UK

A single-pan scanning calorimeter has been developed which eliminates the smearing of latent heat over a range of temperatures, which occurs in a conventional two pan DSC. The smearing is present when latent heat is evolved because the sample thermocouple does not measure the sample temperature and because the rate of temperature rise of the sample thermocouple is not the same as that of the sample. To reduce errors from latent evolution DSC manufacturers usually recommend using very small samples. In the new calorimeter, accurate enthalpy/temperature data can be obtained for large specimens. The calorimeter has been used to investigate microsegregation. In the past studies of microsegregation using calorimetery have been hampered by inaccurate enthalpy data and because it is often assumed that the latent heat is independent of composition. In the present work the enthalpy temperature data is compared with that predicted using a numerical based multicomponent microsegregation model. The enthalpy of each volume element is calculated as a function of temperature, composition and phase using thermodynamic data prediction software (MTDATA). The calorimeter and numerical microsegregation model will be described. The results using the calorimeter indicate that the segregation is between that predicted assuming no mixing in the solid and complete mixing in the solid. The amount of segregation agreed well with that calculated using the numerical model of micro-segregation. It is concluded that the new calorimeter, combined with thermodynamic data base software allows microsegregation to be accurately and quickly measured.

10:30 AM Break

10:40 AM Invited
Oscillatory Microstructures in Peritectic Systems: *Rohit K. Trivedi*[1]; Shan Liu[1]; Prantik Mazumder[2]; [1]Iowa State University, Matls. Sci. & Eng., 100 Wilhelm Hall, Ames, IA 50011 USA; [2]Corning, Inc., SP-TD-02-2, Sullivan Park, Corning, NY 14831 USA

Directional solidification of alloys in the two-phase region of peritectic systems can produce oscillatory microstructures in which the primary and the peritectic phases form a uniform or a particulate banded structure when the growth is controlled by diffusion only. When significant fluid flow effects are present, a variety of oscillatory structures are observed in which the primary phase forms an oscillatory branched structure that is surrounded by the continuous peritectic phase. Detailed experimental results in Sn-Cd and Pb-Bi system will be presented. These experimental results will be analyzed through the direct numerical simulation of the transport processes in Bridgman crystal growth systems. It will be shown that the complex interaction of vertical and lateral gradients of temperature and concentration leads to a range of complex spatio-temporal dynamics in the melt. The coupling of fluid flow dynamics and mass transfer with the phase change predicts spatial patterns observed experimentally.

11:20 AM Invited
Microstructure Map for Peritectic Fe-Ni Alloys: *Stephane Dobler*[1]; Alain S. Karma[2]; Wilfried Kurz[1]; [1]Swiss Federal Institute of Technology–Lausanne, EPFL-CTML, CP 110, Lausanne CH-1015 Switzerland; [2]Northeastern University, Col. of Arts & Scis., Dept. of Phys. & CIRCS, Boston, MA 02115 USA

Peritectic alloys are omnipresent, technically important and show a great variety of microstructures. In an extensive experimental study of the Fe-Ni system, the complete set of microstructures for conditions of directional solidification has been determined. Banding and island banding is obtained over the whole composition range of the two solid phases (δ-Fe and γ-Fe), whereas coupled growth is favored by compositions close to the peritectic g-phase for conditions of δ-plane front growth. Below the critical G/V value for morphological stability of the δ phase, cellular coupled growth is observed, though in some conditions isothermal coupled fibrous growth also appears. At higher solidification velocities (lower G/V) the classical peritectic structure of δ dendrites surrounded by γ precipitates is found. The results of recent theoretical models of peritectic two-phase growth will be discussed in the light of the experimental findings.

General Abstracts: High Temperature Alloys - II

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Tuesday AM Room: 105
November 6, 2001 Location: Indiana Convention Center

Session Chair: Patrice E. A. Turchi, Lawrence Livermore National Laboratory, Matls. Sci. & Tech. Div. (L-353), Livermore, CA 94551 USA

8:30 AM
Synthesis of Nickel Aluminide Foams by Pack Aluminization of Nickel Foams: *Andrea Maria Hodge*[1]; David C. Dunand[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Nickel-aluminide foams were synthesized from unalloyed nickel foams by using a two-step, high-activity pack-aluminizing process at 1273 K. After processing, the nickel aluminide foams exhibited the original structure of the original nickel foams (open-cells with hollows struts and low density). Single-phase NiAl foams, with average composition within 1wt.% of stoichiometry and with 92% open porosity, were produced by first selecting the appropriate aluminizing time, and then annealing to homogenize the structure. Foams of average Ni_3Al composition were produced by the same method, but multiple intermetallic phases remained due to large variations in strut thickness and thus local composition. Nickel wires and tubes were also aluminized at 1273 K and homogenized for different times to further investigate the aluminizing kinetics and the creation of Kirkendall pores. For aluminization depths up to about 100 microns, Kirkendall pores can be avoided, leading to pore-free struts in the foam.

8:50 AM
Numerical Model of Superalloy Gatorized Waspaloy in the Thermodynamic Processing: Hu Jianping[1]; [1]CISDI, Rolling Dept., No.1 Shuanggang Rd., Yuzhong Qu, Chongqing 400013 China

Simulation experiment of the isothermal forging process and hammer forging process of Superalloy Gatorized Waspaloy in industry was introduced in this article in details. The effect of hot-deformation parameters on flow behavior and microstructure changes of Superalloy Gatorized Waspaloy during this two processing was analyzed. Then constitutive equations reflecting the flow behavior were set up and microstructure models predicting the thermodynamic processing were established in terms of dynamic recrystallization and grain growth. Three steps of experiment were conducted during developing the models: (1) cylindrical specimens were compressed in MTS Testing Machine and in High-speed Machine respectively. (2) flow behavior, dynamic recrystallization, and grain growth were discussed. (3) constitutive equations, dynamic recrystallization models and grain growth model were developed. The agreement of simulation results and experimental data is fine.

9:10 AM
Fatigue Crack Growth Behavior of HAYNES HR-120 Superalloy: *Robert L. McDaniels*[1]; Stephen A. White[1]; Lijia Chen[1]; Yuehui He[1]; *Peter K. Liaw*[1]; Rodger R. Seeley[2]; Dwaine L. Klarstrom[2]; [1]The University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

Fatigue crack growth tests were conducted in laboratory air at temperatures ranging from 24°C to 982°C under the increasing and decreasing stress intensity factor range control modes on Fe-Cr-Ni based superalloy HAYNES HR-120. The sine waveform and a frequency of 10 Hz were employed. The applied R ratios (Kmin/Kmax, where Kmin and Kmax are minimum and maximum intensity factor, respectively) were ranging from 0.1-0.8. The influence of temperature and R ratio on fatigue crack growth rates was evaluated. It was found that fatigue crack growth rate per cycle increased with increasing temperature and R ratio. The relationship between the stress intensity factor range and fatigue crack growth rate was determined, based on Paris equation. The fracture surfaces of the tested specimens were characterized using scanning electron microscopy. The results showed that the fatigue crack predominantly propagated in a transgranular mode under the test conditions used in this investigation.

9:30 AM
Nanocrystalline Al-Base Alloy for Elevated Temperature Applications: *Sreekanth Purushotham*[1]; A. K. Ghosh[1]; [1]University of Michigan, Dept. of Matls. Sci. & Eng., 2300 Hayward, 2136 H.H. Dow Bldg., Ann Arbor, MI 48109 USA

Bulk nanocrystalline matrix alloys and composites are of interest for developing high strength for ambient and high temperature applications. Alloys with nanocrystalline dispersed phase and Al-base matrix ranging from partly to fully crystalline have been synthesized in this study. This has been achieved by ball milling, followed by a range of consolidation approaches including various solid-state and semi-solid processing routes. By varying the composition of the alloys, which contained primarily Al, Si and Y, different microstructures and mechanical properties have been obtained. The microstructures vary from a range of nanoparticle reinforced matrix alloy, whose composition is varied from an Al-rich phase to a Si-rich phase. Evolution of phases during consolidation processing was monitored by X-Ray Diffraction and Differential Scanning Calorimetry. Typical room temperature strength of these materials varies in the 700 MPa to 1300 MPa range, with equally impressive high temperature strength. Analysis of fracture surface reveals a quasi-brittle fracture mode which changes with alloy composition and test temperature.

9:50 AM Break

10:10 AM
Oxidation Behaviours of Yttrium-Modified NiCr and NiCrAl Alloys: *Anwar Ul-Hamid*[1]; [1]King Fahd University of Petroleum and Minerals, Matls. Characterization Lab, Ctr. for Eng. Rsrch., Rsrch. Inst., PO Box 1073, Dhahran 31261 Saudi Arabia

The effects of yttrium addition on the isothermal oxidation behaviours of Cr_2O_3 and Al_2O_3 forming alloys were investigated. The alloys used were based on NiCr and NiCrAl systems with and without Y additions. The exposures were carried out in air for 50 hours at 1000°C. The oxide microstructures were characterised by transmission electron microscopy along with scanning electron microscopy, optical microscopy and X-ray diffraction. It was observed that the oxide growth rate decreases and the scale adherence increases due to Y addition. The direction of growth for Cr_2O_3 oxide changes from predominantly outward to inward and counter current diffusion within Al_2O_3 is replaced by inwardly diffusion after Y addition. It was concluded that yttrium scavenges sulphur from the alloy and also segregates to the oxide grain boundaries thus primarily accounting for most of its beneficial effects observed in Y-modified alloys.

10:30 AM
High Temperature Creep Deformation Studies in Ti-6242 Alloy: *Gopal B. Viswanathan*[1]; Robert W. Hayes[2]; Michael J. Mills[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Metals Technology, Inc., 8955 Quartz Ave., Northridge, CA 91324 USA

In a companion talk, the high temperature creep behavior of a Ti-6Al-2Sn-4Zr-2Mo (T-6242) alloy having a lath microstructure with trace amounts of Ni is discussed. There, it was shown that increased amounts of Ni increased the minimum creep rates over the temperature range 510-565°C in the applied stress levels ranging from 170-410 MPa. In addition to the increased minimum creep rates, the activation energy values were found to decrease with increased Ni content. In this study, the dislocation structures after creep deformation have been analyzed. TEM analysis reveal that the dislocation density is very high in the a-phase compared to that of the b-phase, suggesting that the creep deformation is dominated by the a-phase. a-<11~20> type slip dominates the deformation in the a phase, though the configuration of a-<11~20> type dislocations differ depending on the applied stress. At high stresses (170-410 MPa), a-<11~20> type dislocations are seen mostly aligned in screw orientation with jogs pinning screw segments. At low stress (172 MPa) these a-type dislocations have long straight screw segments with no apparent pinning points and the near-edge segments were seen lying in climb configurations. Detailed analysis of these dislocations and other microstructural features along with possible creep mechanism that might be operative in these alloys will be presented.

10:50 AM
Mechanical Properties of Isothermally Forged $MoSi_2$-Based Lamellar Alloys: *Fu-Gao Wei*[1]; Yoshisato Kimura[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Eng., 4259 Nagatsuta, Midori-ku, Yokohama 226-8502 Japan

Two transition metal disilicides, $TaSi_2$ and $NbSi_2$, with the C40 crystal structure, form a lamellar structure with $MoSi_2$ ($C11_b$ structure) by conventional arc-melting method and homogenization in ($C11_b$+C40) two-phase field. A fully lamellar structure composed of C40 and $C11_b$ phases was obtained in $MoSi_2$ with 17mol% $TaSi_2$ or 15.5mol%$NbSi_2$. An isothermal forging at 2023K before heat-treatment at 1773K for 72h was applied on the two alloys in order to eliminate the solidification defects retained after arc-melting. Compression test at 1573K, 1673K and 1773K at a strain rate of $1x10^{-4}$/s reveals that additions of 17mol% $TaSi_2$ and 15.5mol%$NbSi_2$ increase the yield strength of $MoSi_2$ at 1673K to 307MPa and 327MPa respectively. In particular, over 10 percent compressive plastic strain was observed in the alloy with 17mol% $TaSi_2$ when tested at 1673K. Improvement of mechanical properties was discussed in terms of beneficial effect of coherent interfaces, decrease of solidification defects, introduction of mobile dislocations by forging, and low oxygen content by ingot metallurgy.

11:10 AM
High Temperature Oxidation of Ti-Al-Nb Alloys Containing Ta and Zr: *John C. Woo*[1]; S. K. Varma[1]; [1]The University of Texas at El Paso, Dept. of Metlgcl. & Matls. Eng., El Paso, TX 79968-0520 USA

Oxidation curves, graph between weight gain as a function of time of heating, for Ti-44Al-9Nb and Ti-44Al-11Nb alloys containing 2 atomic percents of Ta and Zr have been obtained. Addition of Zr to the ternary alloys produces less oxidation resistance than the quaternary alloy containing Ta. Oxidation curves have been compared with those produced by the ternary alloys. The addition of Ta as a quaternary alloying element appears to be quite beneficial for oxidation purpose. SEM has been used to identify the oxides formed in the scale and the nature of oxides has been investigated by EDX. TEM and optical microscopy results will be used to characterize the microstructures in the quaternary alloys. An attempt will be made to relate the microconstituents to the oxidation behavior.

Materials Design Approaches and Experiences - II

Sponsored by: Structural Materials Division, High Temperature Alloys Committee
Program Organizers: Ji-Cheng Zhao, GE Corporate Research & Development, Physical Metallurgy Laboratory, Schenectady, NY 12301 USA; Michael G. Fahrmann, Special Metals Corporation, Technology Department, Huntington, WV 25705-1771 USA; Tresa M. Pollock, University of Michigan, Materials Science and Engineering Department, Ann Arbor, MI 48109-2136 USA

Tuesday AM — Room: 204
November 6, 2001 — Location: Indiana Convention Center

Session Chair: Pierre Caron, ONERA, F-92322 Chatillon, Cedex, France

8:30 AM Invited
Modeling of Solidification and Microsegregation in Multicomponent Alloys: *Dilip Kumar Banerjee*[1]; [1]GE Global Exchange Services, Global Product Eng., 100 Edison Park Dr., Gaithersburg, MD 21045 USA

Numerical modeling of solidification of cast commercial alloys requires consideration of solidification path and microsegregation in multicomponent alloys. An accurate prediction of solidification path is needed to compute enthalpy as a function of temperature, which is a key input in solidification modeling software. In recent years, a number of thermodynamic databases have become commercially available for performing thermodynamic calculations using various microsegregation models. The effects of solid diffusion and dendrite tip kinetics on primary solidification are described. Predictions of solidification path involving multiphase reactions are also considered. The integration of this thermodynamic calculation of temperature-dependent enthalpy and fraction of solid into solidification modeling software is also discussed. Finally, benefits and shortcomings of using this approach in commercial casting simulation software are highlighted.

9:00 AM
Alloying and Single Crystal Superalloys for Power Generation: *Tresa M. Pollock*[1]; Sammy Tin[1]; [1]University of Michigan, MSE Dept., 2300 Hayward St., HH Dow 2042, Ann Arbor, MI 48109 USA

Advances in the development of nickel-base superalloy single crystals have enabled remarkable improvements in the performance and efficiency of aircraft engines. Similar improvements in the efficiency of electric power generation systems are possible. However, for this to occur, these complex alloys must be reliably cast as single crystals that are much larger, in terms of weight and physical dimensions. Additionally, given the thermal cycles of utility gas turbines, these alloys will be required to endure longer times at higher temperatures, compared to aircraft. The alloying characteristics of present single crystal compositions will be briefly reviewed. Aspects of alloying that strongly influence solidification behavior and alloy stability at elevated temperatures will be discussed for a large set of experimental alloys. Finally, possible directions for new alloys for power generation will be highlighted.

9:20 AM Invited

The Role of Technical Collaboration among Competitors to Advance the State of Technology: *Frank J. Zanner*[1]; James Van Den Avyle[2]; [1]ZanTek Enterprises; [2]Sandia National Laboratories, 1515 Eubank Blvd. S.E., Albuquerque, NM 87123-3453 USA

Improvements in business practices and productivity, fueled by the use of computers and advanced communications technologies, have resulted in a period of unprecedented prosperity. Further improvements are almost assured because of the continual advances in integration of computational capability and communication into a network environment. To flourish in this new environment, metals companies must generate new generic process control technologies which enable the integration of this network environment into the manufacturing process. These technologies will be expensive to generate, and in many instances beyond the scope of resources of a single company. In almost all cases the platform is generic, but the application is proprietary. We propose that a model already exists to generate this generic technology. The Specialty Metals Processing Consortium (SMPC) was created in 1989 by domestic companies that produce and use specialty metals such as Ni base superalloys, titanium, and high strength steels. SMPC was formed for the purpose of pooling technical and financial resources to generate generic technology in the field of liquid metal processing. In this instance the SMPC executed a cost share agreement with the Department of Energy to fund research at Sandia National Laboratories. Both parties contributed a total of about $2M annually to fund this research. Since the inception of this partnership in 1989 significant new technology has been developed that is being used in member company factories. This technology includes multi-variable process control and numerical simulation of vacuum arc remelting, measurement and characterization of ingot boundary conditions, and defect generation during liquid metal processing of superalloy ingots. A new partnership between the SMPC and the FAA has been formed to improve the quality of jet aircraft engine rotor grade materials and the focus will be on the suppression of melt related defects as the material is being processed. *A portion of this work was supported by the United States Department of Energy under Contract DE-AC04-94AL85000. Sandia National Laboratories is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy. Additional support was supplied by the Specialty Metals Processing Consortium.

9:50 AM Invited

PANDAT-A Program for Calculating Thermodynamic Multicomponent Phase and Related Diagrams: *Y. Austin Chang*[1]; Shuanglin Chen[2]; Stephen L. Daniel[2]; Fan Zhang[2]; [1]University of Wisconsin–Madison, MS&E, 1509 University Ave, Madison, WI 53706 USA; [2]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA

PANDAT, a program for thermodynamic calculations, will be presented. Given a set of thermodynamic parameters for all phases in a system and a set of user constraints, PANDAT automatically calculates the correct phase diagram without requiring initial values from the user. In this presentation, examples will be shown to illustrate the computational capability of this program from binary to multicomponent alloy systems. In addition, the calculation engine of PANDAT, named PanEngine, can be used to create custom programs for materials simulations. An example of using PanEngine to calculate the microsegregation of multicomponent aluminum alloys under different cooling conditions will be presented. The calculated results will be validated with data obtained from directional solidification.

10:20 AM Invited

Development of an ODS Alloy for an Aerospace Engine Vane Application: *John H. Weber*[1]; Rick K. Wilson[1]; [1]Special Metals Corporation, Tech. Dept., 3200 Riverside Dr., Huntington, WV 25705 USA

INCONEL® alloy MA754 is a powder metallurgy, oxide-dispersion strengthened (ODS) nickel-chromium alloy. It was developed in response to an urgent need for a commercially available alternative to TD NiCr®. The development criteria required alloy MA754 to have mechanical properties, physical properties, and hot corrosion resistance equal to or better than those of TD NiCr. The alloy design and development processes for alloys containing non-metallic constituents are different from those used in developing more conventional, melted alloys. The additional complexity for ODS alloys arises from the need to establish a stable, controlled size and size distribution of oxides within the alloy matrix, and from the role these dispersoids have on both processing and properties. Concurrent activities of alloy composition development, process development, and alloy scale-up to full commercial production were necessary in order to have a characterized and qualified commercial product within the time frame required.

10:50 AM Break

11:10 AM Invited

The Design of Aluminum Alloys: *Edgar A. Starke*[1]; [1]University of Virginia, Dept. of Matls. Sci. & Eng., 116 Engineer's Way, PO Box 400745, Charlottesville, VA 22904-4745 USA

This paper will discuss the use of phase diagrams and computer simulations for the design of improved age-hardenable aluminum alloys. The use of fundamental concepts that have been developed over the past fifty years from extensive processing/microstructure-development/property relationships can be used along with computer programs, e.g. CALPHAD, to calculate desired compositions. Computer simulations can then be used to identify the microstructure that will result in the best combination of mechanical properties. The talk will be based on our current research aimed at developing new aluminum alloys for moderate temperature applications. The approach should streamline alloy design and therefore aid in the early insertion of new materials through the marriage of phase diagram analysis, simulation and modeling techniques.

11:40 AM Invited

The Role of Quantum Mechanics in "Virtual Aluminum Castings": *Christopher Mark Wolverton*[1]; [1]Ford Motor Company, MD 3028/SRL, PO Box 2053, Dearborn, MI 48121 USA

Increasing demands to further reduce emissions and simultaneously improve fuel economy in automobiles has expanded the need for lightweight materials (such as Al, Mg, and their alloys). In order to optimize alloy design and processing conditions to quickly achieve Al-alloy castings with suitable mechanical properties, researchers at Ford Research Laboratory are developing the Virtual Aluminum Castings methodology: a suite of predictive computational tools that span length scales from atomistic to macroscopic to describe alloy microstructure, precipitation, solidification, and ultimately, mechanical properties. The role of first-principles atomistic computations in the Virtual Aluminum Castings methodology will be described. Because of their highly accurate and predictive nature, there is a growing desire to use these types of theoretical approaches to predict properties of new, experimentally unexplored, or difficult-to-synthesize solids. Application to problems of precipitation, thermal growth, and microstructure evolution during heat treatment has proved very fruitful. Combining these quantum-mechanical results with other modeling and experimental efforts, one can suggest heat treatments which optimize thermal stability and hardness of industrial alloys.

Mechanical Behavior & Constitutive Modeling of Polymers, Fiber- & Particulate-Reinforced Polymer Composites & Polymer-Based Nanocomposites - I: Strain Rate and Temperature Effects on the Behavior of Unfilled Polymers

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials, SPE Polymer Analysis Division, Jt. Composite Materials Committee
Program Organizers: George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Linda Schadler, Rensselaer Polytechnic Institute, Materials Science & Engineering Department, Troy, NY 12180-3590 USA; Carl Schultheisz, National Institute of Standards and Technology, Polymers Division, Gaithersburg, MD 20899 USA

Tuesday AM Room: 201
November 6, 2001 Location: Indiana Convention Center

Session Chairs: George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Cate Brinson, Northwestern University, MEAS Mechl. Eng., Evanston, IL 60208-3111 USA

8:30 AM Keynote

Hierarchical Aspects to Toughening Polymers: *Alan J. Lesser*[1]; [1]University of Massachusetts, Conte Rsrch. Ctr., Polymer Sci. & Eng. Dept., Amherst, MA 01003 USA

Today, many strategies are employed to enhance the modulus, strength, and energy absorbing characteristics of polymer systems. These strategies are applied at a variety of length scales. On the molecular scale, the molecular architecture is altered by changes in backbone stiffness, crosslink density, crosslink functionality, or through the addition of small molecular fortifiers. On the nanoscale, the nanosilicates in either an exfoliated or intercalated form are added to the polymer. On the micron scale, toughening by the addition of rubber particles is commonplace today. In this presentation, the mechanisms of reinforcement and toughening achieved from each of these strategies are discussed. Results of deformation and fracture in a variety of multi-axial stress states are presented. At each length scale, the trade off between reinforcement and toughening through controlled energy dissipation is discussed. Issues associated characteristic size and spacing at the various length scales is also discussed.

9:00 AM Keynote

Nanocomposite Rheological and Performance Modeling: *Jozef Bicerano*[1]; Douglas A. Brune[1]; Jack F. Douglas[2]; [1]The Dow Chemical Company, Corp. R&D, 1702 Bldg., Off. 206 C, Midland, MI 48674 USA; [2]NIST, Polymers Div., Polymers Bldg. (224), Rm. B222, 100 Bureau Dr., MS 8542, Gaithersburg, MD 20899-8542 USA

Modeling plays a significant and rapidly growing role in research and development on new materials such as nanocomposites. Some of the major challenges in nanocomposite rheological and mechanical property modeling will be addressed. A new model was developed for the shear viscosity of dispersions of highly anisotropic particles in polymer melts and in solutions. In addition, the elastic properties of nanocomposites were investigated by means of micromechanical models. Extensions were made in such models, to assess the effects of the possibility of platelet buckling on the compressive modulus, and to predict the effects of incomplete exfoliation and imperfect alignment of the platelets.

9:30 AM

Constitutive Modeling of the Strain Rate Dependent Tensile Response of Polymer Matrix Composites: *Robert K. Goldberg*[1]; Gary D. Roberts[2]; Amos Gilat[3]; [1]NASA Glenn Research Center, Struct. Div./Life Prediction Branch, 21000 Brookpark Rd., MS 49-7, Cleveland, OH 44135 USA; [2]NASA Glenn Research Center, Matls. Div./Polymers Branch, 21000 Brookpark Rd., MS 49-1, Cleveland, OH 44135 USA; [3]Ohio State University, Mechl. Eng., 206 W. 18th Ave., Columbus, OH 43210 USA

Research is underway to develop strain rate dependent deformation and failure models for the analysis of polymer matrix composites subject to high strain rate impact loads. As part of this process, tensile stress-strain curves have been generated for representative ductile polymers and carbon fiber reinforced composites with various fiber orientations over strain rates ranging from quasi-static to several hundred per second. The high strain rate tests are conducted using a unique tensile split Hopkinson bar. The experimental results show that the deformation response if nonlinear and rate dependent. An analytical model has been developed in which state variable constitutive equations are used to simulate the nonlinear, rate dependent deformation of the polymer matrix. A mechanics of materials micromechanics method is then used to compute the effective properties and response of the composite. Stress-strain curves generated using the analytical model match the experimental results well.

9:50 AM

Strain-Rate, Pressure, and Temperature Effects in Rheological Models for Polymers: *Marc Andre Meyers*[1]; David J. Benson[1]; Vlado A. Lubarda[1]; [1]University of California, San Diego, Dept. of Mechl. & Aeros. Eng., La Jolla, CA 92093 USA

Analytical solutions for the stress-strain response of polymers are derived based on one-dimensional viscoplastic rheological models. Both hardening and softening features are encompassed in the analysis, as well as pressure, temperature, and strain-rate effects. The non-Newtonian viscosity is used to describe the viscous part of the response. The material behavior corresponding to a prescribed piecewise-linear strain history is also considered. The results are applied to examine the effects of an abrupt change of strain rate on the stress response during monotonic loading. Comparison with experimental results presented in the literature is presented.

10:10 AM Break

10:30 AM

Effect of Temperature and Strain Rate on the Tensile Behavior of Nylon 6: *P. K. Mallick*[1]; Yuanxin Zhou[1]; [1]University of Michigan–Dearborn, Dept. of Mechl. Eng., Dearborn, MI 48128 USA

In the present paper, tensile behavior of Nylon 6 has been determined at three different strain rates (0.05/min, 0.5/min, 5/min) and at four different temperatures (21.5°C, 50°C, 75°C, 100°C). Experimental results show that Nylon 6 is a typical strain rate and temperature dependent material. Both modulus and ultimate tensile strength of the material increase with increasing strain rate and decrease with increasing temperature. Experimental results also show that both strain rate sensitivity and temperature sensitivity of its modulus change at a temperature between 25 and 50°C, possibly due to glass transition. Based on the observed stress-strain curves, a one-dimensional strain rate and temperature dependent constitutive model has been established to describe the tensile behavior of Nylon 6. The mechanical parameters in this model and their relationships with strain rate and temperature are obtained from the experimental results. The simulated stress-strain curves from the model are in good agreement with test data. The model analysis results also show that the compliance parameter ß, which controls the inelastic deformation of the material, is decreases with strain rate and increases with temperature.

10:50 AM

Large Deformation Torsional Response of Polycarbonate: Time-Temperature and Time-Strain Superposition: *Paul O'Connell*[1]; Greg McKenna[1]; [1]Texas Tech University, Dept. of Cheml. Eng., Lubbock, TX 79409-3121 USA

Data are presented from tests of the stress relaxation response of a polycarbonate under torsional deformations. Tests were performed on samples over a range of strains from 0.0025 to 0.07 and at temperatures from 30°C to 135°C, all at a fixed aging time of 64800 s. Individual data sets at each strain and temperature could be described using a stretched exponential form relaxation function. Over the range of temperatures studied the data at each strain could be superimposed using conventional time-temperature superposition. For strains up to the yield strain the data at each temperature could also be superimposed to form a master curve following the principle of time-strain superposition. Interestingly, the master curves found from time-strain superposition at each temperature did not have the same form. Similarly, the master curves found from time-temperature superposition at each strain did not have the same form.

11:10 AM

Influence of Temperature and Strain Rate on the Constitutive Behavior of Peek and Viton A: *George T. Gray*[1]; William R. Blumenthal[1]; Carl M. Cady[1]; [1]Los Alamos National Laboratory, MS G755, MST-8, Los Alamos, NM 87545 USA

The high-strain-rate stress-strain response of polymeric materials has received increased interest in recent years related to the need for predictive constitutive model descriptions for use in large-scale finite-element simulations of automotive crash-worthiness and aerospace impacts. While numerous studies have investigated the influence of strain rate on the constitutive response of a range of polymers, the influence of temperature at high-strain rate on polymer mechanical behavior has received more limited attention. In this paper the effect of strain rate and temperature on the mechanical responses of PEEK and Viton A is presented. The compressive stress-strain responses of PEEK and Viton A were found to depend on both the applied strain rate and the test temperature. Due to the slow, dispersive wave propagation in PEEK and Viton A, thinner sample thicknesses were found to be required to assure uniform, uniaxial stress conditions within Hopkinson Bar samples.

11:30 AM

The Strength of PMMA and Polycarbonate Polymers as a Function of Temperature and Strain Rate: *William R. Blumenthal*[1]; Carl M. Cady[1]; George (Rusty) T. Gray[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G-755, Los Alamos, NM 87545 USA

Compression stress-strain measurements have been made on commercial polymethylmethacrylate (PMMA) and polycarbonate polymers as a function of temperature (-197°C to +220°C) and strain rate. A split-Hopkinson-pressure bar (SHPB) was used to achieve strain rates of about 2500 1/s and a conventional testing machine was used for low strain rates (0.001 1/s). The mechanical response of these semi-transparent polymers is quite different. The strength of polycarbonate is only moderately dependent on strain rate and temperature and deforms without damage to at least -55°C. Surprisingly, the loading (tangent) modulus of polycarbonate is virtually constant in the range -55°C to +55° C at low strain rates. In contrast, the strength and tangent modulus of PMMA is very temperature and strain rate sensitive above room temperature. Below room temperature, the mechanical response of PMMA becomes insensitive to temperature at high strain rates and brittle fracture occurs at low total strain (7-8%).

11:50 AM

Effects of Nanostructure and Microstructure on Fracture Toughness of Polycarbonate: *Brendan J. Ward*[1]; *David M. Stepp*[1]; George W. Pearsall[1]; [1]Duke University, Dept. of Mechl. Eng. & Matls. Sci., PO Box 90300, 144 Pratt Sch. of Engineering, Durham, NC 27705-0300 USA

Glassy polycarbonate is a premier engineering material because of its combination of optical clarity and high impact strength. Its high impact strength has been attributed in part to a relatively large free volume for a glassy (below Tg) polymer. As crystallization and molecular orientation affect polycarbonate's density, as well as its mechanical and optical properties, this paper presents research results on the effects of crystallization and local amorphous orientation on the fracture toughness of polycarbonate. Several experimental techniques were used to probe the microstructure and nanostructure of crystalline, amorphous, and locally oriented bisphenol-A polycarbonate, including polarized light microscopy, scanning electron microscopy, differential scanning calorimetry, and positron annihilation lifetime spectroscopy.

12:10 PM
Creep Analysis of Thermoplastics Based on Stress Relaxation Measurements: *David A. Woodford*[1]; [1]MPA, Inc., 1707 Garden St., Santa Barbara, CA 93101 USA

One of the major factors limiting the use of thermoplastics in engineering applications is the inadequacy of data spanning appropriate ranges of stress, strain, time and temperature. By differentiating stress vs. time curves obtained from high precision stress relaxation tests, curves of stress vs. creep rate or stress vs. stress rate may be obtained covering up to five orders of magnitude in rate from tests lasting less than one day. These curves, in turn, may be used to generate pseudo stress vs. strain curves, from which secant modulus data may be generated as functions of temperature, time and strain. Using measurements on Lexan, Noryl and Valox* it is shown that conventional creep strain vs. time curves may also be generated which give good agreement with actual test data. * Lexan, Noryl and Valox are registered trademarks of GE Company.

Microstructure Modeling and Prediction During Thermomechanical Processing - II

Sponsored by: Structural Materials Division, Materials Processing & Manufacturing Division, Shaping and Forming Committee, Titanium Committee
Program Organizers: Raghavan Srinivasan, Wright State University, Department of Mechanical & Materials Engineering, Dayton, OH 45435 USA; Armand J. Beaudoin, University of Illinois at Urbana–Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Steve P. Fox, Timet Corporation, Timet Henderson Technical Laboratory, Henderson, NV 89009 USA; Zhe Jin, Reynolds Metals Company, Metallurgy Department, Chester, VA 23836-3122 USA; S. L. Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Tuesday AM — Room: 208
November 6, 2001 — Location: Indiana Convention Center

Session Chairs: Armand Beaudoin, University of Illinois at Urbana–Champaign, Dept. of Mechl. & Indl. Eng., Urbana, IL 61801 USA; Balasubramaniam Radhakrishnan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6359 USA

8:30 AM
Monte Carlo Computer Simulation of Recrystallization and Grain Growth in BCC Metals: *Kaneharu Okuda*[1]; Anthony D. Rollett[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., Wean Hall 4315, 5000 Forbes Ave., Pittsburgh, PA 15213 USA

A Monte Carlo simulation technique was employed to model the recrystallization and subsequent grain growth of deformed BCC materials. Variable grain boundary energy and mobility were incorporated into the model as a function of the crystallographic boundary type. Recrystallization nuclei were distributed in the deformed matrix both uniformly and restricted to prior grain boundaries. Special properties of grain boundaries, notably the high mobility associated with the coincident site lattice sigma-19a boundary, were investigated as a test of the oriented growth theory, which has been used to explain the {111} recrystallization texture of IF steels. Also the effect of second phase particles of various characteristics on texture evolution was simulated. Preliminary results of testing various assumptions about texture development will be presented.

8:55 AM
Monte Carlo Simulation of Annealing of Electrical Steel after Temper Rolling: Anthony D. Rollett[1]; *Soonwuk Cheong*[1]; Erik J. Hilinski[2]; [1]Carnegie Mellon University, Matls. Sci. & Eng., Wean Hall 4315, 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]US Steel, US Steel Rsrch. & Tech. Ctr., 4000 Tech Center Dr., Monroeville, PA 15146 USA

The dependence of magnetic properties such as core loss and peak permeability on the temper rolling process has been studied in a semi-processed, cold rolled magnetic lamination (CRML) steel. The results indicate that temper rolling parameters such as temper mill extension and roll roughness have a significant influence on the magnetic properties. Material processed with high temper mill extension and smooth work rolls shows a sharper texture, which results in highly anisotropic peak permeability values between the rolling direction and the transverse direction. Microstructural and texture analysis suggests that temper rolling with both high extension and smooth work rolls concentrates strain at the surface, which would explain the development of the rolling texture at the surface. In order to learn more about the importance of variations in stored energy with orientation, versus the anisotropy of grain boundary properties, Monte Carlo simulation has been employed. Experimental microstructures measured with electron back-scatter diffraction (EBSD) have been used to define initial structures for input to the simulation. The influence of inhomogeneous stored energy and anisotropic grain boundary properties on the growth of the Goss, Cube and rotated cube components has been investigated.

9:20 AM
Phase Field Modeling of Microstructural Evolution in Ti-Al and Ni-Al: *Yunzhi Wang*[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

The key to predicting and therefore controlling properties of materials is the knowledge of microstructure. Over the last few years, the continuum phase field method has received an increasing amount of attention in simulating microstructure evolution in various material systems and processes. Several reasons may have contributed to its popularity. For example, it is capable of simulating realistic microstructures and their evolution under conditions of high volume fraction, elastically interacting particles, and involving multiple ordered domains and orientation variants. From the simulation, detailed information can be obtained on sizes and shapes of individual particles and domains and their spatial distributions at each time moment during the microstructural evolution. In this presentation, a brief overview of recent applications of the phase field method in simulating microstructural evolution during solid state phase transformations and other related processes will be given. The general formulation of the method and basic parameters that must be determined externally will be discussed. A number of new applications to modeling, for example, complex microstructural development during thermal processing of Ti-Al alloys and Ni-based superalloys will be presented. The work is supported by the National Science Foundation.

9:45 AM
Spectral Representation of Microstructure Evolution in Polycrystals: *Hamid Garmestani*[1]; Surya Kalidindi[2]; Brent Adams[3]; [1]FAMU-FSU College of Engineering, Mechl. Eng., 2525 Pottsdamer Rd. #229, Tallahassee, FL 32310 USA; [2]Drexel University, Matls. Sci. & Eng., Philadelphia, PA 19104 USA; [3]Brigham Young University, Mechl. Eng., B 435 CT, Provo, UT 84602 USA

Fourier series representations of polycrystalline microstructure provide an economical basis to describe the set of all possible microstructures ('material hull') and the set of all possible properties/performance relationships ('iso-property surfaces') for a specified selection of physics and mechanics. The spectral method provides a novel inductive method for the design of microstructures to meet specified performance criteria. It is also possible to directly describe the evolution of microstructures under specified processing conditions as a family of streamlines in the material hull. In some cases it is possible to integrate along streamlines to ascertain the appropriate processing conditions required to achieve specified optimized microstructures. We present the general framework for spectral representation of microstructure evolution, illustrating the methods with a selected example.

10:10 AM Break

10:25 AM
Stochastic Method for the Evolution of Microstructures with Particular Reference to Grain Boundary Networks: *Mukul Kumar*[1]; Roger W. Minich[1]; James Stölken[1]; Wayne E. King[1]; Adam J. Schwartz[1]; [1]Lawrence Livermore National Laboratory, 7000 E. Ave., L-356, Livermore, CA 94550 USA

Grain boundary engineering has been successfully applied to improve materials properties such as intergranular corrosion and cracking, creep, weldability, and ductility. This has been attributed to an increase in the fraction of "special" grain boundaries resulting from thermomechanical processing. Our investigations, however, have led us to conclude that the basis for these improvements is most likely to lie in the break-up of the connectivity of random boundary networks. The experimental determination of the network topology has been accompanied by a microstructure modeling effort that is derived from the percolative nature of the network. The problem of grain boundary networks, under crystallographic constraint, has been simulated using a stochastic approach that considers the probability distribution function for each boundary based on the fluctuations in strain energy (from deformation) and intrinsic characteristics such as interfacial energy. This approach toward microstructural evolution during recrystallization and grain growth will be elaborated. Experimentally derived measures to quantify microstructures such as in the grain boundary character and triple junction distributions will be compared to those obtained from simulations. This work was performed under the auspices of the US Department of Energy by University of California, Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

10:50 AM

Anisotropic Grain Boundary Properties in Aluminum: Effect on Microstructural Evolution: *M. Upmanyu*[1]; D. J. Srolovitz[1]; H. Zhang[1]; B. Radhakrishnan[2]; [1]Princeton University, Mechl. & Aeros. Eng., 418 Bowen Hall, 70 Prospect Ave., Princeton, NJ 08544 USA; [2]Oak Ridge National Laboratory, Compl. Matls. Sci. Grp., PO Box 2008, MS-6359, Oak Ridge, TN 37831-6359 USA

We present results of three-dimensional molecular dynamics simulations of curvature driven grain boundary migration in pure aluminum. Following the work of Shvindlerman et al., the classical U-shaped half-loop bicrystal simulation geometry is employed to observe steady-state boundary migration. Examination of half-loop shrinkage rates for different half-loop widths and temperatures indicates that for sufficiently low driving forces, grain boundary velocity is proportional to boundary curvature and that the boundary mobility is an Arrhenius function of temperature. The grain boundary mobilities and energies are also extracted as function of boundary crystallographic parameters. In particular, these boundary properties are extracted as a function of boundary misorientation for <111> tilt grain boundaries. These anisotropic properties are then used in three-dimensional Potts model to ascertain their effect on microstructural evolution.

11:15 AM

Microstructural Evolution in Sprayformed Steel: Phase Transitions, Inelasticity, Residual Stress and Distortion: *Mark T. Lusk*[1]; Rojer Jaramillo[1]; Phillip Liu[1]; Allen D. Roche[2]; Chijoke Mgbokwere[2]; Samir Deshpande[2]; [1]Colorado School of Mines, Matls. Sci. Prog., Div. of Eng., 1500 Illinois St., Golden, CO 80401-1887 USA; [2]The Ford Motor Company, Ford Rsrch. Lab., USA

Steel parts can be rapidly produced by spraying entrained droplets of melted wire stock onto a ceramic master. In such sprayforming operations, the final distortion, residual stress, and microstructure are known to be sensitive functions of the processing conditions, but even qualitative links between process and property have yet to be firmly established. In an effort to remedy this, a one-dimensional, through thickness, heat transfer equation is used to model mass deposition and temperature evolution in the growing sprayformed piece; instantaneous mass deposition at regular intervals idealizes the actual spray gun pattern at a point. Because of the large temperature gradients involved, the implicit scheme employed is checked against a separate model in which the temperature field is mapped back to a reference domain of fixed length. In this second paradigm, the deposition rate is taken to be the average of that in the first approach, and thermal properties that are regularized at the interface between sprayform and ceramic. The models are shown to compare well. The temperature field is then linked to internal state variable equations (a set of coupled, nonlinear, ordinary differential equations) for the decomposition of austenite to ferrite, pearlite, bainite and martensite as well as the evolution of dislocation structure through plastic flow and strain hardening. This model is used to obtain the final residual stress and shape as functions of processing conditions by feeding the microstructural data to a final routine that simulates the removal of the ceramic master. It is argued that, in addition to thermal and elastic strains, the timing of the interplay between inelastic deformation and phase transitions plays a crucial role in determining the final distortion and residual stress in the part. Model predictions are compared with experimental results for various deposition rates, initial ceramic temperatures, and wire stock carbon levels.

Modeling the Performance of Engineering Structural Materials - II: Fracture and Fatigue

Sponsored by: Structural Materials Division, Structural Materials Committee
Program Organizers: Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA

Tuesday AM Room: 140
November 6, 2001 Location: Indiana Convention Center

Session Chairs: E. M. Taleff, University of Texas, Dept. of Mechl. Eng., Austin, TX 78712 USA; D. L. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

8:30 AM

Low-Cycle Fatigue Behavior and Creep-Fatigue Life Prediction of Three Superalloys: *Lijia Chen*[1]; *Peter K. Liaw*[1]; Robert L. McDaniels[1]; James W. Blust[2]; Scott A. Thompson[2]; Paul F. Browning[2]; Rodger R. Seeley[3]; Dwaine L. Klarstrom[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Solar Turbines, Inc., 2200 Pacific Hwy., PO Box 85376, MZ R-1, San Diego, CA 92186-5376 USA; [3]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The low-cycle fatigue tests with and without hold times were conducted in laboratory air at 816°C and 927°C under a total axial strain range control mode to investigate high-temperature fatigue and creep-fatigue interaction behavior of three superalloys, including HAYNES 188, 230, and HASTELLOY X. It was found that under both low-cycle fatigue and creep-fatigue loading conditions, the strain fatigue lives of three superalloys were dependent on the type of the alloy, imposed total strain range, and test temperature. The scanning electron microscopy examination on the fracture surfaces of the tested specimens revealed that both transgranular and intergranular crack propagation modes could be observed. The frequency-modified tensile hysteresis energy modeling was used to correlate the present strain fatigue life data. The result showed that this modeling could give a satisfactory prediction on the creep-fatigue lives of three superalloys.

8:55 AM

The Fatigue Fracture Behavior of an Aluminum Alloy-SiC Particulate Metal-Matrix Composite: *Meslet Al-Hajri*[1]; T. S. Srivatsan[1]; X. Gao[1]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

In this technical presentation the influence of discontinuous ceramic particulate reinforcements on high cycle fatigue response and final fracture behavior of 7034 aluminum alloy metal-matrix composites will be highlighted. The cyclic stress amplitude-controlled fatigue properties and fracture characteristics of the 7034 aluminum alloy metal matrix discontinuously-reinforced with silicon carbide particulates will be highlighted for a range of cyclic stress amplitudes and at two different load ratios. The influence of test temperature and load ratio on stress amplitude versus fatigue life stress response will be rationalized. Also an attempt will be made to elucidate the intrinsic mechanisms governing stress response and fatigue fracture characteristics in light of the competing and mutually interactive influences of composite microstructural effects, deformation characteristics of the composite constituents, cyclic stress amplitude, load ratio and test temperature. Research partially supported by: Air Force Materials Laboratory (USAF), Wright Laboratories (Dayton, OH), and The University of Akron (Akron, OH)

9:20 AM

A Weibull Stress Model for Prediction of Cleavage Fracture in Ferritic Steels: *X. Gao*[1]; R. H. Dodds[2]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA; [2]University of Illinois at Urbana–Champaign, Dept. of Civil Eng., Urbana, IL 61801 USA

This study applies recent advances in the Weibull stress model to predict cleavage fracture in ferritic steels. The procedure to calibrate the Weibull stress parameters builds upon the toughness scaling model between two crack configurations having different constraint levels and eliminates the recently discovered non-uniqueness that arises in calibrations using only fracture toughness data obtained under small scale yielding (SSY) conditions. The introduction of a non-zero threshold value for Weibull stress in the expression for cumulative failure probability is consistent with the experimental observations that there exists a minimum toughness value for cleavage fracture in ferritic steels, and brings numerical predictions of the scatter in fracture toughness data into better agreement with experiments. In this work, the Weibull stress parameters are calibrated using fracture toughness data of deep-notch C(T) specimens and shallow-notch SE(B) specimens of an A515-70 steel. The calibrated model is then applied to predict the measured response of surface-cracked plates of the same material loaded in different combinations of tension and bending. The model predictions accurately capture the measured distributions of fracture toughness values.

9:45 AM

Fracture Initiation and Slip Transfer at Gamma-TiAl Grain Boundaries: *Benjamin A. Simkin*[1]; Darren E. Mason[1]; Thomas R. Bieler[1]; Martin A. Crimp[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering, E. Lansing, MI 48824 USA

Fracture initiation in the intermetallic compound TiAl has been examined by studying the characteristics of grain boundaries at which cracks form or extensive deformation transfer is observed. The characterization of deformation defects and relative grain orientation has been accomplished through the use of selected area channeling patterns (SACPs) and electron channeling contrast imaging (ECCI). True grain orientations (including the grain tetragonality) are obtained via superlattice information available in the SACPs, while the use of ECCI allows the imaging of dislocations and deformation twins. The combination of the two allows trace analysis, from which grain-grain conformality factors may be calculated. The shear transmission is examined for grain boundaries that display fracture initiation, as well as those for which there is no separation at the grain boundary and shear transmission is apparent. The grain to grain shear transmission is correlated with deformation compatibility factors already in the literature. Preliminary investigations into a new variational model of slip transfer will also be discussed.

10:10 AM Break

10:30 AM
Fatigue Behavior of Type 316 Stainless Steel: Experiment and Theoretical Modeling: *Hongbo Tian*[1]; P. K. Liaw[1]; D. Fielden[1]; C. R. Brooks[1]; M. D. Brotherton[1]; J. P. Strizak[2]; L. K. Mansur[2]; J. R. DiStefano[2]; K. Farrel[2]; [1]The University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Type 316 stainless steel is being considered as a candidate target container material for the Spallation Neutron Source (SNS). Satisfactory behavior under fatigue loads is a requirement for the target container. Stress-controlled fatigue tests were performed on the 316 stainless steel at 0.2 Hz and 10 Hz with an R ratio of -1. Note that R=smin/smax, where smin and smax are the minimum and maximum applied stresses, respectively. Significant differences in fatigue lives as a function of temperature were observed with shorter fatigue lives at higher temperatures. At R=-1, a large specimen temperature increase at 10 Hz was observed, which approaches about 340°C at the stress amplitude of 245 MPa. The temperature at 0.2 Hz was about room temperature. Different temperatures were achieved by varying the load cycling frequencies. A model based on the dissipation energy of the specimen during fatigue tests was developed to explain the fatigue life data and predict the specimen temperature.

10:55 AM
Temperature Evolution during Cyclic Fatigue of ULTIMET Alloy: *Liang Jiang*[1]; Hsin Wang[2]; Peter K. Liaw[3]; Charlie R. Brooks[3]; Dwaine L. Klarstrom[4]; [1]GE CRD, Physl. Metall. Lab., Schenectady, NY 12309 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., Oak Ridge, TN 37831 USA; [3]University of Tennessee, Matls. Sci. & Eng., Knoxville, TN 37996 USA; [4]Haynes International, Inc., 1020 W. Park Ave., Kokomo, IN 46904 USA

High-speed, high-resolution infrared thermography, as a non-contact, full-field, and nondestructive technique, was used to study the temperature variations of a cobalt-based ULTIMET alloy subjected to cyclic fatigue. During each fatigue cycle, the temperature oscillations, which were due to the thermal-elastic-plastic effect, were observed and related to stress-strain analyses. The change of temperature during fatigue was utilized to reveal the accumulation of fatigue damage. A constitutive model was developed for predicting the thermal and mechanical responses of ULTIMET alloy subjected to cyclic deformation. The model was constructed in light of internal state variables, which were developed to characterize the inelastic strain of the material during cyclic loading. The predicted stress-strain and temperature responses were found to be in good agreement with the experimental results. In addition, the measured temperature was utilized as an index for fatigue-life prediction.

11:20 AM
Modeling the Influence of Ductile Phase Reinforcement on the Cyclic Fatigue Fracture Behavior of Dispersion Strengthened Copper-Niobium Microcomposite: *T. S. Srivatsan*[1]; Meslet Al-Hajri[1]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

A study has been made to understand the cyclic stress response characteristics, cyclic stress-strain response, cyclic strain resistance and fracture behavior of an oxide dispersion strengthened copper-niobium composite. The composite specimens were cyclically deformed over a range of strain amplitudes and stress amplitudes at both ambient and elevated temperatures. Under strain-amplitude control the composite specimens displayed combinations of hardening and softening to failure. The cyclic stress-strain characteristics, fatigue properties and fracture behavior of the composite will be highlighted and temperature influences on behavior will be rationalized in light of the competing and mutually interactive influences of cyclic strain amplitude and resultant response stress, cyclic stress amplitude, intrinsic microstructural effects, matrix deformation characteristics and macroscopic aspects of fracture. Research supported by The University of Akron (Akron, OH) and State of OH: Board of Regents (Columbus, OH), with material support from OMG Americas (Raleigh, NC, Program Monitor: Mr. J. D. Troxell).

11:45 AM
Effect of Strain-Rate on Compressive Failure of a Unidirectional Carbon-Epoxy Composite: *Sathiemoorthy Sivashanker*[1]; C. S. Meng[1]; [1]Nanyang Technological University, Sch. of Mechl. & Productions Eng., Vanyang Ave., Singapore, 639798 China

This paper is on an experimental investigation of the high-strain-rate compressive failure of a carbon fibre-epoxy composite using a Split Hopkinson Pressure Bar (SHPB) facility, suitable for dynamic compression testing of high-strength composite materials. Our investigation shows that the uniaxial compressive strength of T800924C epoxy based fibre reinforced composite is insensitive to high-strain rates of up to 3500/s. Failure is by fibre microbuckling with attendant splitting and delarmination, similar to those observed in quasi-static compressive failure. It is also noted that in the dynamic compression testing of unidirectional fibre-reinforced composites along the fibre direction, high strain rates can only be attained by using large impact velocities rather than by a decrease in composite specimen length, if the results are to be meaningful and representative of bulk material properties of the composite.

Polymers and Other Organics for Electronic Applications: Micro-to Nano-Technology

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, SPE Polymer Analysis Division
Program Organizers: Christos Dimitrakopoulos, IBM T.J. Watson Research Center, Yorktown Heights, NY 10598 USA; Shubhra Gangopadhyay, Texas Tech University, Department of Physics, Lubbock, TX 79409 USA; Linda Schadler, RPI, Materials Science & Engineering Department, Troy, NY 12180-3590 USA

Tuesday AM — Room: 202
November 6, 2001 — Location: Indiana Convention Center

Session Chairs: Christos Dimitrakopoulos, IBM T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; Shubhra Gangopadhyay, Texas Tech University, Dept. of Phys., Lubbock, TX 79409 USA

8:30 AM Keynote
Charge Injection and Transport in Organic Semiconductor Devices: *George Malliaras*[1]; [1]Cornell University, Matls. Sci. & Eng., 327 Bard Hall, Ithaca, NY 14853-1501 USA

Over the last few years organic materials are being considered as active layers in a variety of optoelectronic devices that include light emitting diodes, thin film transistors, photovoltaic cells and photorefractives. Combining advantages such as ease of processing, compatibility with large area and flexible substrates and optoelectronic properties that can be tailored via chemical synthesis, organic semiconductors are on the verge of commercialization. In all these devices, charge injection at metal/organic interfaces and charge transport in the organic layer play a vital role. Recent experiments that probe the fundamentals of these two processes will be discussed. The process of charge injection was studied at the contact between Indium Tin Oxide (ITO) and the molecularly doped polymer triphenyl diamine doped polycarbonate (PC:TPD). We find that the contact is current-limiting, despite the fact that the current has a similar electric field dependence as the space charge limited current. This behavior is independent of the mobility in the organic layer. The data are analyzed in terms of a recent injection model for disordered materials. It will be shown that the contact can be made Ohmic with the introduction of ultra-thin Pt or Pd layers at the interface between ITO and PC:TPD. The process of electron transport was studied in Alq3, which is the most commonly used electron transporter in organic light emitting diodes. Non-dispersive photocurrent transients indicate the absence of intrinsic traps in well-purified films. Exposure of the films to the ambient atmosphere results in highly dispersive transport, indicating that oxygen is a likely candidate for a trapping site. The mobility was found to obey the Poole-Frenkel law. We use the correlated disorder model to determine an effective dipole moment for Alq3, and the corresponding meridional to facial isomeric ratio.

9:00 AM
Modeling Nanoporosity Development in Crosslinked Systems for Low-k Applications: Prathamesh Doshi[1]; *Sindee L. Simon*[1]; [1]Texas Tech University, Cheml. Eng., MS 3121, Lubbock, TX 79409 USA

A model has been developed to describe nanoporosity development in crosslinked polymer films for use as low-k dielectrics. The process modeled consists of first swelling the polymer with supercritical CO2 at high pressures followed by a pressure quench. The pressure quench results in supersaturation which leads to bubble formation and growth. Subsequent stabilization of the bubbles produces nanopores. A parametric analysis has been performed to analyze how material properties and process variables affect nanopore size and distribution. In particular, we examine the effects of crosslink density and film thickness, as well as the time, temperature and pressure process history. The model components include time-temperature and time-CO2 concentration superposition to characterize the viscoelastic behavior of the polymer film and the diffusivity of CO_2 in the film; an equation of state to describe supercritical CO_2 behavior; and a lattice model to quantify the equilibrium concentration of CO_2 in the film.

9:20 AM

Novel Nanoporous Low-k Dielectrics: Juan Sun[1]; Prathamesh Doshi[1]; Jorge Lubguban[2]; Narendra Mehta[2]; Bashar Lahlouh[2]; Sindee L. Simon[1]; *Shubhra Gangopadhyay*[2]; [1]Texas Tech University, Cheml. Eng., MS 4121, Lubbock, TX 79409 USA; [2]Texas Tech University, Phys., MS 1051, Lubbock, TX 79409 USA

We are investigating new routes to making low-k dielectric films for use in next generation interconnects. Plasma enhanced chemical vapor deposition (PECVD) of novel monomeric and conventional organosilicate precursors are used to deposit the films. In addition, model PMMA spin-coated films are investigated. Supercritical carbon dioxide processing at pressures ranging from 500 to 3000 psi is used to produce nanopores. The physical, electrical, and chemical properties of the films are characterized. We have observed a decrease in the dielectric constant of 40% in an amorphous silicon carbide film with CO_2 treatment at 200°C followed by 400°C annealing. For PMMA, we find a change of approximately 15% for CO_2 processing at room temperature.

9:40 AM Break

10:00 AM

Thermally Stimulated Luminescence in Electroluminescent Pi-Conjugated Polymers: *Andrey K. Kadashchuk*[1]; Vladimir I. Arkhipov[2]; Heinz Bässler[2]; [1]National Academy of Science, Inst. of Phys., Prospekt Nauki, 46, Kiev 03028 Ukraine; [2]Philipps–Universität Marburg, Inst. of Physl., Nuclear & Macromolecular Chem. & Matl. Sci. Ctr., Hans-Meerwein-Strasse, Marburg D-35032 Germany

Low-temperature photo-induced thermally stimulated luminescence (TSL) was studied for the first time in electroluminescent pi-conjugated polymers such as methyl-substituted ladder-type poly(paraphenylene) (MeLPPP) [1], some novel substituted PPVs and polyfluorene, as well as in vapor deposited Alq3 films. The obtained data are interpreted in terms of the thermally assisted hopping model of TSL [1,2] we recently developed. Analysis of the obtained TSL data allows evaluation of the width of the density-of-states (DOS) distribution, which agrees well with that determined from charge transport studies, and elucidates important details of geminate recombination in these materials. Possible mechanisms of charge carrier photogeneration and origin of charge carrier traps in PPV-based polymers are discussed. [1] A. Kadashchuk, Yu. Skryshevski, A. Vaknin, N. Ostapenko, E. V. Emelianova, V. I. Arkhipov, H. Bässler, Phys. Rev. B. 63, 115205 (2001). [2] V. I. Arkhipov, E. V. Emelianova, H. Bässler, A. Kadashchuk, Chem. Phys. (in press).

10:20 AM

Electrical Property Characterization of Polymer Nanocomposites: *Jung-il Hong*[1]; Petra Winberg[1]; Dongling Ma[1]; Linda S. Schadler[1]; Richard W. Siegel[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Eng., 110 Eighth St., Troy, NY 12180 USA

The electrical properties of polymer nanocomposites are expected to be different from conventional polymer composites due to the small size and high surface area of the filler nanoparticles. Low-density polyethylene/ZnO and ethylene-propylene-diene monomer (EPDM) synthetic rubber/ZnO nanocomposites with various filler contents were prepared by mechanically mixing 50 nm nanoparticles with polyethyelene and EPDM rubber. The electrical properties such as permittivity, resistivity (as a function of field) and breakdown strength were measured at several volume fractions of filler and at several temperatures. The results were compared with the properties of composites filled with micron-size ZnO particles. It was found that the permittivity is not affected by the size of the filler particle, while the resistivity increases with smaller particle size; resistivity is generally more affected by the properties of the interface between matrix and filler particles. The breakdown strength of the composite is also found to increase with nanoparticle fillers as compared to micron-size fillers. This research is supported by ABB.

10:40 AM

Effect of Heat Treatment in Electrical Field on the Properties of Polyethylene Filled with Carbon Black: *Vaclav Bouda*[1]; Jiri Chladek[1]; Jana Mikesova[2]; Josef Sedlacek[1]; [1]Czech Technical University in Prague, Dept. Mechl. & Matls. Sci., Technicka 2, Prague 166 27 Czech Republic; [2]Institute of Macromolecular Chemistry, Heyrovskeho nam. 2, Prague 162 06 Czech Republic

Resistivity of polyethylene that has a lower carbon black (CB) concentration than the theoretical percolation threshold shows a sharp step increase in conductivity when the sample is held for a some time in alternating electrical field at temperature higher than the melt temperature of the polymer matrix. The colloid theory is the best tool for the interpretation of the results. The equally charged CB-particles repel each other and form a metastable colloidal suspension in a specific range of ionic strength of polymer melt between the critical dispersion concentration and critical coagulation concentration of counter-ions. The most interesting slow growth of CB-soft assembly starts at "soft coagulation" concentration. The combination of processes is expected to be effective in the generation of complex carbonaceous structures.

Powder Materials: Current Research & Industrial Practices: Nanostructured Materials

Sponsored by: Materials Processing & Manufacturing Division, Powder Materials Committee

Program Organizers: Fernand D.S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgical Engineering, Rapid City, SD 57701-3995 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Tuesday AM — Room: 209
November 6, 2001 — Location: Indiana Convention Center

Session Chairs: Naresh N. Thadhani, Georgia Institute of Technology, Sch. of Matls. Sci. & Eng., Atlanta, GA 30332 USA; Jan A. Puszynski, South Dakota School of Mines and Technology, Chem. & Cheml. Eng. Dept., Rapid City, SD 57701-3995 USA

8:30 AM Keynote

Advances in the Formation of Metal and Ceramic Nanopowders: *Jan A. Puszynski*[1]; [1]South Dakota School of Mines and Technology, Chem. & Cheml. Eng., 501 E. St. Joseph St., Rapid City, SD 57701 USA

The interest in nanostructured materials has been stimulated by the fact that owing to the small particle/grain size and high surface-to-volume ratio, these materials are expected to demonstrate unique mechanical, optical, electronic, and magnetic properties. Processing and manufacturing of nanostructures is one of the most challenging tasks of the materials science and engineering. This paper addresses current advances in the formation of ceramic and metallic nanopowders. The review of available synthesis methods with the emphasis on critical technical aspects, including scale-up, will be presented as well.

9:10 AM

Nanotube/Zirconia Materials from Powder Materials Tape Casting Routes: *Enrique V. Barrera*[1]; Leonard Yowell[1]; [1]Rice University, Dept. of Mechl. Eng. & Matls. Sci., MS-321, Houston, TX 77005 USA

Ceramic tapes made from yttria stabilized zirconia have been processed with dispersed nanotubes for thermal conductivity enhancement. Carbon single wall nanotubes are nanosize fibers with highly anisotropic thermal properties. Nanotubes have high thermal conductivity like diamond in the fiber direction and are insulators in the transverse direction. We have determined that low amounts of nanotubes dispersed in zirconia can lead to significant reductions in thermal conductivity for temperatures measured as high as 1400C. Our processing has led to stable nanotubes being processed in tapes for a range of temperatures and heat treatment times. Portion of our work has focused on nanotubes serving as a template for stable porosity formation. This research has been supported by the Office of Naval Research under grant no. N00014-99-1-0246. Thermal conductivity measurements were conducted at Oak Ridge National Laboratory.

9:35 AM

Nanophase and Ultrafine-Grained Powders Prepared by Mechanical Milling: Consolidation to Full Density and Unusual Mechanical Behavior: *Evan Ma*[1]; [1]Johns Hopkins University, Matls. Sci. & Eng., 3400 N. Charles, Baltimore, MD 21218 USA

Constrained sinter-forging has been used to achieve full density while maintaining nanophase grain sizes for powders prepared by mechanical milling, including elemental metals, intermetallics, and two-phase composites, with potential for near net-shape manufacturing. The addition of a second phase, especially when alloyed into the matrix uniformly during milling, facilitates the retention of nanostructures in consolidation. The versatile, solid-state, milling/consolidation approach leads to easy preparation of extremely uniform nanophase composites with multiple components having very different melting points and properties. Under uniaxial compression, very high strength and appreciable elongation to failure have been observed in the consolidated samples, reflecting the high density and nanocrystalline microstructure in these samples. Under tensile forces, however, the residual processing flaws may trigger premature catastrophic failure, and the high-strength material is susceptible to plasticity instabilities. In this regard, ultrafine-grained microstructure (e.g., submicron grains), with grain sizes still at least a couple of orders of magnitude finer than conventional materials, can offer a balance for the desired combination of strength and ductility and at the same time be more amenable to full density processing. Unusual deformation modes, such as shear banding as the dominant deformation mechanism from the onset of plastic deformation, have been observed. Such a behavior is very different from those in conventional metals. It is not only of interest to scientific studies of the dependence of deformation mechanisms on grain size but also of practical value in certain high-rate applications such as kinetic energy penetrators. Our systematic characterization indicates that the shear localization is promoted by the diminishing strain hardening and strain rate hardening in these high-strength materials. Such mechanical responses resemble those of metallic amorphous alloys. BCC Fe alloys, which typically exhibit obvious strain hardening and strain rate hardening at large grain sizes, will be analyzed in this talk to illustrate these points.

10:00 AM Break

10:20 AM
Mechanical Behavior of Nano- and Submicron-Grained Titanium Aluminide/Titanium Silicide Composites Prepared by High Energy Milling: *Rainer Bohn*[1]; Georg Fanta[1]; Thomas Klassen[1]; Rüdiger Bormann[2]; [1]GKSS Research Center Geesthacht GmbH, Inst. for Matls. Rsrch., Max-Planck-Strasse, Geesthacht D-21502 Germany; [2]Technical University Hamburg–Harburg, AB 5-06, Matls., Phys. & Tech, Eißendorfer Strasse 42, Hamburg D-21073 Germany

This study was launched to systematically investigate the mechanical properties of intermetallic/ceramic composites with grain sizes in the nano- and submicron range. As model systems, gamma TiAl-based matrices with varying amounts of finely dispersed Ti5Si3 particles were chosen. Dense and highly clean composite material was prepared by high energy milling and subsequent hot isostatic pressing. At room temperature, the grain size dependence of hardness and yield strength can be described by the well-known Hall-Petch relationship. Contrary to the behavior of conventional alloys, the ductility of submicron-grained composites drops if the grain size is further reduced.This is attributed to arising difficulties evolving for deformation by single dislocation glide. In the high temperature range, the flow stress is strongly reduced. Superplastic deformation becomes feasible already at 800°C, allowing for easy forming of parts. The mechanisms of deformation are similar to those established for coarse-grained materials at temperatures ≥ 1000°C. Via a recrystallization treatment, the fine globular microstructure can be transformed into a coarse-grained lamellar microstructure with advanced application properties.

10:45 AM
Metal Reduction using Photocatalytic Characteristics of Homogeneously Precipitated TiO2 Nano-Sized Powders: *Whungwhoe Kim*[1]; Sunjae Kim[1]; [1]KAERI, Nuclear Matl. Dvpt. Grp., Yousung-gu Ducjin dong, Teajon 305-353 Korea

Photocatalytic characteristics of nano-sized TiO2 powder produced by homogeneous precipitation process (HPP) were compared with those of commercial Degussa P-25 powder, eliminating metal ions such as Pb and Cu from aqueous equi-molar metal-EDTA solutions. In aqueous Pb-EDTA solution, the TiO2 powder by HPP showed 3.5 times higher and 1.5 times faster in initial adsorption and complete elimination of Pb ions, respectively, than Degussa P-25 powder did. The TiO2 powder by HPP also showed higher initial adsorption and faster elimination rate of Cu ion than Degussa P-25 powder in aqueous Cu-EDTA solution, similarly to in the aqueous Pb-EDTA solution. It was thought that the photocatalytic properties are enhanced with increasing specific surface area and that the TiO2 powder by HPP, which consists of primary particle size of 20nm coagulated with chestnut bur shape, has larger specific surface area (~170m2/g) than that (40-70m2/g) of Degussa P-25.

11:10 AM
Nanotube Recovery: A Method of Recycling Nanotubes from Polymeric Nanocomposites: *Fernando Rodríguez-Macías*[1]; Felipe Chibante[2]; Fernand Marquis[3]; Enrique V. Barrera[1]; [1]Rice University, Dept. of Mechl. Eng. & Matls. Sci., Houston, TX 77005 USA; [2]NanoTechnologies of Texas, Inc., Houston, TX 77081 USA; [3]South Dakota School of Mines and Technology, Dept. of Matls. Eng., Rapid City, SD 57701 USA

Even though single wall nanotube production is on the rise, methods of recovering nanotubes from nanocomposites gives us reusable material and the opportunity to further evaluate their durability to various processing conditions. In this work, HiPco single wall nanotubes are removed from a polypropylene that had a melt flow index of 1000. The nanotubes were characterized for residual polymer and other residuals and features signifying disruption of the cage structure. The study also evaluated whether the starting impurities in the HiPco nanotubes were subsequently removed, in part, due to the mechanical processing in the nanocomposite formation. Our ability to recycle nanotubes will have a long term impact from the standpoint of the environment and with regard to their availability to the research and commercial markets. This research is supported by the Welch Foundation

11:35 AM Invited
Equal Channel Angular Extrusion for Processing Materials with an Ultrafine Grain Structure and for Consolidating Powder Processed Nanostructured Metals: *V. Provenzano*[1]; [1]NIST, Magnetic Matls. Grp., Gaithersburg, MD 20877 USA

Severe plastic deformation through equal channel angular extrusion (ECAE) is an innovative and effective method for processing metallic-based materials. Also, this method is quite versatile being that it can be used both to refine the microstructure of metals and alloys down to the nanoscale dimensions as well as it can be effectively employed to consolidate powder processed nanostructured metallic-based materials to their full density, while avoiding the major drawbacks that are commonly associated with conventional sintering processing, such as the introduction of contamination products, defects, and incomplete densification. In this paper we shall first review the basic principles of equal channel angular pressing together with some of the tangible benefits that be derived from this method in terms of ultrafine microstructure and enhanced mechanical properties. As a way of illustrating the processing principles and the resulting properties and structure, property data and microstructure details will be presented for a number of metals and alloys that had been processed by ECAE method. These data will show that ECAE processing is capable of producing materials with novel and enhanced mechanical properties, as for example low temperature superplastic behavior observed in some aluminum alloys, increased low temperature ductility observed in some intermetallics and significantly enhanced tensile strength. Finally, a number of examples will be presented to illustrate how the ECAE method was successfully used to fully consolidate nanostructured metals that had been produced by powder processing routes.

Affordable Metal-Matrix Composites for High Performance Applications - III: Fatigue, Fracture and Creep of MMCs - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: Awadh B. Pandey, Pratt & Whitney, Liquid Space Propulsion, West Palm Beach, FL 33410-9600 USA; Kevin L. Kendig, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Watson, Pratt & Whitney, East Hartford, CT 06108 USA

Tuesday PM — Room: 139
November 6, 2001 — Location: Indiana Convention Center

Session Chair: Thomas J. Watson, Pratt & Whitney, E. Hartford, CT 06108 USA

2:00 PM Invited
Creep Behavior of Powder Metallurgy SiC-Al Composites and their Al Matrices: *Farghalli A. Mohamed*[1]; [1]University of California–Irvine, Dept. of Cheml. & Biocheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

A systematic comparison between the creep characteristics of discontinuous metal matrix composites and those of their unreinforced matrix alloys under similar experimental conditions is of practical and scientific significance. From a practical point of view, such a comparison is essential to the process of assessing the potential of these composites for use as structural materials for high temperature applications. From a scientific point of view, information obtained from such a comparison is critical to the identification of various processes that may control the creep behavior of the composite. At present, there are several sets of creep data on Powder metallurgy (PM) discontinuous SiC-Al composites and their unreinforced PM Al matrices. A close comparison between the data of the composites and those of the alloys has led to significant findings. These findings are reviewed and discussed with emphasis on: (a) the source of the anomalous values of the stress dependence of creep rate and the temperature dependence of creep rate, (b) the role played by both the reinforcement and the matrix alloy during the creep of the composite, (c) the characteristics and nature of the deformation processes controlling creep behavior, (d) the details of creep substructure in both the composites and the matrix alloys (e) the origin of creep strengthening in SiC-Al composites, and (f) the reason of the loss of creep strengthening in some SiC-Al composites at high strain rates.

2:30 PM
The Effect of Zirconia Particulate Reinforcements on Superalloy Creep Behavior: *R. K. Oruganti*[1]; A. K. Ghosh[1]; [1]University of Michigan, Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48105 USA

Graded and layered interfaces have been proposed as a means to minimize rapid stress buildup induced by abrupt changes in the thermal expansion coefficient between a ceramic barrier coating and a metallic substrate. Such approaches are suggested for turbine blades and hot structures, however the effect of grading of ceramic particle distribution on the creep response of metallic matrices (e.g. Ni base alloy) is not clearly known. To understand the effect of these compositional modifications in the case of oxide particulate reinforced materials, composite samples of Rene’ 95 as model superalloy matrix and partially-stabilized zirconia as thermal barrier material, were prepared by the powder metallurgy route. TEM and XRD examination showed apparent dissolution of g’ (Ni3Al) phase near the ZrO2 particles where a sparse distribution of Al2O3 particles formed due to chemical reaction between ZrO2 and Ni3Al. The creep response of these composites, relative to that of the matrix, is characterized by a strengthening effect at low stresses, which increases with increasing particulate content. However, the composites exhibit weakening at high stresses relative to the matrix. It is believed that this weakening at high stresses is related to the loss of densely packed g’ phase, which provides the primary strengthening contribution. Modified chemical composition in this region raises the stress exponent, n, for the composite, permitting initiation of glide/climb at high stresses aided by a larger spacing between the Al2O3 particles.

2:50 PM
High Cycle Fatigue Strength Improvement of Titanium Matrix Composites by Residual Stress Modification: *Joachim Hausmann*[1]; Christoph Leyens[1]; Jeorg Hemptenmacher[1]; Wolfgang A. Kaysser[1]; [1]DLR, German Aeros. Ctr., Linder Hoehe, Cologne 51170 Germany

The longitudinal fully reversed high cycle fatigue strength of continuous silicon carbide fiber reinforced titanium matrix composites (TMCs) in the as processed condition is slightly above or same as the neat matrix material. This is predominantly caused by premature matrix cracking due to high thermal residual tensile stresses in the matrix. A well defined modification of residual stresses in the matrix by prestraining of TMC specimens leads to a significant improvement of the HCF-strength of about 30% compared to specimens in the as processed condition. The way of optimization is simulated by finite element modeling. Experimental results with the system SCS-6/Ti-6-2-4-2 confirm the effectiveness of residual stress modification.

3:10 PM Invited
Effect of Reinforcement Particle Morphology on the Tensile Response of 6061/SiC/20p Discontinuously-Reinforced Aluminum: *Jonathan Edward Spowart*[1]; [1]UES, Inc., AFRL/MLLM, Bldg. 655, 2230 Tenth St., Ste.1, Wright-Patterson AFB, OH 45433 USA

In order to study the effect of particle morphology on the tensile response of Discontinuously-Reinforced Aluminum (DRA), two P/M 6061/SiC/20p materials were fabricated using established powder blending, compaction and extrusion techniques. One of the materials contained abrasive-grade SiC (F-600) whilst the second material was fabricated using “rounded grain” (RG) SiC product. Care was taken to ensure that each material contained the same size and volume fraction of SiC particles, and to ensure that each material experienced an identical processing route. Mechanical testing was completed at ambient, elevated and cryogenic temperatures, in order to measure the effect of particle morphology on both the elastic and plastic tensile response of the DRA. Extensive microstructural and fractographic analyses were also carried out on the as-processed and as-tested specimens, using optical and electron microscopy. Analytical and numerical modeling was then used to relate the observed mechanical behavior to microstructural differences between the two materials.

3:40 PM Break

3:55 PM Invited
Fracture Toughness and R-Curve Behavior of Particle Reinforced Metal Matrix Composites: *Bhaskar S. Majumdar*[1]; [1]New Mexico Tech, Dept. of Matls. & Metall. Eng., Socorro, NM 87801 USA

Fracture toughness is a key parameter in the design of metal matrix composites (MMCs). In our work, we have used different ASTM procedures to estimate the crack initiation toughness and R-curve behavior for SiC-particle reinforced aluminum matrix composites, with both macroscopically homogeneous and laminated microstructures. Although the initiation toughness was similar, the crack resistance behavior varied over a wide range, depending upon the technique used to analyze the load-displacement-crack length data. Analysis showed that the cracks essentially grew under small-scale yielding (SSY) conditions, implying that the R-curve should plateau within a very short increment of the initially sharp fatigue crack. Results are presented, along with modeling of the toughness based on the microstructural parameters. The implication of crack growth under SSY conditions is further analyzed in the context of development of many brittle systems where an R-curve behavior is suggested as a remedy for low crack initiation toughness. It is argued that if SSY conditions are operative, and if crack wake shielding (such as bridging) is small, then R-curve effects provide little insurance against catastrophic fracture.

4:25 PM
The Tensile Deformation and Fracture Characteristics of 7034 Aluminum Alloy Metal Matrix Composite: *Meslet Al-Hajri*[1]; T. S. Srivatsan[1]; X. Gao[1]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

Development and emergence of discontinuously-reinforced metal-matrix composites has certainly created a need for a thorough and systematic understanding of microstructural effects on tensile deformation and fracture characteristics in order to develop a framework for predicting their behavior under a quasi-static load. This presentation will focus on rationalizing using principles of materials science and mechanics of solids the underlying mechanisms governing tensile deformation and fracture characteristics of a SiC particulate-reinforced 7034 aluminum alloy metal-matrix composite. The emphasis will be on understanding composite microstructural effects on strength, ductility, and failure and associated fracture modes. The damage mechanisms will be rationalized in light of concurrent and mutually interactive influences of nature of load, test temperature, deformation characteristics of the composite constituents and intrinsic composite microstructural effects.

4:45 PM
Metastable Equilibria in Al-21.5Si Alloy: *V. Mazur*[1]; A. Mazur[1]; [1]Institute for Problems of Materials Science–Kyiv, 3 Krzhizhanovskogo Str., Kyiv 03142 Ukraine

Physical, chemical and technological properties of Al-Si alloy castings are greatly improved by high temperature treatment of the melt. Similarly to alloying and modification it ensures far better mechanical properties of particular value is the improvement of both strength and plastic behavior. In hypoeutectic alloys these effects are produced by formation and dissociation (complete or partial) of metastable phases and eutectics based on them. For hypereutectic alloys, however, the microkinetics of solidification is not yet clearly understood. Therefore study into genesis of phase constituent in these industrially important alloys and into mechanism and kinetics of their solidification is a high-priority task. Solidification of the Al-21,5%(wt.)Si was studied in isothermal conditions using small graphite capsule technique. The values of preliminary heating temperature (830 and 900°C) were chosen based on the result of previous study into kinematic viscosity of Al-21,5% Si melt where critical points of violation of viscosity polytherm monotony were discovered. The temperatures of isothermal holdings was fixed at a value that was varied over the range from 530 to 830°C with pitch of 10°C. After isothermal holding for 3, 5, 7,…, 230 s the capsule was immersed in salty water with ice. Isothermal solidification diagrams were plotted from the experimental data. It was discovered that the actual process of solidification of Al-21, 5Si alloy is more complicated in nature than the one which could be visualized previously on the basis of the conventional phase equilibrium diagram for Al-Si system. It involves nucleation, growth and melting – partial or complete – of metastable phases X, ç, ù, ê and any variants of nucleation and growth of stable phases á-Al, â-Si, (Fe,Al)Si in the liquid phase which can be considered primary (starting), secondary (restructured after cooling or during holding) or tertiary (brought about by melting of metastable phases) liquid. Schematic diagrams of metastable phase equilibrium for preliminary melt heating temperatures of 830 and 900°C are shown.

5:05 PM
Low Cost Preformless Squeeze Casting: *S. M. Pickard*[1]; J. K. Kim[1]; J. C. Withers[1]; R. O. Loutfy[1]; [1]MER Corporation, 7960 S. Kolb Rd., Tucson, AZ 85706 USA

MER has developed a low cost squeeze casting technique to produce high quality composites with filler composition of 40-50% Vf. The filler material can be ceramic particulate such as Si, Al2O3, B4C, fly ash, diamond, etc., as well as discontinuous or continuous fiber reinforcements. The reinforcements are added to an arm die just before hot metal. Cycle time can be in the one to four minute cycle depending on complexity, size, etc. Casting is to near net shape to minimize final machining costs. The filler material can completely fill the die to produce homogenous reinforcement or can be selectively placed to provide specific reinforcement for special wear requirements such as brake rotors. Suitable die materials, shot tube velocity, matrix alloy compositions and means of powder dispersion will be discussed. Without the need of preforming, cost analysis of the process confirms that cost can be lower than for compositing routes such as stir casting with projected cost of $1-1.50/lb depending on aluminum alloy cost and the reinforcing metal cost.

5:25 PM Withdrawn
Structure and Properties of Gas-Solid Reaction Synthesized Titanium Matrix Composite: *Hyungsik Chung*[1]; Yong-Jin Kim[2]; Yong-Hyun Kim[1]; Jae-Whan Ahn[1]; [1]Ajou University, Molecular Sci. & Tech., San 5, Wonchun-dong, Paldal-gu, Suwon, Gyunggi 442-749 Korea; [2]Korea Institute of Machinery & Materials, Matls. Eng., 66 Sangnam-dong, Changwon, Gyungnam 641-010 Korea

Applications of Computational Thermodynamics in Materials Processing - III

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee
Program Organizers: Qingyou Han, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; Ravi Vijayaraghavan, Ford Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Tuesday PM Room: 111
November 6, 2001 Location: Indiana Convention Center

Session Chairs: Qingyou Han, Oak Ridge National Laboratory, Metals & Cer. Div., Oak Ridge, TN 37831-6083 USA; Aniruddha Mukhopadhyay, Fluent, Matls. Proc. Div., Lebanon, NH 03766 USA

2:00 PM
Thermal Analysis of Secondary Refining of Steel in Ladles: *Aniruddha Mukhopadhyay*[1]; [1]FLUENT, Inc., Matls. Proc. Div., 10 Cavendish Ct., Lebanon, NH 03766 USA

In the steel industry, ladles are used to transport liquid iron and steel between various processing stations, for example, BOF, Secondary Steel Refining Shop, and the Continuous Caster (CC). Between the 'tapping' of liquid metal into a ladle and before it is teemed out, there are uncertain holding periods resulting in thermal stratification in the bath due to refractory heat loss. To alleviate this problem argon is purged into the liquid steel that generates enough buoyancy and turbulence and helps regain thermal homogeneity. The argon stirring also enhances mixing in the ladle when chemicals are added. In this example, FLUENT is used to simulate the process using momentum sources to mimic the gas purging action. Phenomenological modeling of chemical additions is implemented for transient analysis of heat effects of aluminum, ferromanganese and lime additions for Al-killed steel refining process. The emphasis of this study is on validating a simple single phase CFD model that is capable of capturing all the essential features of the multiphase flow situation in the gas-stirred ladle. This model illustrate how a continuous temperature monitoring model can be constructed from 'tap' to 'teem' in a single integral modeling environment by dynamically varying transient boundary conditions.

2:25 PM
Thermodynamics and Temperature Monitoring during Secondary Refinement of Liquid Steel: *Aniruddha Mukhopadhyay*[1]; [1]Fluent, Inc., Matls. Proc. Div., 10 Cavendish Ct., Lebanon, NH 03766 USA

Chemical additions during secondary refining target to achieve desired composition of liquid steel. Often kinetics of deoxidizing and desulfurizing agents control the rate of processing. While aluminum based deoxidation of liquid steel is very fast, it involves loss of aluminum due to burning with atmospheric oxygen as aluminum floats on high temperature liquid steel. On the other hand, lime based desulfurization is a slower process. However, considering the overall cycle time per ladle, temperature and composition respond to chemical additions in relatively shorter time. In this study, a simulation tool has been developed that can effectively assess the impact of chemical additions on liquid steel temperature based on thermodynamics of the involved reactions. This approach takes care of transient process steps e.g., natural convection in the melt, during holding and fast homogenization, during purging of argon gas. Finally, this tool can be used to predict time-temperature variation in steel during the entire process of secondary steel refinement.

2:50 PM
Computational Analysis of Inclusion Distribution in Tundish and Mold: *Eung Kyu Lee*[1]; Jae Hwan Ahn[1]; Jong Kyu Yoon[1]; [1]Seoul National University, Div. of Matl. Sci. & Eng., san 56-1, Shillimdong, Kwanakgu, Seoul 151-742 Korea

As iron and steelmaking process technology develops remarkably, cleanliness of molten steel in a tundish is improved gradually through second refinement. And, it is now increasingly clear that a continuous casting tundish and mold has a far more important function as a continuous refinery than originally expected. In this study, a mathematical model was developed to predict the behavior of inclusions including the generation, growth, removal of inclusions in tundish and mold. And, based on this model, several operational conditions were calculated. To consider the complex shape of tundish and mold, body-fitted-coordinate was used. Firstly the temperature and fluid flow in the tundish were analysed. The k-å turbulence model was used. In the tundish and mold, the removal rate of inclusions by floatation was calculated with the inclusion trajectory model based on the analysed 3-D fluid flow. And when inclusions were removed by floatation only, in steady state flow, the distribution of inclusions in the tundish was analysed through the inclusion diffusion model. And, in this work, the lumped inclusion removal model with size evolution was used. This model was applied previously to investigate inclusion removal from batch type reactor. And this model, based on uniform average properties within the tundish, can consider the various phenomena (agglomeration, reoxidation, adhesion) of inclusions in the tundish. Moreover, several calculations are proceeding.

3:15 PM
Austenite-to-Ferrite Phase Transformation in Low Alloy Steels: *Ernst Gamsjäger*[1]; Franz Dieter Fischer[1]; Jiri Svoboda[2]; Christian M. Chimani[3]; [1]Montanuniversität, Institut für Mechanik, Franz-Josef-Str. 18, Leoben A-8700 Austria; [2]Academy of Sciences of the Czech Republic, Inst. of Phys. of Matls., Zizkova 22, Brno CZ-61662 Czech Republic; [3]Voest-Alpine Industrieanlagenbau GmbH, Turmstr. 44, Linz A-4031 Austria

During continuous casting low alloy steels undergo a pro-eutectoid phase transformation from the austenite to the ferrite phase at high temperatures of about 1000K and above. The gradual rearrangement of the iron-lattice (fcc to bcc) is accompanied by the diffusion of interstitially dissolved atoms like carbon and substitutionally dissolved atoms like manganese or chromium. The molar Gibbs energies of the ferrite- and the austenite-phase in the ternary system Fe-C-Mn have been calculated with the "Gibbs energy minimizer" ChemSage. The data base, obtained by this calculation, has been the starting point to evaluate the chemical driving forces in dependence on the composition and the temperature. The growth of a ferrite layer has been investigated. Contrarily to diffusion-controlled models, there are no restrictions with respect to the mole fractions of the components at the interface. Furthermore, the different processes due to diffusion of interstitially or substitutionally solved atoms are taken into consideration.

3:40 PM Break

3:50 PM
A Numerical Model of a Conventional Heat Flux Differential Scanning Calorimeter that Allows Accurate Thermophysical Data to be Extracted from Experiments: Hongbiao Dong[1]; *John D. Hunt*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

A differential scanning calorimeter (DSC) is often used to measure thermophysical properties. When a significant amount of heat is liberated at one temperature, the release of heat appears to be spread over a wide range of temperature. This is known as smearing and arises because large temperature differences exist between the sample and the measurement thermocouple and because in analyses of the technique it is usually assumed that the temperature difference between the sample and reference is proportional to the difference in heat capacity between the sample and reference. To minimise smearing DSC manufacturers recommend using very small samples but this limits the size of the signal and thus accuracy. The smearing also limits the ability of a DSC to resolve peaks that differ by a few degrees. A numerical model of a heat flux DSC has been written. The program can either be used to extract heat capacity as a function of true sample temperature (that is to remove the smearing from experimental data). Alternatively it can be used to predict how a DSC should respond to different sample enthalpy/temperature input. The program can handle any form of DSC operation including temperature modulation. The model has been used to de-smear data for alloys and pure materials measured using various different commercial apparatus. The program is written in Visual Basic and is available on request.

4:15 PM
Carbide Precipitation during Carburisation: *André Schneider*[1]; Gerhard Inden[1]; Georg Frommeyer[1]; [1]Max-Planck-Institut fuer Eisenforschung GmbH, Max-Planck-Str. 1, Duesseldorf 40237 Germany

The kinetics of carbide precipitation in iron was studied experimentally by gas carburisation in CH_4-H_2-H_2S and O-H_2-H_2O-H_2S gas mixtures at 700°C and 800°C. The mass gain kinetics was determined by thermogravimetric analysis. The carbon transfer coefficient was derived by fitting the experimental data with calculations using the software DICTRA. These mass transfer coefficients were then used to calculate the microstructural evolution of Fe-Al-C-X (X = Ti, V, Nb) alloys under carburisation conditions. In these calculations the carbides M_3C, MC, M_3C_2 and k-carbide (Fe_3AlC_x)

TUESDAY PM

were treated as dispersed particles and diffusion was assumed to take place in the matrix only.

4:40 PM
Thermodynamic Model of the Fe-Rich Corner of the Liquid Fe-Al-N-B System: *Maziar Sahba Yaghmaee*[1]; George Kaptay[1]; [1]University of Miskolc, Dept. of Physl. Chem., Egyetemvaros, Miskolc 3515 Hungary

The associate solution model has been developed to describe thermodynamic properties of the Fe-rich corners of liquid ternary Fe-Al-N and Fe-B-N systems and also the quaternary Fe-Al-N-B system. The following associates have been taken into account: FeAl, Fe2B, AlN, BN. The activity coefficients of these species were fitted from the measured solubilities and other thermodynamic properties of the binary and ternary systems. Finally, the solubilities of AlN, BN and N2 gas have been calculated as function of composition and temperature. The inadequacy of the solubility product to describe the solubilities of AlN and BN has been shown. General thermodynamic conditions of the validity of the solubility product has been established.

5:05 PM
Convex Phenomenon in Viscosity Curve of Ti-Bearing Slag: *Yuhu Xia*[1]; *Zhitong Sui*[1]; [1]Northeastern University, 119#, Shenyang, LN 110006 China

The composition of the Ti-bearing slag is a key property of controlling the distributing of components and the crystallization process of Perovskite phase. Previous study showed that to obtain a proper basicity of the slag is good for the crystallization of Perovskite. And steel slag is a very good substitute of pure CaO as additive agent which will save great cost. But the adding of steel slag will change the viscosity of the slag, which is critical to the crystallization of Perovskite. In the measuring of the viscosity, a convex was found in viscosity curve, it covers the range of the crystallization period of Perovskite. This is mainly caused by the great latent heat releasing which increasing the system temperature fairly and decreasing the system viscosity. In this paper, the relation between the volume fraction of Perovskite and time was calculated, thus the variation of latent heat and viscosity vs. time were deduced and drew a good explanation of the convex phenomenon of viscosity curve. Some other reasons may be conducted this convex were also analyzed.

Friction Stir Welding and Processing - III: Microstructure and Properties - II

Sponsored by: Materials Processing and Manufacturing Division, Shaping and Forming Committee, Texture & Anisotropy Committee
Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; Shigeo Saimoto, Queens University, Ontario K1L 3NG Canada; S. L. Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Tuesday PM — Room: 115
November 6, 2001 — Location: Indiana Convention Center

Session Chairs: David P. Field, Washington State University, Pullman, WA 99164-2920 USA; S. Lee Semiatin, Air Force Research Laboratory, Dayton, OH 45433 USA

2:00 PM
Microstructures and Properties of Friction Stir Welds on Ti-15 V-3 Al-3 Sn-3Cr: *Thomas J. Lienert*[1]; R. Beereddy[1]; W. Tang[1]; A. P. Reynolds[1]; [1]University of South Carolina, Dept. of Mechl. Eng., 300 S. Main St., Columbia, SC 29208 USA

Ti-15 V-3 Al-3 Sn-3Cr (Ti-15-3-3-3) is a metastable β alloy that offers increased fracture toughness relative to α-β alloys of similar strength. Friction stir welding (FSW) is a solid-state process that involves in-situ heating and forging to produce welded joints. Friction stir welds were produced on 0.080" thick sheets of a Ti-15-3-3-3 alloy. Microstructures of the base metal and welds were examined using optical and scanning electron microscopy, and mechanical properties were evaluated by tensile testing at room temperature. Additionally, microhardness traverses were completed across the entire weld region. Microstructures of the various weld regions will be discussed, and the mechanical properties will be reported. The microstructures of the different weld regions were found to vary in accord with the local thermo-mechanical cycle experienced.

2:25 PM
A Study of Deformation Mechanisms and Residual Stress in Titanium Alloy Friction Stir Welds: *Nikhil U. Karogal*[1]; G. B. Viswanathan[1]; Hamish L. Fraser[1]; Jim Williams[1]; Mary C. Juhas[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Friction stir welding (FSW) of Ti alloys is now a maturing process since the recent identification of appropriate tool materials have resulted in the production of reasonable lengths of sound weld. This study is aimed at understanding the complex deformation mechanisms that occur in the evolving microstructure in an attempt to ultimately control weld mechanical properties. The material under study includes both mill annealed and beta annealed Ti-6Al-4V. Microstructural characterization of the various weld regions, as compared with the base material, will be presented. Results of microtexture studies performed using Orientation Imaging Microscopy (OIM) and X-ray diffraction will also be discussed. Microhardness profiles generated across various regions the weld will be related to the microstructural results. Finally, this study will include residual stress measurements in the various weld zones and its effect on the microstructural evolution.

2:50 PM
Microstructural Analysis of 2519 Friction Stir Welds: *Richard W. Fonda*[1]; [1]Naval Research Laboratory, Washington, DC, USA

The microstructures responsible for property variations across friction stir welds of 2519 armor plating have been characterized and correlated to mechanical properties of those welds. The locations of various microstructural regimes in these friction stir welds were identified by two-dimensional microhardness maps and texture maps of the transverse cross section of these welds. The microstructures of these regions were then characterized by optical microscopy, scanning electron microscopy, and transmission electron microscopy. Crystallographic orientation mapping was used to characterize the material flow and texture variations of these regions. These results will be discussed in terms of their impact on the observed yield strength and fracture behavior of the friction stir welds.

3:15 PM Withdrawn
Neutron Diffraction Study of Residual Stresses in Friction Stir Welds: *Thomas H. Gnaeupel-Herold*[1]; [1]National Institute of Standards and Technology, Ctr. for Neutron Rsrch., 100 Bureau Dr., MS 8562, Gaithersburg, MD 20899 USA

3:40 PM Break

3:50 PM
Complex Flow Phenomena Associated with Friction-Stir Welding of Aluminum Alloys: *Sajjad H. Kazi*[1]; *Lawrence E. Murr*[1]; [1]The University of Texas at El Paso, Metall. & Matls. Eng., M 201, El Paso, TX 79968-0520 USA

Friction-stir welding (FSW) has already been demonstrated to involve dynamic recrystallization as a mechanism for solid-state plastic deformation. Studies on aluminum alloys have also revealed complex flow patterns in the microstructure of the weld. However the flow characteristics remain somewhat unexplained. In this study, microstructural features of some friction-stir welded commercial aluminum alloys are being investigated involving light metallography (LM) and transmission electron microscopy (TEM). Similar (7075/7075, 2017/2017, 5052/5052) and dissimilar (7075/5052, 2017/5052, 7075/2017) aluminum plates of 3 mm thickness have been friction-stir welded at a rotational speed of 1250 rpm and traverse speed of 1 mm/sec. Complex vortex and swirl-like structures are observed to be more prominent in the dissimilar systems. Comparisons of microhardness profiles with microstructure for friction-stir welded aluminum alloys indicate that large differences in hardness may influence the complex spiral flow pattern at the weld zone. Microstructures of dissimilar systems have been observed to be different when two dissimilar plates change their sides. In other words, for a constant tool rotation direction, the weld flow pattern and weld efficiency change when the position of the base plate changes and when a very hard aluminum alloy (140-160 VHN) is stirred with a very soft aluminum alloy (30-60 VHN) or vice-versa. This study also compares the results with Al 7075 and Al 1100 respectively. GSA Grant PF-90-018 supports this research.

4:15 PM
Mechanical Behavior of Friction Stir Welded Powder Metallurgy Aluminum Alloy and Composite: *Siddharth R. Sharma*[1]; Rajiv S. Mishra[1]; Murray W. Mahoney[2]; Kumar V. Jata[3]; [1]University of Missouri, Dept. of Metall. Eng., Rolla, MO 65409 USA; [2]Rockwell Science Center, Structl. Metals Dept., Thousand Oaks, CA 91360 USA; [3]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

Friction stir welding (FSW) is a solid state joining technique. This makes it an attractive joining method for aluminum alloys and composites produced by a powder metallurgy route. In this study, the mechanical behavior of both a powder metallurgy monolithic aluminum alloy and composite are evaluated, i.e., IN 9052XL and 7093 Al-SiC. Mini-tensile specimens were used to determine property variations in different FSW regions including the weld nugget, the partially recrystallized zone adjacent to the nugget, heat affected zone, and parent metal. A comparison of the microstructures and mechanical properties of these regions will be presented. No damage to SiC particulates was observed in the friction stirred region. The grain sizes in the nugget region of both materials also remained comparable to the fine grain parent materials. This is a critical advantage of FSW over fusion welding techniques. The support of the University of Missouri Research Board is gratefully acknowledged.

4:40 PM
Friction Stir Weld Evaluation of DH-36 and Stainless Steel Weldments: *Maria Posada*[1]; John DeLoach[1]; Anthony Reynolds[2]; Michael Skinner[3]; Jack P. Halpin[3]; [1]Naval Surface Warfare Center, Welding & ND, Code 615, 9500 MacArthur Blvd., W. Bethesda, MD 20817-5700 USA; [2]University of South Carolina, Dept. of Mechl. Eng., 300 Main St., Columbia, SC 29208 USA; [3]MTS Systems Corporation, Adv. Eng. Solutions Div., 14000 Technology Dr., Eden Prairie, MN 55346 USA

Friction stir welds were produced from ¼-inch thick DH-36 steel and 304L stainless steel. Single-sided DH-36 and both single-sided and double-sided 304L SS weldments were evaluated. During weld trials, the welding parameters were varied to develop a relationship between weld parameters and weld quality. As part of the friction stir welding process development, the microstructure and hardness profiles were evaluated as a function of weld energy. Stir-zone and heat-affected-zone microstructures of the DH-36 and 304L weldments were observed using optical microscopy and scanning electron microscopy. Unlike 304L, DH-36 experiences readily observable solid-state phase transformations during FSW. These DH-36 phase transformations resulted in three distinct zones consisting of a stir zone and 2 heat-affected zones. Hardness values decreased as the distance increased from the stir zone. Results from mechanical property testing showed higher tensile strengths in the weld region than in the base metal for both DH-36 and 304L.

5:05 PM
Characterizing Joint Efficiency in Friction Stir Welding: *Keith M. Williamson*[1]; Robert A. Hafley[2]; Eric K. Hoffman[2]; [1]Old Dominion University, Dept. of Mechl. Eng., Norfolk, VA 23529 USA; [2]NASA Langley Research Center, Metals & Thermal Struct. Branch, Struct. & Matls. Competency, MS 188A, 2 W. Reid St., Hampton, VA 23665 USA

A model is presented to describe joint efficiency in the stir welding process. The model formulates efficiencies for butt and lap joints and confirms that double-sided stirring and additional tool passes improve the joint efficiency. In the proposed model, a concept for periodic stir patterns is introduced and calculations are presented to show that these periodic patterns increase the efficiency of stir welded lap joints by a factor based on a ratio of tool displacements along an efficiency vector and the welding direction. Besides the joint efficiency model, the study investigates vertical hardness profiles in the stir zone and predicts joint strength based on an empirical relation between strength, hardness and a strain-hardening exponent.

Fundamentals of Solidification - II

Sponsored by: Materials Processing & Manufacturing Division, Solidification Committee
Program Organizers: John H. Perepezko, University of Wisconsin–Madison, Department of Materials Science & Engineering, Madison, WI 55706 USA; Christoph Beckermann, University of Iowa, Department of Mechanical Engineering, Iowa City, IA 52242-1527 USA; William J. Boettinger, National Institute of Standards & Technology, Gaithersburg, MD 20879 USA

Tuesday PM — Room: White River Ballroom
November 6, 2001 — Location: Indiana Convention Center

Session Chair: Christoph Beckermann, University of Iowa, Dept. of Mechl. Eng., Iowa City, IA 52242-1527 USA

2:00 PM Invited
A New Theory of Fibrous Monotectic Growth: *Lorenz Ratke*[1]; [1]German Aerospace Center, Inst. of Space Simulation, Linder Hoehe, Cologne 51147 Germany

Directional solidification of monotectic alloys can lead under certain conditions of growth velocity and temperature gradient in the melt to composite microstructures with a rodlike appearance. For a theoretical description most often the Jackson and Hunt model of rod eutectic growth is used. A relation between the mean rod distance R and the solidification velocity v similar to eutectics is observed generally in experiments. A comparison between theory and experiments always led to discrepancies not yet resolved. In the approach presented here we propose an additional mode of mass transport in front of the zone of coupled growth, since in our mind the main difference between monotectic and eutectic solidification is the liquid phase state of the (rod) L2 phase growing simultaneously within a nearly perfectly pure solid matrix. We assume that the thermocapillary effect causes convection at the surface of the fibrous liquid phase. This Marangoni convection induces a flow field in front of the solidification front and has a strong influence on the solute transport, depending on the local temperature gradient and the Peclet number. We developed an analytical model using a suitably modified convective diffusion equation to describe the concentration field in front of the advancing interface and also developed a numerical description of monotectic composite growth. We find analytically a new relation between R and v in the case of small Peclet numbers. The analytical theory uses some simplifications which make it tractable, but does not correctly reflect the physics. In the numerical model we solve numerically the diffusion equation coupled with the Navier-Stokes equation in the matrix phase to find the minimal undercooling for a given velocity and temperature gradient. We derive a Jackson and Hunt diagram and show that the fluid flow leads to a strong dependence of the interrod distance on the temperature gradient opposite to eutectic solidification. A comparison with the few experimental results available shows a good agreement to the theoretical predictions. Behind the solidification front the liquid L2 fibres can become Rayleigh unstable. There has been a long standing controversy on the origin of this instability. We propose a new mechanism of the fibre break up into string of pearls based upon a Mullins Sekerka analysis.

2:40 PM Invited
Phase-Field Modeling of Solidification Microstructures: *Alain Serge Karma*[1]; [1]Northeastern University, Dept. of Phys., Boston, MA 02115 USA

The phase-field approach has emerged as a powerful tool to model the formation of solidification microstructures. As is well-known, this method avoids to track the interface by making its width finite and handles automatically singular changes in its connectedness. Everything would be just wonderful if computers of today were fast enough to carry out phase-field simulations with a microscopically small, nanometer scale, interface width. Unfortunately this is not the case, not today and not any time soon. Therefore, as phase-field modelers, we are forced to compromise by choosing the interface width 10 or even 100 times larger than in nature if we want to simulate actual microstructures that form on a much larger scale in the 10-100 micron range. In this talk, I will highlight the consequences of this unavoidable constraint on the interface width that have prevented the phase-field approach from reaching its full potential as a quantitative modeling tool. For this purpose, I will focus on the concrete case of a dilute binary alloy with a realistically small solutal solid diffusivity. I will then describe a successful remedy to this problem that requires to rethink the way in which one formulates the phase field model and analyzes its thin interface limit. Finally, I will discuss applications of phase-field simulations that have yielded new fundamental insights into long standing microstructural pattern formation issues in equiaxed and directional solidification and present quantitative comparisons with experiments.

3:20 PM Break

3:30 PM Invited
Phase-Field Models of Peritectic, Eutectic and Monotectic Alloys: *Adam A. Wheeler*[1]; Britta Nestler[2]; [1]University of Southampton, Fac. of Mathl. Stud., Highfield, Southampton SO17 1BJ UK; [2]RWTH-Aachen, Foundry Inst., Intzestr. 5, Aachen D-52072 Germany

In this talk we will discuss how a multi-order parameter phase-field method can be developed to provide a general framework to model phase transitions in a peritectic, eutectic and monotectic binary alloy systems. In the case of a monotectic alloy we will describe two different phase-field models, one involving three different phase-field variables and the other using Cahn-Hilliard type equation to distinguish the two liquid phases by their concentration. We also include a generalised stress tensor in the momentum equation to model both fluid motion in both liquid phases as well as capillarity effects. This in turn allows the inclusion of Marangoni convection at the interfaces between the two liquid phases. We will present numerical simulations that exhibit a wide range of realistic phenomena including eutectic lamellae spacing selection by both competition from neighbouring lamellae and tip splitting events, preferential growth of the peritectic phase over the properitectic phase by solid diffusion, and coarsening, particle pushing and wetting in monotectic systems. We will also show that our numerical simulations for the lamella growth in eutectic alloys are consistent with the classical Jackson & Hunt theory.

4:10 PM Invited
Modeling Dendritic Growth with Fluid Flow in Three Dimensions: *Jonathan A. Dantzig*[1]; Jun-Ho Jeong[1]; Nigel D. Goldenfeld[2]; [1]University of Illinois, Mechl. & Indl. Eng., 1206 W. Green St., Urbana, IL 61801 USA; [2]University of Illinois, Phys., 211 Loomis Lab., 1110 W. Green St., Urbana, IL 61801 USA

Understanding pattern selection during dendritic solidification is an important problem for materials scientists and engineers. In this talk, computations are described using phase-field models to directly simulate dendritic growth. We employ an adaptive gridding procedure for solving the phase field equations, where high resolution is available near the interface, and more appropriate grid dimensions are used to resolve the diffusion and velocity fields. Recent work examining the role of fluid flow in the pattern selection process will also be presented. Fully three-dimensional simulations of growth of pure materials are presented, and it is demonstrated that the three dimensional aspects of the flow are essential. Parallel implementation of the code is also described.

4:50 PM Invited
The Morphological Evolution of Dendritic Microstructures during Coarsening: J. Alkemper[1]; R. Mendoza[1]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The coarsening process in systems consisting of spherical particles in a matrix has been studied extensively. In contrast, coarsening in systems that possess both positive and negative curvature, such as those present following dendritic solidification, have received less study. Recent advances in experimental technology now allow for the routine analysis of metallic microstructures in three dimensions. A method has also been developed to determine the distributions of mean and Gaussian interfacial curvature, the analogs of the particle size distribution for spherical particles. The evolution of dendritic microstructures during coarsening is analyzed. The experiments employed a directionally solidified Al-15wt.% Cu alloy. Samples were taken from this ingot and isothermally coarsened for various times. Presented are reconstructions of the three dimensional microstructures, the temporal evolution of the average mean curvature and the evolution of the distributions of both mean and Gaussian interfacial curvatures.

Lead-Free, Lead-Bearing Solders & Alternative Surface Finishes for Electronic Packaging - I: Materials, Microstructures, Alloy Design

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee
Program Organizers: C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Srini Chada, Motorola, Department APTC, Fort Lauderdale, FL 33322 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu City 00143 Taiwan; Gautam Ghosh, Northwestern University, Department of Materials Science, Evanston, IL 60208-3108 USA; Prasad Godavarti, FlipChip Technologies, Phoenix, AZ 85034 USA; Zequn Mei, Agilent Technologies, Inc., Palo Alto, CA 94304-1392 USA

Tuesday PM	Room: 202
November 6, 2001	Location: Indiana Convention Center

Session Chairs: Paul Thomas Vianco, Sandia National Laboratories, Dept. 1835, Albuquerque, NM 87185 USA; Laura J. Turbini, University of Toronto, Ctr. for Microelec. Assembly & Pkgg., Toronto, ON M5S 3E4 Canada

2:00 PM Opening Remarks

2:05 PM Invited
Synchrotron Radiation Study of Sn Whisker Growth on Leadframe with Pb-Free Surface Finish: T. Y. Lee[1]; W. J. Choi[1]; *K. N. Tu*[1]; N. Tamura[2]; R. S. Celestre[2]; A. A. MacDowell[2]; Y. Y. Bong[3]; Luu Nguyen[3]; [1]UCLA, Dept. of Matls. Sci. & Eng., Los Angeles, CA 90095 USA; [2]LBNL, Adv. Light Source, Berkeley, CA 94720 USA; [3]National Semiconductor Corporation, Santa Clara, CA 95051 USA

The growth of a Sn whisker of length over 300 micron on a leadframe surface is a reliability issue in electronic packaging. It can connect two legs of the leadframe and becomes a short. This is a concern for the use of eutectic SnCu (0.7 atomic % Cu) solder as leadframe surface finish. We have performed synchrotron radiation micro-diffraction analysis of Sn whiskers grown on the finish of eutectic SnCu and pure Sn. Using the measured lattice parameters of Sn whiskers as references of stress-free Sn, we found that the matrix of the finish at the root of the whisker is under compression. The whisker morphology, orientation relationship between the whisker and the grains in the finish, stress distribution, whisker growth kinetics, and the effect of Cu on whisker growth will be presented.

2:35 PM
Understanding Whisker Phenomenon: *Chen Xu*[1]; Chonglun Fan[1]; Anna Vysotskaya[1]; Joseph A. Abys[1]; Yun Zhang[1]; Leslie Hopkins[2]; Fred Stevie[3]; [1]Lucent Technologies, EC&S, 236 Richmond Valley Rd., Staten Island, NY 10309 USA; [2]Lucent Technologies, Bell Labs., Murray Hill, NJ 07974 USA; [3]Lucent Technologies Microelectronics, Orlando, FL 32819 USA

The electronic industry is under extreme pressure to remove tin-lead solders from electronic components. Pure tin is one of the alternatives and may be the simplest system as a drop-in replacement. However, fear of whiskers has been a major concern inhibiting its implementation. This is because that tin whiskers, with a length from a few micrometers to several millimeters, may grow from electroplated Sn and cause electric shorts in electronic devices, particularly, in fine-pitch high I/O (input/output) components. In this paper, we will describe our systematic, and in-depth investigation of various factors effecting whisker growth, and the dynamic nature of the whisker growth phenomenon. We will also offer our understanding of the competitive nature of various whisker growth mechanisms and what set of parameters determine a particular mechanism. This understanding provides the foundation for the recommendations that we offer to the electronic industry.

2:55 PM
The Kinetics of Microstructural Coarsening in Sn-Pb Eutectic Solder: *Paul Thomas Vianco*[1]; Jerome Rejent[1]; Gary Zender[1]; Alice Kilgo[1]; [1]Sandia National Laboratories, Dept. 1835, PO Box 5800, MS1411, Albuquerque, NM 87185-1411 USA

The mechanical properties of Sn-Pb solder depends upon the response of its physical metallurgy to temperature and load environments. Understanding the microstructural coarsening kinetics for 63Sn-37Pb eutectic solder (Sn-Pb) will better define constitutive models for this material and lend insight for developing microstructurally-based constitutive relationships for Sn-based, Pb-free solders. Microstructural coarsening kinetics were determined for Sn-Pb solder from samples fabricated under well-controlled cooling rates (1, 10, and 100°/min). Quantitative image analysis determined the size distribution of Pb-rich phase particles as a function of aging. The coarsening kinetics were represented by a time power law/ Arrhenius temperature law. The time exponenets were from 0.2 to 0.4; the apparent activation energy values were from 20 to 30 kJ/mol. The implications of temperature dependence by the kinetics parmeters will be discussed. Sandia is operated by Sandia Corp., a Lockheed-Martin Company, for the USDOE under contract DE-AC04-94AL85000.

3:15 PM
Effect of Stress on the Electrical Resistivity of Solder: *Deborah D.L. Chung*[1]; Taejin Kim[1]; [1]University at Buffalo, Dept. Mechl. & Aeros. Eng., The State University of New York, Buffalo, NY 14260-4400 USA

Because solder encounters thermal and mechanical stresses during use in a soldered joint, the effect of stress on the electrical resistivity of solder deserves investigation. Previous work emphasized the effect of stress on the joint much more than that on the solder itself. Concerning the latter, previous work addressed the mechanical and thermomechanical behavior, but not the electrical behavior, which is relevant to the use of solder as an interconnection material. Electrical measurement is nondestructive and is thus attractive for monitoring in real time during cyclic loading. Real-time monitoring enables observation of both reversible and irreversible effects. We observed that the electrical resistivity of tin-lead eutectic solder increased upon tension. The effect was partially reversible. The fractional change in resistance per unit strain was 60. The irreversible part of the effect was due to plastic deformation.

3:35 PM Break

3:55 PM Invited
Design of Micro-Soldering Materials using Thermodynamic Database: Xingjun Liu[1]; *Shuanglin Chen*[2]; I. Ohnuma[1]; R. Kainuma[1]; K. Ishida[1]; Y. A. Chang[3]; [1]Tohoku University, Dept. of Matls. Sci., Grad. Sch. of Eng., Sendai 980-8579 Japan; [2]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA; [3]University of Wisconsin, Dept. of Matls. Sci. & Eng., Madison, WI 53706 USA

A thermodynamic tool, ADAMIS (Alloy Database for Micro-Solder), has been developed for designing micro-soldering materials. ADAMIS is a Windows-based program, which combines a thermodynamic database (Ag, Bi, Cu, In, Sb, Sn, Zn and Pb) with phase diagram calculation software. It has a user-friendly interface with ability to calculate the liquidus projection, isothermal and isopleth sectional diagrams, thermodynamic properties, etc. It also has functions to calculate surface tension and viscosity of liquid phase. Examples on using ADAMIS to assist design of micro-soldering alloys and Pb-free solders in the packaging of electronic devices will be presented.

4:25 PM
Examining the Nature and Morphology of Conductive Anodic Filament Formation: *Laura J. Turbini*[1]; W. Jud Ready[2]; [1]University of Toronto, Ctr. for Microelect. Assembly & Pkgg., 184 College St., Rm. 150B, Toronto, ON M5S 3E4 Canada; [2]Microcoating Technologies, 5315 Peachtree Industrial Blvd., Chamblee, GA 30341 USA

Conductive anodic filament (CAF) is a failure mode in printed wiring boards first identified by researchers at Bell Laboratories in 1976. It occurs under high voltage gradient and high humidity conditions. CAF involves the electrochemical formation of a copper-containing filament, subsurface along the epoxy-glass interface, growing from anode to cathode. Catastrophic field failures in multilayer boards have been linked to this failure mechanism. CAF is enhanced by certain soldering chemicals, in particular those used in water soluble fluxes, and hot air solder leveling (HASL) fluids. The morphology of CAF formation differs among control boards that have not been processed with water soluble flux, boards processed with flux containing polyethylene propylene glycol, and boards processed with flux containing a linear aliphatic polyether. The variation in these results will be presented and discussed. Finally, transmission electron microscopy data, used to determine the chemical nature of one of the filaments, will be presented.

4:45 PM Withdrawn
Study of the Effect of Lead on Lead-Free Alloys: A Comparison of the Viable Alloys: *David K. Suraski*[1]; [1]AIM, 25 Kenney Dr., Cranston, RI 02920 USA

5:05 PM
Tin-Bismuth-The New Plating of Choice for Leadframe Components?: *Marc Dittes*[1]; [1]Infineon Technologies AG, CPD AIT LP, Wernerwerkstr. 2, Regensburg 93049 Germany

In terms of lead-free soldering lead-free terminations of semiconductor devices are strongly demanded–especially when the advantage of elevated service life tempearature of lead-free solders is aimed at. One option for post mold solder plating of leadframe based components is an Sn-Bi-alloy. But Bi is often reported to be not compatible to current SnPb-solders and therefore not accepted. Hence, SnBi-plating shall be introduced in terms of processability, properties, requirements and limitations. The limits in terms of solderability and cracking are identified. Also the tendency to whisker formation and the influence of base metal on this issue will be presented. Additionally board level reliability trials have been perfomed and will be reported with the question of compatibility to lead-bearing and lead-free alloys in focus.

Materials Design Approaches and Experiences - III

Sponsored by: Structural Materials Division, High Temperature Alloys Committee
Program Organizers: Ji-Cheng Zhao, GE Corporate Research & Development, Physical Metallurgy Laboratory, Schenectady, NY 12301 USA; Michael G. Fahrmann, Special Metals Corporation, Technology Department, Huntington, WV 25705-1771 USA; Tresa M. Pollock, University of Michigan, Materials Science and Engineering Department, Ann Arbor, MI 48109-2136 USA

Tuesday PM — Room: 204
November 6, 2001 — Location: Indiana Convention Center

Session Chair: Frank Zanner, ZanTek Enterprises

2:00 PM Invited
Subs, Coins, Pipes, Jets, Epees, Golf Clubs, PCs, LEM and Chicken Gizzards: *Raymond F. Decker*[1]; [1]Thixomat, Inc., 620 Technology Dr., Ann Arbor, MI 48108 USA

Some development history of Inconel alloy 600, gamma prime hardening of superalloys, Ductile Iron, Inconel alloy 713C, Maraging Steels, Inconel alloy 690, US coins, Mechanical Alloying and Thixomolding will be elaborated. Observations will then be gleaned on market targeting, ultimate end uses, time for commercialization, creativity funding, dependence on ROI analyses, critical screening tests and experimental techniques of the inventors.

2:45 PM Invited
Computational Systems Design of Materials: *G. B. Olson*[1]; [1]Northwestern University/QuesTek, MSE, 2225 N. Campus Dr., Evanston, IL 60208 USA

A systems approach integrates processing, structure, property and performance relations in the computational design of materials as dynamic multilevel structures. For high-performance alloy steels, numerical implementation of materials science, applied mechanics and quantum physics principles provides a hierarchy of computational models defining subsystem design parameters that are integrated through multicomponent computational thermodynamics and kinetics. Designed properties combine strength, toughness, and impurity embrittlement resistance. Calibration of precipitation-strengthening models via atomic resolution metallography has allowed quantitative design of nanostructured steels for ultrahigh strength, and a new class of ultrahard case-hardening nanosteels is being marketed by QuesTek for a range of applications. The design approach is now being applied to Ni-base and Al-base alloys, and extended to the acceleration of the full materials development and qualification cycle.

3:15 PM Invited
Grain Boundary and Microstructure Design of Steel: *Clyde L. Briant*[1]; [1]Brown University, Div. of Eng., 182 Hope St., Providence, RI 02912 USA

The microstructural design of steel has been examined for many years. Early studies focused on the various carbides that formed in the microstructure and their role in determining the mechanical properties. Later studies considered the effect of grain boundary composition on the embrittlement and fracture of steels. In particular, it was shown that impurity elements could have major effects on both low temperature and high temperature failures in steels. Thus it was important to design and process materials in such a way that segregation of harmful impurities did not occur. More recently new techniques, such as orientation imaging, have allowed us to consider a more detailed picture of the microstructure and to design microstructures that consider such features as texture and atomic grain boundary structure. This approach requires that we integrate much of the previous work with this new knowledge. This paper will present a series of examples from ferrous metallurgy where these ideas have been used to design new materials.

3:45 PM Invited
Advances in Modeling Grain Growth and Recrystallization: *Elizabeth A. Holm*[1]; [1]Sandia National Laboratories, Matls. & Proc. Modlg., PO Box 5800, MS 1411, Albuquerque, NM 87185-1411 USA

Because microstructure mediates the properties of polycrystalline materials, understanding and controlling microstructural development are critical to alloy and process design. Advances in computational modeling of microstructural evolution allow a new level of microstructural complexity to be examined. In this talk, we review discrete, mesoscale computer simulations for grain growth and recrystallization in single-phase and multiphase systems under a variety of processing conditions, including variable temperature and mechanical strain. These simulations enable evaluation of proposed methods and mechanisms for tailoring grain size, morphology, and boundary types. Microstructures generated by these models may be used in subsequent materials response simulations. The limitations and needs of microstructural models will also be presented.

4:15 PM Break

4:35 PM
Development of High Strength Martensitic Microalloyed Steel for Heavy-Duty Engine Connecting Rod: *Young Sang Ko*[1]; Hyounsoo Park[1]; Sung Do Wang[2]; Seung Cheal Jung[1]; [1]Hyundai Motor Company, Adv. Rsrch. Ctr., 772-1, Changduk-Ri, Namyang-Myun, Whasung-Si, Kyunggi-Do 445-850 Korea; [2]KIA Steel Co., Ltd., R&D Ctr., 1-6, Soryong-Dong, Kunsan-Si, Cheollabukdo 573-400 Korea

Newly developed martensitic microalloyed steel, targeted to heavy-duty engine connecting rod, is designed to maximize strength and toughness from the effects of alloy element addition, esp. with B, Nb and Mn in low carbon steel matrix. We tailored optimum alloy combinations of 0.12C-1.0Mn-0.04Nb-0.002B steels among six different blends to make sure the best mechanical properties. The correlation of manufacturing variables and microstructures of forged samples has been evaluated by using thermomechanical simulator. We proposed the optimum forging parameters such as, temperature, time and forging pressure to obtain the desirable precipitation hardening and grain refinement during dynamic recrystallization. Consequently it is found that our new alloyed connecting rods exhibit 20% of increase in fatigue limit compared to the conventional steel connecting rod. Therefore, the new alloyed connecting rod can be more compact sized, which in turn, enables better fuel economy as well as improved engine performance.

4:55 PM
Advances in the Use of Stress Relaxation Data for Alloy Optimization and Design: *David A. Woodford*[1]; [1]MPA, Inc, 1707 Garden St., Santa Barbara, CA 93101 USA

High-precision stress relaxation tests (SRT) have now been used for several years as a basis for accelerated creep strength evaluation. In tests lasting less than one day a creep rate range of about five decades may be covered. The approach has been applied to materials selection, design analysis and remaining life assessment for serviced components. This presentation uses examples drawn from superalloys to demonstrate how the approach may be used to accelerate alloy development. By parameterizing the SRT results over a range of temperature it is possible to attain design stress levels in less than a week of testing. It is shown how the test may be used to determine the effect of microstructural features such as grain size and grain orientation, thermal-mechanical exposure conditions, test specimen section size, and the effect of location within a component.

5:15 PM
Control and Modeling of Bainitic Transformation in Ductile Iron Aided by Austempering of Steel with Equivalent Chemical Composition: *Susan Farjami*[1]; *Mahmoud Nili Ahmadabadi*[1]; [1]University of Tehran, Fac. of Eng., Dept. of Metall. & Matls. Eng., Tehran, Iran

Ductile iron can be defined as a composite of steel matrix with nodular graphites as the second phase. Alloying elements such as Si and Mn are frequently added to ductile iron to prevent pearlite transformation during austempering. During solidification, one expects microsegregation of both Si and Mn, and because of the low diffusion coefficients of these substitutional elements, this segregation should persist even after long heat treating at high temperature. This makes modeling of bainitic transformation in ductile iron rather difficult. However, in steels with homogenous matrix, it is easier to study bainitic transformation than ductile iron. The main aim of this work is a comparison between mechanism and kinetics of bainitic transformation in iron and steel with the same matrix composition after austenizing and prediction and modeling of bainitic transformation in ductile iron using dilatometric data of iron and steel with the same composition.

Microstructure Modeling and Prediction During Thermomechanical Processing - III

Sponsored by: Structural Materials Division, Materials Processing & Manufacturing Division, Shaping and Forming Committee, Titanium Committee
Program Organizers: Raghavan Srinivasan, Wright State University, Department of Mechanical & Materials Engineering, Dayton, OH 45435 USA; Armand J. Beaudoin, University of Illinois at Urbana–Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Steve P. Fox, Timet Corporation, Timet Henderson Technical Laboratory, Henderson, NV 89009 USA; Zhe Jin, Reynolds Metals Company, Metallurgy Department, Chester, VA 23836-3122 USA; S. L. Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Tuesday PM — Room: 208
November 6, 2001 — Location: Indiana Convention Center

Session Chairs: Anthony Rollett, Carnegie Mellon University, Dept. of Matls. Sci. & Eng., Pittsburgh, PA 15213-3890 USA; Zhe Jin, Alcoa Technical Center, Thermomechl. Procg. & Alloy Dvpt., Alcoa Center, PA 15069 USA

2:00 PM
Investigating Strain Path Effects during Hot Working: *B. P. Wynne*[1]; G. Angella[1]; W. M. Rainforth[1]; J. H. Beynon[1]; [1]IMMPETUS (Institute for Microstructural and Mechanical Process Engineering: The University of Sheffield), Mappin St., Sheffield, S. Yorkshire S1 3JD UK

In most industrial hot working processes the material experiences significant changes in strain path. Conversely, the majority of material models used to predict loads and microstructure evolution for these processes are described in terms of equivalent stresses and strains and calibrated using linear unidirectional laboratory tests. This discrepancy often leads to inadequate final microstructure and physical property predictions. To help address this problem a test machine capable of performing either concurrent or sequential torsion and uniaxial deformation under hot working conditions has been developed. This work intends to demonstrate the potential of the machine by examining the flow behavior, deformation microstructure, recrystallization behavior, and texture development of 316L stainless steel and titanium alloy Ti-6Al-4V deformed under nontrivial strain paths. Particular attention is paid to the effect of deforming to the same level of equivalent strain via different strain paths to determine strain path sensitivity of the above mentioned parameters.

2:25 PM
Optimisation of Microstructure Development during Hot Working of Austenitic Stainless Steels using Models for Evolution of Microstructure: *Venugopal Srinivasan*[1]; V. Madurai Muthu[1]; Sardari Lal Mannan[1]; [1]Indira Gandhi Centre for Atomic Research, Matls. Tech. Div., Kalpakkam, Tamil Nadu 603 102 India

The deformation behavior of 304L stainless steel was evaluated, under compression and torsion in the temperature range of 600°C to 1200°C and strain-rate range of 0.001 s^{-1} to 100 s^{-1} with the view to establishing processing-microstructure relationships during hot working. The material exhibits a dynamic recrystallisation domain (DRX) in the temperature range of 1000°C to 1200°C and strain-rate range of 0.01 s^{-1} to 5 s^{-1}, which is the optimum one for hot working. In order to control the final microstructure of the product, an analytical model for the evolution of microstructure during hot working (in DRX domain) was obtained. Using the above model, the optimum strain, strain-rate, and temperature trajectories were arrived at for obtaining a grain size of 35 μm in an extruded product. Process control parameters, such as ram velocity, die profiles and billet temperature, which achieve the optimal trajectories were calculated using a process model. Extrusion trials were conducted at optimal conditions and a good agreement with those predicted in the design stage has been achieved.

2:50 PM
Modelling of Microstructural Development during the Warm Rolling of LC and IF Steels: *A. O. Humphreys*[1]; D. S. Liu[1]; S. Serajzadeh[1]; J. J. Jonas[1]; [1]McGill University, Dept. of Metall. Eng., Montreal, Canada

Warm rolling has been shown to be a cost-effective method of producing certain sheet steels. However, in the presence of interstitial solute atoms, the rate sensitivity of the material can be significantly affected. This can influence the microstructural development during processing, particularly shear band formation and the subsequent development of the annealing texture, resulting in products with poor formability. The rate sensitivities of various LC and IF steels were measured; these are compared to the corresponding warm-rolled microstructures. A model is proposed that should enable warm rolled LC steel sheets to be produced with good formability.

3:15 PM
Formation of Aligned Microstructure in Steels and Recrystallization Behavior: *Kei-ichi Maruta*[1]; Michio Shimotomai[1]; [1]Kawasaki Steel Corporation, Techl. Rsrch. Labs., Kawasaki-cho, Chuo-ku, Chiba 260-0835 Japan

Application of magnetic field during the transformation in steels was found to yield two-phase microstructures aligned along the direction of the magnetic field. The developed structures were columnar or chain-like depending on the extent of the transformation. When the aligned microstructures were deformed by cold-rolling or compression, many nucleation sites for recrystallization were introduced in the inhomogeneously deformed regions if the deformation is carried out along the direction of the alignment axis. Subsequently, they were subjected to annealing for recrystallization. We have succeeded to obtain fine-grained steels without heavy deformation.

3:40 PM Break

3:55 PM
Precipitation Kinetics in Nb-V-Ti Microalloyed HSLA Steels: *S. G. Hong*[1]; K. J. Lee[2]; K. B. Kang[3]; C. G. Park[1]; [1]Pohang University of Science & Technology, Dept. of Matl. Sci. & Eng., Ctr. for Adv. Aeros. Matls., Pohang 790-784 Korea; [2]Hanyang University, Div. of Matl. Sci. & Eng., Seoul 133-791 Korea; [3]Technical Research Laboratories, POSCO, Pohang 790-785 Korea

It has been recognized that mechanical properties of HSLA steels containing Nb, V and Ti are considerably influenced by precipitation of carbonitrides. During the last three decades, most of the investigations in this area have been concerned with Nb and/or V bearing steels, while relatively few studies have been carried out for Nb, V and Ti bearing HSLA steels. In addition, the quantitative analysis of fine precipitates and their roles on phase transformation and mechanical properties are still controversial. In the present study, the prediction of precipitation kinetics in Nb-V-Ti micro-alloyed steels has been accomplished using laboratory experiments as well as computer modeling. In order to obtain the precise information regarding precipitation kinetics, two stage interrupted compression tests were conducted with the cylindrical specimens (16mm x 20mm). Quantitative analysis for carbonitrides was also performed by measuring softening parameter and the distribution of carbonitrides using FEG-TEM. The softening parameter was obtained by measuring the area under the flow curve of an interrupted compression test. Thermodynamic and kinetic analysis is based on sub-lattice model and nucleation and growth model respectively. The experimental results have been compared with the thermodynamic and kinetic analysis.

4:20 PM
The Influence of Austenite Grain Size on Hardenability in Deformed Microalloyed Bar Steels: *Dominique A. Shepherd*[1]; [1]NIST, Metall. Div., Bldg. 223, Rm. B254, 100 Bureau Dr., MS 8553, Gaithersburg, MD 20899-8553 USA

Using deformation temperatures between 850 and 1000°C and strain rates between 0.2 and 20 s^{-1}, the effect of microalloying additions (Ti, Nb, V, and Mo) on deformation behavior in bar steels was investigated. Deformation tests consisted of a single strike, compression test at temperature. After deformation, all samples were rapidly quenched with helium. Prior austenite grain size strongly influenced deformation and transformation behavior such that coarse, prior austenite grain sizes increased hardenability when deformation temperatures decreased while fine, prior austenite grain sizes increased hardenability when deformation temperatures and strain rates increased. Also, microstructural observations suggested that dynamic recrystallization was achieved in the steels, independent of composition, during the present deformation conditions.

4:45 PM
Optimum Design of Process Parameters to Minimize Distortion during Gas Quenching Process: *Zhichao Li*[1]; Ramana V. Grandhi[1]; Ragahavan Srinivasan[1]; [1]Wright State University, Dept. of Mechl. & Matl. Eng., Dayton, OH 45435 USA

During gas quenching process, the controllable process parameters include the preheated temperature of the component, the temperature of the quenching gas and the heat transfer coefficient between the component and the circulated gas. The heat transfer coefficient is designed non-uniformly along the surface of the component. These controllable parameters are investigated for the reasonability of being used as design variables to optimize the gas quenching process. The objective function of optimization is to minimize the distortion. Constraints on the residual stress and surface hardness distributions are subjected to improve the service qualities. The finite element package, DEFORM, is used to predict the material responses. The response surface method is used to fit the analytical models of the objective function and constraints in terms of the design variables. Once the closed-form response surface models are obtained, a design optimization tool, DOT, is used to search for the optimum design point.

5:10 PM
Computer Simulation of Microstructural Evolution of Waspaloy and Nickel Alloy 718 during Hot Forging Processes: *W. T. Wu*[1]; D. Huang[1]; D. Lambert[1]; S. L. Semiatin[2]; [1]Scientific Forming Technology Corporation, Columbus, OH 43220 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA

Computer simulation of microstructural evolution of superalloys during hot forming is of great interest particularly for manufacturing critical components for aerospace applications. Recrystallization and grain growth were modeled using phenomenological approaches, which were implemented with a commercial metal forming code DEFORM that determines the thermo-mechanical history. Necessary material parameters used in the analysis were collected from available literature. To validate the model with Waspaloy, an industrial disk forging process was simulated. The predictions of recrystallization fractions and grain size were in good agreement with the measurements. For nickel alloy 718, simple upsetting and double-cone forging were simulated and compared against experimental data in the literature. A 3D cogging simulation was also carried out using as-cast 718 material data to demonstrate the capability of the model.

Modeling the Performance of Engineering Structural Materials - III: Microstructure and Characterization

Sponsored by: Structural Materials Division, Structural Materials Committee
Program Organizers: Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA

Tuesday PM — Room: 140
November 6, 2001 — Location: Indiana Convention Center

Session Chairs: S. V. Raj, NASA Glenn Research Center, Cleveland, OH 44135 USA; P. K. Liaw, University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA

2:00 PM Invited
Deformation Banding and Grain Boundaries in Aluminum and Aluminum Alloys: *T. R. McNelley*[1]; D. L. Swisher[1]; [1]Naval Postgraduate School, Dept. of Mechl. Eng., 700 Dyer Rd., Monterey, CA 93943-5146 USA

Orientation imaging microscopy (OIM) methods have been employed to assess the evolution of microstructure during deformation processing of pure aluminum and selected aluminum alloys. Grain maps constructed from orientation data confirm the presence of deformation bands in which the lattice orientation alternates between the symmetric variants of the main texture component upon traversing from band to band. Various shear texture components were observed in pure aluminum following equi-channel angular pressing, depending upon the details of the processing route. The B texture component (<112>{110}) is prominent in rolled Supral 2004 while a C-type texture component (<111>{112}) is present following rolling of the Al-5%Ca-5% Zn alloy. In all of these materials the high-angle grain boundaries (disorientation of 50E-62.8E) in the microstructure are mainly the interfaces between the deformation bands while lower-angle boundaries (disorientation of 5E-20E) separate cells within the bands. Models of microstructural development that include deformation banding during severe plastic deformation may be employed to describe both texture as well as the evolution of the grain boundaries.

2:30 PM
Spinodal Decomposition in Small, Elastically Stressed Spheres: *William C. Johnson*[1]; Steven M. Wise[1]; [1]University of Virginia, Matls. Sci. & Eng., PO Box 400745, 116 Engineer's Way, Charlottesville, VA 22904-4745 USA

The influence of sphere size, surface stress, and compositional strain on microstructural evolution in small, isotropic spheres is examined numerically and analytically for binary systems. The surface stress and external traction interact with the second compositional derivative of the strain and can either enhance or diminish the stability of the alloy depending on their signs. Numerical solutions of the evolution equations are given that show the dependence of the phase compositions on sphere radius and the existence of two equilibrium states for a given alloy composition and temperature. This work is supported by the National Science Foundation under grant DMR-9902110.

2:55 PM
Competetive Growth of a Three Phase Thin Film: *Steven Matthew Wise*[1]; *William C. Johnson*[2]; [1]University of Virginia, Eng. Phys. Prog., 116 Engineers Way, PO Box 400745, Charlottesville, VA 22904 USA; [2]University of Virginia, Matls. Sci. & Eng. Dept., 116 Engineers Way, PO Box 400745, Charlottesville, VA 22904 USA

The influence of compositional strain, transformational strain, and temperature on the competetive growth between three phases in a thin film is examined. A dynamical system comprised of coupled Cahn-Hilliard and Allen-Cahn type equations is developed and solved numerically using a dual-grid finite difference scheme. The long-range elastic fields alter the sequence of phase formation from that observed in stress free systems. The magnitudes of these effects for both free standing film and film attached to a complient substrate will be presented. This work is supported by the United States Department of Energy under grant DE-FG02-99ER45771.

3:20 PM
The Grain Growth Simulation of IF Steel during the Continuous Annealing: *Yi Jing*[1]; Wang Zianjin[1]; [1]University of Science & Technology, Dept. of Metalforming, Beijing 100083 China

The annealing process plays an important role in the production of IF steel. It is well known that grain growth after the primary recrystallization can increase the preferred texture and the grain size, etc. The temporal evolution of 2D grain growth of recrystallized Ti-added IF steel is simulated by the Monte-Carlo model. In the simulation, the pinning effect of precipitates on growth kinetics is incorporated. By the conversion of Monte-Carlo step (MCS) to real time based on diffusion controlled mechanisms and the conversion of IF steel boundary energy to the boundary energy factor J, the simulated grain size is in good agreement with the experimental data.

3:45 PM Break

4:05 PM Invited
Damage Assessment using Nondestructive Evaluation Models: *M. Nabil Bassim*[1]; [1]University of Manitoba, Mechl. Eng., Winnipeg, MB R3T5V6 Canada

Models relating observed crack growth lengths and rates during fatigue of structures and components have been developed. These models are based on the Paris Law model for crack growth. Particularly, acoustic emission was used for characterization of the energy released during crack growth and related to the stress intensity factor and hence, the number of cycles to failure. The models developed were tested extensively on both laboratory fatigue specimens as well as on structural components. The models accurately predict the evolution of cracks and form a basis for assessment of remaining fatigue life.

4:35 PM Invited
Applications of Digital Image Analysis for Simulation of Micro-Mechanical Response of Microstructures of Complex Geometry: *A. M. Gokhale*[1]; Z. Shan[1]; S. Yang[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Eng., Atlanta, GA 30332 USA

Microstructures of engineering alloys often contain non-uniformly distributed features of complex geometry present at widely different length scales. Attributes of microstructural geometry have dominant influence on the mechanical behavior of materials. Therefore, it is of interest to incorporate detailed quantitative description of the geometry of microstructures in micro-mechanical analysis of materials. In this contribution, a digital image analysis based methodology is presented for performing finite elements (FE) based simulations on the microstructural digital images, and for linking the interactions among microstructural features at different length scales. The practical application of the method is demonstrated via FE-analysis on the microstructure of a cast Al-alloy, where the length scales of micro-pores and silicon particles differ by two orders of magnitude. The simulation captures the effect of non-uniformly distributed micro-pores at length scales of 200 to 500 microns on the local stresses and strains around silicon particles that are at the length scales of 3 to 5 microns. Three-dimensional FE-based computations are presented for simulation of the growth of non-uniformly distributed micro-pores of complex geometry under applied tensile load.

5:05 PM

Thermography Detection of Fatigue Damage of Reactor Pressure Vessel (RPV) Steels and Life Prediction: *Bing Yang*[1]; Liang Jiang[1]; Lijia Chen[1]; Peter Liaw[1]; D. Fielden[1]; Hsin Wang[2]; J. G. Huang[3]; R. C. Kuo[4]; J. Y. Huang[4]; [1]University of Tennessee–Knoxville, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [3]Taiwan Power Company, Taipei, Taiwan; [4]Institute of Nuclear Energy Research (INER), PO Box 3-14, 1000 Wenhua Rd., Chiaan Village, Lungtan 325 Taiwan

An infrared (IR) thermography technique, as a nondestructive evaluation technique, was applied to investigate the fatigue damage of Reactor Pressure Vessel (RPV) Steels during 0.5 Hz, 20 Hz, and 1000 Hz fatigue testing. Five stages of temperature profiles were observed: an initial specimen temperature increase, a followed temperature decrease, an equilibrium temperature region, an abrupt temperature increase, and a drop of temperature following specimen failure. The relationship among temperature, stress-strain state, and fatigue behavior is discussed. Both thermodynamics and heat-transfer theories are applied to model the observed temperature variation during fatigue. The predicted and measured temperature evolutions during fatigue were found to be in good agreement. The back calculation from the observed temperature to obtain inelastic deformation indicates the material damage during fatigue. Furthermore, fatigue life prediction by temperature has been conducted and compared with experimental data. Acknowledgements: The present research is supported by the Institute of Nuclear Energy Research, Taiwan, the Taiwan Power Company, the National Science Foundation (EEC-9527527, DMI-9724476, and DGE-9987548), Oak Ridge National Laboratory, and the US Department of Energy Secretary for Energy Efficiency and Renewable Energy, and Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program under contract DE-AC05-96OR22464, managed by Lockheed Martin Energy Research Corporation.

5:30 PM

Improved Method and Equipment for Adhesion Tests: *V. Komarova*[1]; M. Kireytsev[1]; [1]National Academy of Sciences of Belarus, Lab. of Tribology & Mech., Inst. of Machine Reliability, Minsk, Belarus

An adhesion strength between coating and base metal is one of the most important characteristics for machine reliability and durability. As a rule, measurement of an adhesion strength of coatings requires special methods and equipment. In engineering practice are well known the glue method and the pin method for an adhesion measurement. The glue method based on a separation of the glued together disks on one of which a coating is formed. The main disadvantages of the glue method and reasons it's called are the following: Instability of properties of glue composition. Some disadvantages of the pin method and reason it's called are the following: Van der waals forces are generated during adhesion measurements because of a gap up to 10 microns between a pin and a tube expander. To eliminate the above-mentioned disadvantages of the methods of adhesion measurement in our institute was designed and was tested modernized method and upgraded equipment. To improve the method and equipment we suppose that more reliable results can be obtained at an equipment, in which one the end surface coated with thermal flame spray process is made spherical above a surface of a yoke. In this construction a roof surface texture of a pin increases rigidity of the coating formed and reduces its trough. At minimal gaps there is no jamming. However difficulty to reproduce exact parameters of a hemispherical profile result in considerable decrease of accuracy of the measurements. To eliminate above-mentioned disadvantages an adaptation of the equipment for an adhesion estimation of layers of two-layer coating was made. It allows to increase accuracy of the measurements.

Powder Materials: Current Research & Industrial Practices: Composite Materials

Sponsored by: Materials Processing & Manufacturing Division, Powder Materials Committee

Program Organizers: Fernand D.S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgical Engineering, Rapid City, SD 57701-3995 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Tuesday PM — Room: 209
November 6, 2001 — Location: Indiana Convention Center

Session Chairs: Fernand D.S. Marquis, South Dakota School of Mines and Technology, Col. of Matls. Sci. & Eng., Rapid City, SD 57701-3995 USA; F. H. (Sam) Froes, University of Idaho, Moscow, ID 83844 USA

2:00 PM Keynote: Marc A. Meyers

2:40 PM

Processing of Microstructurally-Biased Two-Phase TiB2/Al2O3 Ceramics: Concerning the Influence of Particle Size: *Louis Ferranti*[1]; [1]Georgia Institute of Technology, Matls. Sci. & Eng., Love Bldg., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

Efforts to design ceramic materials with unique microstructures are receiving great interest. The processing of TiB2/Al2O3 ceramics with a biased microstructure is formed using self-propagating high-temperature synthesized (SHS) reactions. Once the SHS reaction is complete, additional processing steps are required for the formation of dense ceramic material. This includes the comminution of the porous SHS product to a desired particle size and hot pressing. The properties of advanced ceramics are largely dependent on the quality of the starting powder materials and variables introduced during processing. Control of the ceramic fabrication requires a deep understanding of powder properties. It is also important to control the processing conditions while analyzing the overall structure of the material in each stage of the fabrication process. A detailed study of the particle size distribution for the optimal densification of the ceramic material while developing an interconnected microstructure is thus performed for this material. The effect of varying ball-milling time (following reaction synthesis and prior to hot pressing) on the microstructure is examined and quantitatively characterized using integral mean curvature. In this paper, the results obtained to date will be described. Funded by ARO grant No. DAAG55-98-1-0454 (Dr. David Stepp, program monitor).

3:10 PM

Dense, Near Net-Shaped, Oxide/Intermetallic Composites by the Displacive Compensation of Porosity (DCP) Method: *P. J. Wurm*[1]; P. Kumar[1]; K. D. Ralston[1]; K. H. Sandhage[1]; M. J. Mills[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA

Dense, near-net shaped, ceramic/metal composites of high ceramic content have been fabricated using a novel, reactive infiltration approach at modest temperatures: The Displacive Compensation of Porosity (DCP) method. This process involves pressureless infiltration of a shaped ceramic preform by a metallic liquid that can undergo a displacement reaction with the preform. Because a larger volume of ceramic is produced than is consumed, the pore volume within the preform becomes filled (i.e., reaction induced densification), unlike other reactive infiltration processes. This paper demonstrates the fabrication of MgO/Fe-Al intermetallic composites using the DCP method. Relative to monolithic Fe-Al, this system has many potential advantages, including lighter weight and improved mechanical properties. By the pressureless infiltration of Mg(1) into, and then reaction with, porous MgA1204/MgO/Fe-bearing preforms at 1000°C, dense MgO/Fe-A1 composites have been produced. Final microstructure and composite properties resulting from processing conditions will be discussed.

3:40 PM Break

4:00 PM

Mechanical Properties of DC Carbide Dual Composites: *Xin Deng*[1]; B. R. Patterson[1]; K. K. Chawla[1]; M. C. Koopman[1]; Zak Fang[2]; Greg Lockwood[2]; A. Griffo[2]; [1]University of Alabama in Birmingham, Matls. & Mechl. Eng., 1150 10th Ave. S., BEC254, Birmingham, AL 35294 USA; [2]Smith International, Inc., 16740 Hardy St., PO Box 60068, Houston, TX 77205 USA

Double Cemented Carbide (DC Carbide) is a dual composite composed of spherical granules of conventional cemented carbide (WC+Co) embedded in a metal matrix. Compared with conventional cemented carbide, DC Carbide has a superior combination of toughness and high stress wear resistance due to having a greater matrix mean free path. This combination of properties indicates useful applications in oil well drill bit inserts and other mining tools. DC Carbide with different granule properties, granule size, matrix volume fraction, different metal matrices (Co and steel) and heat treatment (for steel matrix) have been fabricated and mechanically tested. Mechanical property comparisons between DC Carbide and conventional cemented carbide will be presented. The relationships between mechanical properties and microstructural parameters of DC Carbide have been modeled. *Supported by NSF, DMR-9904352.

4:30 PM
Low-Temperature Fabrication of Tungsten/Zirconium Carbide Composites by the Prima-DCP Process: *Matthew B. Dickerson*[1]; Robert L. Snyder[1]; Ken H. Sandhage[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA

The PRIMA-DCP process is a novel, low temperature and near net shape route that is an attractive alternative to hot pressing methods traditionally used to fabricate high performance tungsten/zirconium carbide components. The creation of dense materials by the PRIMA-DCP process is accomplished by the pressureless infiltration of a Zr-Cu liquid into a porous WC ceramic body whereupon a displacement reaction occurs. The sum of the molar volumes of the reaction products, ZrC and W, is about twice the molar volume of WC. Such in-situ, reaction assisted pore filling causes the non-reactive molten copper to be squeezed out of the body. Copper acts as a catalytic processing agent, by dramatically lowering the reactive infiltration temperature by about 800°C and then leaving the final composite. The effects of processinty parameters on the phase content, microstructure and ultimate properties of the formed composite materials will be discussed.

5:00 PM
Hot Explosive Fabrication of B4C-Al Composites: Structure/Properties Relationships: *Roman Aptsiauri*[1]; Akaki A. Peikrishvili[1]; Georg Kalandadze[2]; Archil Eristavi[2]; Fernand D.S. Marquis[3]; Nikoloz Chikhradze[1]; Zakaria Chikviladze[1]; [1]Institute of Mining Mechanics, Tbilisi 380086 Georgia; [2]Institute of Stable Isotopes, Tbilisi 380086 Georgia; [3]South Dakota School of Mines and Technology, Dept. of Matls. & Metlgcl. Eng., Col. of Matls. Sci. & Eng., Rapid City, SD 57701 USA

Hot explosive technology with cylindrical loading geometry was used in order to consolidate B4C-Al composite powders up to near theoretical density and to obtain long-body cylindrical samples with improved properties. The temperature before shock loading was set within 20-8000C range. The intensity of loading was set under 10 GPa. The results of this investigation show that the compaction of powders at elevated temperatures under controlled shock parameters is an effective process designed to obtain bulk composites with near theoretical densities. The initial density of the compacting powders was observed to be an important parameter in hot compacting technology and its accurate control provides a way of obtaining composites with high density and correct geometry. The effect of the parameters mentioned above on the hardness and strength, and the structure/properties relationships obtained in these composites will be discussed.

Processing/Property Relationships in Polymer-Matrix Composites and Nanocomposites - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee, SPE Polymer Analysis Division
Program Organizers: Linda Schadler, Rensselaer Polytechnic Institute, Materials Science & Engineering Department, Troy, NY 12180-3590 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; Carl Schultheisz, National Institute of Standards and Technology, Polymers Division, Gaithersburg, MD 20899 USA; Sindee Simon, Texas Tech University, Department of Chemical Engineering, Lubbock, TX 79409 USA

Tuesday PM Room: 201
November 6, 2001 Location: Indiana Convention Center

Session Chairs: Linda Schadler, Rensselaer Polytechnic Institute, Matls. Sci. & Eng. Dept., Troy, NY 12180-3590 USA; Marc Meyers, University of California–San Diego, San Diego, CA 92093 USA

2:00 PM Keynote
Synthesis and Properties of Composite Materials Containing Chemically-Modified Multiwalled Carbon Nanotubes: *Rodney Andrews*[1]; John Anthony[2]; Robert G. Bergosh[2]; Chad Landis[2]; Mark S. Meier[2]; Terry Rantell[1]; [1]University of Kentucky, Ctr. for Appl. Energy Rsrch., 2540 Research Park Dr., Lexington, KY 40511 USA; [2]University of Kentucky, Dept. of Chem., Lexington, KY 40506-00 USA

Harnessing the unique physical properties of multiwalled carbon nanotubes (MWNTs) in materials applications has yet to be fully realized. One practical approach to producing MWNT composites is by shear mixing of MWNT into polymer matrices followed by extrusion or injection molding. Sufficient dispersion has been found to be the key in realizing the potential of these unique nano-reinforcements. Polystyrene-nanotube composites were prepared using two different chemically modified multiwalled carbon nanotubes and unmodified MWNT for comparison. After shear blending, thin composite films were pressed and the mechanical properties of the films measured. The composites prepared from modified nanotubes exhibited improved tensile and flexural properties over native polystyrene films, and were significantly higher than unmodified MWNT composite films. This suggests that the interaction between the nanotube and the polymer matrix has been significantly enhanced by the chemical treatment, and presents a path to super-strong composite materials based on carbon nanotubes.

2:30 PM
Effects of Curvature on the Elastic Modulus of Carbon Nanotube-Reinforced Polymers: *Cate Brinson*[1]; Frank Fisher[1]; [1]Northwestern University, ME Dept, 2145 Sheridan Rd, Evanston, IL 60208 USA

We will investigate the effects of nanotube curvature on the elastic modulus of carbon nanotube-reinforced polymers. Recent experimental results suggest that substantial increases in the modulus of such materials can be attained through the addition of very small amounts (~ 1% weight fraction) of carbon nanotubes as a reinforcing phase. In addition, microscopic images of nanotubes dispersed within a polymer show that they tend to exhibit significant curvature. Thus the goal of this work is to develop a model that will incorporate curvature effects into micromechanical analyses of carbon nanotube-reinforced polymers. An analytical model describing the decrease in effective reinforcement as a function of nanotube curvature will be presented. This model will be compared to the results of a finite element analysis study that will relate the moduli of straight and curved nanotubes. Finally, we will compare micromechanical predictions of the effective Young's modulus of nanotube-reinforced polymers, based on TEM images of actual nanotube geometry, with experimentally obtained data.

2:50 PM
Effectiveness of Nano-Reinforcement in Enhancing Polymeric Resin for Improved Composite Performance: *G. P. Tandon*[1]; R. Y. Kim[1]; [1]University of Dayton Research Institute, 300 College Park, Dayton, OH 45469-0168 USA

The objective of this study is to investigate the modification of polymer matrices with nano-particles to enhance the properties of fiber reinforced composites. In order to fully realize and exploit the gains resulting from nano-reinforcement, two fundamental questions need to be answered. Firstly, we need to assess the success of nano-particles as fillers and secondly, we need to ascertain whether the fundamental concepts pertaining to continuum mechanics of traditional fiber composites can be applied and validated at the nanometric level. This will be accomplished by using fibers of intermediate diameters and size (100-200 nm diameter, chopped), using an integrated experimental and analytical approach. Four groups of test specimens will be made. The first set will correspond to the neat resin alone to establish the baseline properties. In the second group, nano-fibers will be added to the neat resin to assess the benefits of nano-reinforcement. The third group will be a unidirectional IM7/Epon 828 composite using neat resin while in the fourth set, nano-fibers will be added to the epoxy resin in addition to fiber (IM7) reinforcement. The third and the fourth set will help in evaluating the benefit of resin modification in improving composite performance. We will characterize the mechanical properties of these nano-particulate composites and their constituents, and utilize/develop micromechanical theories to predict their thermo-mechanical performance.

3:10 PM
Thermal and Mechanical Properties of Alumina/Polymethylmethacrylate (PMMA) Nanocomposites: *Benjamin Jesse Ash*[1]; Linda S. Schadler[1]; Richard W. Siegel[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Eng., 110 8th St., MRC 142, Troy, NY 12180 USA

Alumina/polymethylmethacrylate nanocomposites were synthesized by free-radical polymerization. The 39 nm diameter alumina nanoparticles used in the study were produced by a forced gas condensation method and were used as received, or coated using a two step process resulting in the attachment of an azo-initiator directly to the surface of the particle. At an optimum weight percent, the resulting uncoated nanocomposites showed an average of 600% increase in their strain-to-failure and the appearance of a well-defined yield point when tested in uniaxial tension. Concurrently, the glass transition temperature of the nanocomposites dropped by as much as 20°C, while the ultimate strength and the Young's modulus decreased by 20% and 15%, respectively. We investigate the role of the particle/polymer interface in altering the Tg and in changing the mode of deformation in the bulk polymer.

3:30 PM Break

3:50 PM
Characterization of the Viscoelastic Creep Response of Polypropylene and Polypropylene Montmorillonite Nanocomposites: *Alejandro Hernandez-Luna*[1]; Nandika Anne D'Souza[1]; [1]University of North Texas, Matls. Sci., PO Box 305310, Denton, TX 76203-5310 USA

Tensile dog bone samples of Polypropylene were tested at room temperature by following a creep-recovery cycle of 2 hours. The constant stresses corresponded to values below the yield point of polypropylene (PP). The viscoelastic response was analyzed and a master curve for the behavior of the material on time was acquired. Nanocomposites (NC) of montmorillonite, polypropylene and maleated PP (mPP) were also prepared and tested under the same conditions. Small changes in the surface temperature were observed in all the specimens using an infrared (IR) camera. Changes were related to the structural changes on the samples. mPP was used as a bonding agent between the PP and the nanoclay. In order to prove that the changes are not influenced by the mPP, blends of mPP and clay, and mPP and polypropylene were also tested. X ray and DSC analysis was done to the specimens to determine crystallinity changes.

4:10 PM
Viscoelastic Performance of Epoxy Nanocomposites: *Andre Lee*[1]; [1]Michigan State University, Matls. Sci. & Mech., Rm. 3514, Engineering Bldg., E. Lansing, MI 48824 USA

There is considerable interest in the polymer behavior at the non-equilibrium glassy state. It has been suggested that the polymer property in the glassy state can be influenced by the surrounding environment (topological constraints) and the chain architecture (interal constraints). Using model epoxy systems incorporated with either the non-functionalized, nanoscopic sized polyhedral oligomeric silsesquioxane, POSS (topological constraints) or epoxy functionalised POSS macromers (internal constraints), we investigated the effects on the kinetics and nonlinearity of the structural recovery in the glassy state and on the viscoelastic performance of these nanocomposites.

4:30 PM
Processing and Characterization of Nanomers Modified Epoxy Resin for VARTM: *Wayne Reitz*[1]; Uday Vaidya[1]; Walter Young[1]; [1]North Dakota State University, Mechl. Eng., 111 Dolve Hall, Fargo, ND 58105 USA

Recent studies reported in literature have shown that nanocomposites have the potential to improve structural, thermal, mechanical and barrier properties. Surface modified montmorillonite mineral nanomers are suited for dispersion in epoxy resin system. The dispersion (exfoliation) creates a near-molecular blend. Fiber reinforced composite processing has traditionally involved high aspect ratio fillers. Because nanometer-sized particles approach the scale of resin molecules it is feasible to process and use them in conjunction with epoxy resin. Vacuum assisted resin infusion/transfer molding (VARTM) has emerged as a low-cost out-of-autoclave processing alternative for fiber reinforced composite materials. The VARTM process is attractive because selective tailoring of properties can be achieved by infusion of nanomer modified resin over a desired area of the perform (fiber). The first stage of testing evaluated thin sheets of epoxy nanocomposites. They were microstructurally characterized and correlated to physical test results. Microstructure evaluation included dispersion characterization (density and segregation), optical clarity determination, hardness testing and compression testing. The second stage of evaluation examined 2- and 3-ply composite laminates. The results from processing and testing of the epoxy nanocomposites in light of the VARTM process will be presented.

4:50 PM
Structure, Morphology, and Mechanical Response of Polyurethane Surface Domains using X-Ray Photoelectron Spectroscopy (XPS), Static Secondary Ion Mass Spectrometry (SSIMS), and Scanning Probe Microscopy (SPM) Techniques: *Jeffrey E. Ramsdell*[1]; Sudipta Seal[1]; [1]University of Central Florida, Adv. Matls. Proc. & Analy. Ctr. (AMPAC), 12443 Research Pkwy., Ste. 404, Orlando, FL 32826 USA

This study investigates the chemical structure, morphology, and mechanical response of surface domains of polyurethane materials. X-ray photoelectron spectroscopy (XPS) and static secondary ion mass spectrometry (SSIMS) are used to map the changes in the surface chemistry of polyurethanes due to curing conditions, aging, chemical exposure, and abrasive use. We are able to discern shifts in elemental binding energies due to changes in the ratio of rigid to flexible segments of the polyurethane material at the surface. Results include a reorientation of the rigid and flexible backbone segments at the surface as the materials are used in such processes as chemical mechanical polishing (CMP). We also report on the use of atomic force microscopy (AFM) and other scanning probe microscopy (SPM) techniques to further characterize the surface of polyurethanes. These techniques allow for correlation of morphological and mechanical response changes with surface chemistry changes found using XPS and SSIMS.

Affordable Metal-Matrix Composites for High Performance Applications - IV: Processing of MMCs - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: Awadh B. Pandey, Pratt & Whitney, Liquid Space Propulsion, West Palm Beach, FL 33410-9600 USA; Kevin L. Kendig, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Watson, Pratt & Whitney, East Hartford, CT 06108 USA

Wednesday AM	Room: 139
November 7, 2001	Location: Indiana Convention Center

Session Chair: Daniel B. Miracle, Air Force Research Laboratory, Matls. & Mfg. Direct., Dayton, OH 45433 USA

8:30 AM Invited
Forgability Determination Study for Aluminum Composite Materials: *John J. Lewandowski*[1]; Nishad Prabhu[1]; Erik Hilinski[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Eng., Cleveland, OH 44106 USA

Unique forging simulation equipment exists at CWRU that combines high load capacity (i.e. 110/220 kip) and the ability to test "large specimens", loading rates which span industrially relevant ranges (i.e. 0.004–120 in/sec); the ability to provide single or multiple deformation sequences at desired strain levels/loading rates; and data acquisition (e.g. load, deflection) at rates sufficient to capture the load history during the single/multiple deformation sequences. The presentation will begin with description of the equipment capabilities and examples of preliminary work on a range of materials, including both fully dense and porous P/M aluminum composites. In the case of discontinuously reinforced aluminum composites, preliminary tests on the effects of changes in test temperature, forging rate, and forging strain on evolution of flow stress, reinforcement distribution, and cracking in the billet will be illustrated on sub-scale cylindrical forging specimens with initial dimensions ranging from 1" diameter X 2" height to 2" diameter X 3". Forging strains of 0.7 and 1.3 were imparted to the fully dense P/M aluminum composites. In the case of the porous P/M aluminum composites, the equipment was used to determine the effects of changes in forging rate on the densification/billet cracking of forging strains were imparted to determine the effects of changes in forging rate and forging strains on the final density of the powder forged composite. The range of conditions that such equipment can simulate and the examples provided illustrate the important effects of deformation processing conditions on the resulting structure and properties of such materials. The ability to direct powder forge such composites provides another mechanism to potentially reduce processing costs of such materials.

9:00 AM
Processing and Mechanical Behavior of Particle Reinforced Metal Matrix Composites by a Novel Sinter-Forging Technique: *Jason J. Williams*[1]; Nikhilesh Chawla[1]; [1]Arizona State University, Dept. of Chem. & Matls. Eng., PO Box 876006, Tempe, AZ 85287-6006 USA

This study focuses on the characterization and mechanical behavior of an aluminum matrix composite with SiC particle reinforcement processed by a novel, affordable sinter-forging technique. Reinforcement particles and matrix alloy powders were blended, cold pressed, sintered, and hot forged, eliminating costly extrusion and secondary finishing steps to produce a net-shaped part. The microstructure, as well as the ambient-temperature monotonic and cyclic fatigue behavior of composites will be reported. The effects of SiC particle size and alloy powder size on processing and properties will be discussed. Comparisons to composites produced by conventional powder metallurgy and extrusion have also been conducted. It will be shown that, although the sinter-forged composites have a lower ductility than their extruded counterparts, the strength and fatigue resistance are comparable. Research sponsored by the United States Automotive Materials Partnership (USAMP) and the Office of Naval Research (ONR).

9:20 AM
Infrared Infiltration of Cu/W and Cu/WC Composites in Argon: *P. Deshpande*[1]; K. Chokalingam[1]; J. H. Li[1]; R. Y. Lin[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., M. L. 12, Cincinnati, OH 45221-0012 USA

Cu/W and Cu/WC composites have been prepared with infrared infiltration processes in a flowing argon atmosphere. The infiltration temperature was 1250°C for time up to 2 minutes. Powder preforms of reinforcement particles were prepared with a cylindrical die at pressures between 45 ksi (310 MPa) and 135 ksi (930 MPa). Wettability between molten copper and tungsten particles was excellent under the processing conditions of this study. High density composites have been obtained. Image analysis on the cross section of the composite indicates that up 95 vol% W can be prepared with this infrared infiltration technique. Hardness measurements show that the composite hardness is close to that of pure tungsten. Very little dissolution of tungsten in copper was observed. This is in agreement with that disclosed in the literature.

9:40 AM
In-Situ Processing of Aluminum Alloy Matrix Composites: Qingjun Zheng[1]; *Ramana G. Reddy*[1]; [1]The University of Alabama, Dept. of Metall. & Matls. Eng., Tuscaloosa, AL 35487-0202 USA

In-situ processing of aluminum alloy composite was investigated and reviewed. The composites reinforced with SiC or AlN particulates have been formed in the temperature range of 950-1200°C by bubbling methane, nitrogen or ammonia gas through Al alloy melt. This novel method is superior to the traditional solidification method. In-situ growth of the expensive but highly useful reinforcement, without formation of any metastable phases, will not only reduce materials and production cost, but also improve the mechanical properties of the composites. Experimental studies showed that the reinforcing particles formed in-situ, such as AlN and SiC, were small in size and were uniformly dispersed in Al alloy matrix. A thermodynamic model and a kinetic model were developed to understand the process mechanism. Also, the effects of materials variables and process variables on the in-situ synthesis of the particulate reinforcement were discussed.

10:00 AM Break

10:15 AM Invited
In-Situ Formation of Fiber Reinforced Intermetallic Matrix Metal Matrix Composites: *H. J. Rack*[1]; [1]Clemson University, Dept. of Cer. & Matls. Eng., 110 Olin Hall, Clemson, SC 29634-0907 USA

A variety of techniques are now available for fabrication of metal matrix composites. Generally these procedures attempt to minimize the reaction between the metal matrix and the reinforcement through control of process temperature, time and/or matrix composition. Reinforcement coating procedures have also been considered to prevent this “adverse” reaction. Alternatively it has been shown that it is possible to fabricate thermodynamically stable short fiber reinforced metal matrix composites through control of the reaction process. This presentation will examine the fabrication of alumina reinforced intermetallic matrix composites through the controlled reaction of Al with TiO_2, Ta_2O_5 and Nb_2O_5 during either liquid metal infiltration or subsequent heat treatment of un-reacted pressure infiltrated ceramic preforms.

10:45 AM
Gas Atomization and Spray Forming of Aluminum Alloy Matrix Pre-Composite Powders: *Andrea Zambon*[1]; Brando Badan[1]; [1]Università di Padova, DIMEG, via Marzolo, 9, Padova 35131 Italy

The production of pre-composite aluminum alloy based powders, via melting of an Al-Si alloy in the induction furnace of a gas atomizing unit, and adding in situ a suitable amount of particulate dispersoids is described. The wetting of the reinforcing particles was improved by a properly chosen melt temperature and enhanced by means of mechanical stirring under an Argon shielding atmosphere. Gas atomization via sonic close coupled discrete jets nozzles was performed, and a part of the spray cone was intercepted by a still substrate. Microstructural characterization of both the pre-composite powders and of the spray deposited sample was carried out by microscopy examination and X-ray diffraction.

11:05 AM
Interfacial Reactions at Elevated Temperatures in New Low-Cost Al/SiC Metal Matrix Composites: *Glenn J. Grant*[1]; Darrell R. Herling[1]; David E. McCready[2]; [1]Pacific Northwest National Laboratory, Matls. Proc. & Perfor., 902 Battelle Blvd., P8-35, Richland, WA 99352 USA; [2]Pacific Northwest National Laboratory, Wm. R. Wiley Lab., EMSL, 3020 Q Ave., K8-93, Richland, WA 99352 USA

The mechanical properties of Metal Matrix Composites (MMCs) are strongly affected by the quality of the bond between the matrix and the reinforcing particle. In aluminum MMCs reinforced with SiC particles, the particle/matrix interface can be degraded at high temperature by the formation of aluminum carbide and magnesium and aluminum/magnesium oxides. The temperature of these reactions is an important process limit during casting, remelting, and eventual product recycling. Recently, lower cost Al/SiC MMCs have become available that utilize less well graded particulate and unique, rapid-mixing techniques. However, as a result of the relaxed control on the particle size fraction, a significantly larger percentage of the particulate is found in the finer size ranges. This leads to an increase in the area of particle to matrix surface and raises the possibility that detrimental aluminum carbide and/or oxide formation could occur at lower temperatures, or lower time-at-temperature, than in current industrial products. In this study, we quantify by conventional, and in-situ liquid metal XRD, the time-temperature relationship for interfacial carbide/oxide formation, and compare commercially available MMC materials to MMC material produced from less well-graded SiC particulate.

11:25 AM
Coating Process Options for Continuous Fiber AMC Filament Winding: *Brian L. Gordon*[1]; [1]Touchstone Research Laboratory, RD1, Box 100B, The Millennium Centre, Triadelphia, WV 26059 USA

Improvements have been made to the unique AMC manufacturing method that enables the development of high performance continuous fiber aluminum matrix composite aircraft and spacecraft components. Touchstone’s patented brazing process combines AMC properties with low-cost PMC production techniques, such as filament winding. The key to this technology is an in situ brazing process analogous to cure-on-the-fly processing of polymer composites. This process requires a thin coating of brazing alloy on the surface of the AMC prepreg tape. This paper discusses several options that have been identified and utilized for applying coatings of the desired thickness. Test results demonstrating the improvements are also presented.

11:45 AM Invited
The Effect of Pulse on the Laser Welding Pool of SiCp/6061 Composite: *Niu Jitai*[1]; Zhang Deku[1]; Xiuyu Yu[1]; Wang Muzhen[1]; [1]Harbin Institute of Technology, Harbin 150001 China

In this paper, the restraint effects of pulse frequency and pulse duty cycle on the precipitates of harmful needle-shape phase Al_4C_3 are studied in pulse laser welding of SiCp/6061 aluminum matrix composites, and its microstructures under the different process parameters (pulse time from 1ms to 20ms, duty cycle from 50% to 91%) are analyzed. In order to contrast, the samples welded by continuous laser are adopted as the controlled. The results show that the proper laser pulse frequency and duty cycle can depress the formation of harmful needle-shape Al_4C_3 effectively. At the same time, the stir action of laser pulse makes the reinforcement phase distribute more evenly so that the strength of welded joint can be further increased.

12:10 PM Invited
Characterization of Damage Evolution in Al Matrix Alloys for Metal Matrix Composites: *Arun M. Gokhale*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Eng., Atlanta, GA 30332-0245 USA

Numerous metal matrix composites (MMC) have Al-alloy matrix. These matrix alloys often contain brittle second phase inclusions. The deformation, damage evolution, and fracture of MMCs are affected by the cracking and debonding of the matrix inclusions and growth of voids around cracked/debonded inclusion particles. In this contribution, particle cracking, debonding, and void growth in important matrix Al-alloys are quantitatively characterized. The fraction of damaged particles, their size distributions, and orientation distribution of particle cracks are measured by using image analysis and stereological techniques. Particle cracking and debonding are predominant damage modes. Particle debonding is observed only under externally applied tensile loads. The relative contributions of particle debonding and fracture to the total damage strongly depend on stress-state, temperature, and alloy chemistry. The rate of damage accumulation is different for different loading conditions, and it also varies with the particle and alloy chemistry. The anisotropy of the damage is highly dependent on the deformation path and stress triaxiality. Under uniaxial tension, the cracks in the broken particles are mostly perpendicular to the loading direction, whereas in the compression test specimens they are parallel to the loading direction. The particle cracks in the torsion and notch-tension test specimens do not exhibit preferred orientations. The quantitative microstructural data are used to test damage evolution models.

Friction Stir Welding and Processing - IV: Friction Stir Processing

Sponsored by: Materials Processing and Manufacturing Division, Shaping and Forming Committee, Texture & Anisotropy Committee

Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; David P. Field, Washington State University, Pullman, WA 99164-2920 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; Shigeo Saimoto, Queens University, Ontario K1L 3NG Canada; S. L. Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Wednesday AM   Room: 115
November 7, 2001   Location: Indiana Convention Center

Session Chairs: Rajiv S. Mishra, University of Missouri, Rolla, MO 65409-0340 USA; Charles J. Warren, Alcoa Technical Center, USA

8:30 AM
High Strain Rate, Thick Section Superplasticity Created via Friction Stir Processing: *Murray W. Mahoney*[1]; Rajiv Mishra[2]; Tracy Nelson[3]; Jonathan G. Flintoff[1]; Rinat Islamgaliev[2]; Yuri Hovanski[3]; [1]Rockwell Science Center, Structl. Metals, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA; [2]University of Missouri, Metall. Eng., 218 McNutt Hall, 1870 Miner Cir., Rolla, MO 65409 USA; [3]Brigham Young University, 265 CTB, Provo, UT 84602 USA

This work demonstrates high strain rate (>10-3), thick section (>5mm) superplasticity in friction stir processed (FSP) 7050 Al. Elements of process control and microstructural evolution are characterized in detail. Superplastic behavior is illustrated as a function of friction stir processing variables as well as superplastic test conditions of temperature, accumulated strain, and strain rate. High strain rate and thick section superplasticity are two material properties never before demonstrated on a practical scale. For example, high uniform elongation (>500%) is achieved for strain rates >1x103s-1 at temperatures less than 460°C. Further, friction stir processing produces a relatively uniform fine grain size through the sheet thickness. This allows fine grain microstructures to be created in a section thickness >5mm, i.e., a thickness considerably greater than attainable by conventional thermomechanical processing. It will be shown that high levels of elongation, even at the highest strain rates, remain uniform, i.e., no diffuse necking. This offers the potential to form complex shaped parts at a higher rate and in section thickness never before possible.

9:00 AM
Friction Stir Welding and Processing of Aluminum Metal Matrix Composites: *Glenn J. Grant*[1]; Darrell R. Herling[1]; Richard W. Davies[1]; [1]Pacific Northwest National Laboratory, Matls. Proc. & Perfor., 902 Battelle Blvd., P8-35, Richland, WA 99352 USA

The friction stir process has the potential to provide weld-bonded solutions for many combinations of materials formerly considered un-weldable. One of the materials considered difficult to weld by conventional fusion methods is the family of aluminum metal matrix composites. The main problem encountered in both fusion and solid state welding methods is the detrimental affect that the process has on the interface between the matrix and the reinforcement particle. Melting associated with fusion welding can de-wet the particle from the matrix. As a solid state joining technique imparting large plastic deformation on the parent material, the friction stir-welding process will potentially create a weld with unique characteristics. However, this process may also affect the nature of the particle-matrix interface by creating cavitation and pull-apart voids around sharp corners of the particles due to the extreme plastic flow in the matrix. In the current study, microstructural data on friction stir welds between and among 6000 series Al, Al/SiC and Al/Al2O3 MMC sheets and castings will be presented. Characteristics of weld defects and data on tool wear and process parameters will be presented.

9:25 AM
Superplasticity in Aluminum and Magnesium Alloys Prepared by Friction Stir Processing: *Darrell R. Herling*[1]; Glenn J. Grant[1]; Rich W. Davies[1]; [1]Pacific Northwest National Laboratory, Energy Sci. & Tech. Div., 902 Battelle Blvd., MSIN: P8-35, Richland, WA 99352 USA

There have been many scientific advances over the last decade in the area of superplasticity and superplastic forming. In addition, a lot of the development work has focused on aluminum sheet materials and studying the effects of thermomechanical processing on the mechanical properties and superplastic behavior. It is well known that one of the important requirements for superplastic behavior is an equiaxed, fine-grain microstructure. The primary method of processing aluminum sheet materials for superplastic behavior is through a series of hot and extensive cold rolling operations. This is a robust and efficient method to produce superplastic sheet materials, but due to the high degree of cold working required to significantly refine the microstructure, sometimes rolling is unpractical. Typically, materials with grain sizes larger than 10 microns are not advantageous for superplastic forming. Large and inhomogeneous microstructures are evident in fusion weld joints, and hence are not typically superplastic. Recently, research has focused on alternative processing methods to assist in grain refinement through severe plastic deformation. Friction stir processing is one such technology that has promise in creating submicron-scale structures in weld materials. Aluminum alloys 2195 and 5083, as well as magnesium alloy AZ31 were subjected to friction stir processing, and then characterized for superplastic behavior. This paper presents the process parameters used, as well as the results from uniaxial tensile testing under typical superplastic conditions.

9:50 AM
Superplastic Deformation of a Friction Stir Welded 7050 Aluminum Alloy at High Temperatures: *Venkat Seetharaman*[1]; Kumar V. Jata[2]; S. L. Semiatin[2]; [1]UES, Inc., Matls. & Proc. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

Wrought plates of a 7050-T7451 aluminum alloy were friction stir welded to produce high quality butt joints. Cylindrical compression test specimens corresponding to a longitudinal orientation were machined from the friction stir zone, thermomechanically-affected zone, and the base material. Isothermal upset tests were carried out under step-strain rate change conditions at constant temperatures in the range 400°C-480°C to a total strain of 0.7. The microstructures of the stir zone, heat affected zone, and the base material both in the as-welded condition and after high temperature deformation were characterized. The weld nugget contained very fine recrystallized grains; in contrast, the base material and the thermomechanically-affected zone consisted of relatively large grains containing fine and coarse precipitates of the eta phase, respectively. While the specimens from the stir zone exhibited several characteristics of superplasticity over strain rates ranging from 10-2 to 10-5 s-1, the specimens extracted from the base material and the thermomechanically affected zone deformed by lattice diffusion controlled dislocation glide/climb processes. The paper describes high temperature plastic flow characteristics obtained from the three regions and also the microstructure changes resulting from the plastic deformation.

10:15 AM Break

10:25 AM
Abnormal Grain Growth during Heat Treatment in Friction Stir Processed 7050 and 2519 Aluminum Alloys: *Rajiv S. Mishra*[1]; Rinat K. Islamgaliev[1]; Tracy W. Nelson[2]; Yuri Hovanski[2]; Murray W. Mahoney[3]; [1]University of Missouri, Dept. of Metall. Eng., Rolla, MO 65409-0340 USA; [2]Brigham Young University, 265 CTB, Provo, UT 84602 USA; [3]Rockwell Science Center, Structl. Metals Dept., Thousand Oaks, CA 91360 USA

Friction stir welding (FSW) and friction stir processing (FSP) lead to a fine grained ‘nugget’ region. The microstructural refinement and uniformity depends on processing parameters and tool design. During post-processing at high temperatures, friction stir processed 7050 and 2519 aluminum alloys show abnormal grain growth that has not been previously reported. The locations for onset of abnormal grain growth are different for these two alloys. The nucleation and growth characteristics of abnormal grains have been investigated using differential scanning calorimetry, orientation imaging, and transmission electron microscopy. The role of texture, precipitate distribution, and microstructural non-uniformity in abnormal grain growth will be presented. The observation of abnormal grain growth has important implications for post-FSW heat treatment and superplastic properties of FSP material.

10:50 AM

Production of Fine-Grained Aluminum Alloys by Friction Stir Processing: *Ichinori Shigematsu*[1]; Naobumi Saito[1]; Takeshi Komaya[2]; Takaharu Tamaki[2]; Goro Yamauchi[3]; Mamoru Nakamura[1]; [1]National Institute of Advanced Industrial Science and Technology, METI, 1-1, Hiratecho, Kita-ku, Nagoya, Aichi 4628510 Japan; [2]Daido Institute of Technology, Grad. Sch. Dept. of Mechl. Eng., 10-3 Takiharu-cho, Minami-ku, Nagoya, Aichi 4578530 Japan; [3]Daido Institute of Technology, Dept. of Mechl. Eng., 10-3 Takiharu-cho, Minami-ku, Nagoya, Aichi 4578530 Japan

Friction stir processing (FSP) was applied to 1050 aluminum alloy (commercially pure aluminum) to produce fine-grained structure. By varying the processing condition, the temperature at the FSP zone could be controlled from 393K to 733K. When the temperature at the FSP zone was below 473K, fine-grained 1050 aluminum alloy could be produced. For example, the grain size in the FSP zone is 0.5-1micron meters and 4-8micron meters for the rolled sample and the annealed sample, respectively. That is, grain size in the FSP zone depended on the condition of the base metal, e.g. the rolled sample and the annealed sample. These results show that FSP can be a useful processing to produce fine-grained aluminum alloy.

11:15 AM

Superplasticity Behavior of Friction Stir Processed Aluminum-Lithium Alloy: *Indrajit Charit*[1]; Rajiv S. Mishra[1]; Kumar V. Jata[2]; [1]University of Missouri, Dept. of Metall. Eng., Rolla, MO 65409 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

Friction stir processing (FSP) is currently being developed as a grain refinement tool for several commercial aluminum alloys. The fine grain size in the nugget region leads to superplasticity. However, the optimum strain rate and overall ductility depends on the alloy composition. In this paper, the superplastic behavior of FSP Al-Li-Cu alloy (AF/C458-T3) is reported. The nugget region of the FSP alloy showed equiaxed grains of 15-16 µm. So far the maximum ductility of ~320% was achieved at 500°C and strain rate of 10^{-3} s^{-1}. The mechanism of superplasticity will be discussed and compared with other FSP aluminum alloys as well as conventionally processed Al-Li alloys. Present results show the potential of FSP to achieve superplasticity in a single step as compared to conventional multi-step thermo-mechanical processing. The support of the National Science Foundation through a grant DMI-0085044 is gratefully acknowledged.

11:40 AM

Microstructure and Mechanical Behavior of Friction Stir Processed Al-Ti-Cu Alloy: *Jing Zheng*[1]; Rajiv S. Mishra[1]; Patrick B. Berbon[2]; Murray W. Mahoney[2]; [1]University of Missouri, Dept. of Metlgcl. Eng., Rolla, MO 65409-0340 USA; [2]Rockwell Science Center, Structl. Metals Dept., Thousand Oaks, CA 91360 USA

Friction stir processing (FSP) is a new technique for thermo-mechanical processing of materials. Recently, FSP has been applied to process a powder metallurgy alloy. By this technique, a hot pressed sheet can be converted into a fully dense material. In this paper, the mechanical properties and microstructure of a friction stir processed Al-Ti-Cu alloy are reported. FSP results in a remarkably uniform microstructure eliminating the banded structure commonly observed in extruded powder metallurgy alloys. The uniform microstructure in FSP Al-Ti-Cu gives strength of over 700 MPa at ambient temperature, with more than 10% elongation. The variation of strength and ductility with temperature are also presented. FSP provides a new opportunity to produce high strength powder metallurgy materials with a tailored microstructure. The support of Air Force Office of Scientific Research through grant #F49620-00-1-0022 is gratefully acknowledged.

Fundamentals of Solidification - III

Sponsored by: Materials Processing & Manufacturing Division, Solidification Committee

Program Organizers: John H. Perepezko, University of Wisconsin–Madison, Department of Materials Science & Engineering, Madison, WI 55706 USA; Christoph Beckermann, University of Iowa, Department of Mechanical Engineering, Iowa City, IA 52242-1527 USA; William J. Boettinger, National Institute of Standards & Technology, Gaithersburg, MD 20879 USA

Wednesday AM — Room: White River Ballroom
November 7, 2001 — Location: Indiana Convention Center

Session Chair: William J. Boettinger, NIST, Metall., Gaithersburg, MD 20899 USA

8:30 AM Invited

Last Stage Solidification of Primary Phases: *Michel Rappaz*[1]; William J. Boettinger[2]; A. Jacot[1]; [1]EPFL, Matls. Dept., MXG, Lausanne CH-1015 Switzerland; [2]NIST, Metall. Div., Gaithersburg, MD 20899 USA

Solidification of metallic alloys has been extensively studied (dendrite tip kinetics, microsegregation, coarsening of dendrite arms, etc.) but surprisingly, little is known about the stage of solidification where the primary phase regions impinge. This impingement can occur between adjacent dendrite arms of the same grain or of different grains. It is a common occurrence in alloys containing no eutectic reaction such as Ni-Cu or in alloys containing a small volume fraction of second phase. Yet, this late stage of solidification is very important in relationship to hot-tearing. Hot-tearing is caused by a lack of tensile strength of the thin continuous liquid film that remains between dendrite arms up to very high volume fraction of solid. It is therefore important to know when dendrite arms bridge (or coalesce) to form a coherent solid that can sustain tensile stresses. The influence of the thickness of the liquid film on the interfacial equilibrium (i.e., disjoining pressure) is first considered for a pure material using a multi-phase field approach. The temperature at which the liquid film disappears as a function of the crystallographic misorientation (grain boundary energy) is determined. The dynamic predictions of the model and its extension to include solute diffusion will be described.

9:10 AM Invited

Crystal Growth of Compound Semiconductors: Current Issues: *David H. Matthiesen*[1]; [1]Case Western Reserve University, Matls. Sci. & Eng., 402 White Bldg., 10900 Euclid Ave., Cleveland, OH 44106 USA

The electronic property advantages of compound semiconductors over those of elemental semiconductors were recognized very early in the development of electronic devices. Teal and Little developed an economical technique to pull germanium and then silicon single crystals from the melt. Following the development of this 'Czochralski' (CZ) pulling technique, efforts were made to modify the CZ technique to overcome the challenge of the volatile nature of the group V and VI elements. These efforts culminated with the invention of the liquid encapsulated Czochralski (LEC) technique for the growth of gallium arsenide (GaAs). The LEC technique allowed for favorable economics and for the production of reproducible material. However, several deficiencies exist in LEC grown materials that are a direct consequence of the CZ development's legacy. Several innovations have been developed that seek to overcome these deficiencies, including: horizontal Bridgman (HB) technique; Vertical Gradient Freeze (VGF) technique; growth in microgravity; and direct growth of single crystal ternary compound from the melt.

9:50 AM Invited

Fundamental Properties of Bulk Metallic Glass Forming Melts: *William L. Johnson*[1]; [1]California Institute of Technology, Dept. of Matls. Sci., 138-78 Keck Lab., Pasadena, CA 91125 USA

The development of multicomponent "bulk" metallic glass forming alloys over the past decade has enabled direct study of the undercooled liquid alloys over the entire temperature range from the glass transition to the thermodynamic melting temperature (liquidus surface). Research on BMG alloys has included studies of rheology, atomic diffusion, thermodyanmic state functions (specific heat, entropy, molar volume, elastic modulii, etc.), the kinetics of crystal nucleation and growth from shallow to deep undercooling, and liquid/liquid phase separation. Experimental results have revealed a rich variety new phenomena including "strong" liquid behavior, decoupling of atomic diffusion constants from fluidity, and opening of complex miscibility gap structures in the undercooled melt. TTT- and CCT- diagrams suggest that crystallization kinetics are often controlled by liquid phase separation and other types of chemical clustering kinetics including the chemical clustering of impurities. Crystallization of the alloys at varying degrees of undercooling results in microstructures which span length scales from nanometers to millimeters. This offers new opportunities to produce partially crystallized materials with novel controlled microstructures and properties. The talk will present an overview and summary of work to date in this area.

10:30 AM Break

10:40 AM
Devitrification Reactions in Amorphization Al-Based Alloys: *John H. Perepezko*[1]; Robert I. Wu[1]; Rainer J. Hebert[1]; [1]University of Wisconsin–Madison, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA

Among different processing pathways for the formation of nanoscale microstructures, the devitrification of amorphous precursors yields the highest nanocrystal number densities. Amorphous alloys produced by solidification can be grouped into two categories. For nucleation controlled synthesis methods, bulk volumes are slowly cooled to the glassy state. Rapid solidification is on the other hand necessary for the second group of glasses, including Al- and Fe-based glasses. These marginal glasses are synthesized under growth controlled kinetic conditions. An analysis of the glass transition is difficult for these glasses since partial crystallization occurs within the same temperature range as the glass transition. As a result of the growth controlled processing, clusters of the order of 10 Angstroem are quenched in the as-spun ribbon samples but can despite of their small size be detected by small-angle X-ray scattering. In addition, the response to low-temperature annealing that has a major impact on the overall thermal stability and microstructure evolution has been investigated. The initial analysis of the primary crystallization reaction kinetics based upon measurements of the Al nanocrystal particle size distribution during isothermal annealing of Al-7Y-5Fe suggests volume dependent heterogeneous nucleation as the controlling mechanism. The nucleation process is also influenced by transient conditions before reaching steady state rates of about $5x10^{19}$ (m^{-3} s^{-1}). The heterogeneous catalysis has been modified by the incorporation of additional nucleant sites, enhancing the overall number density to $3x10^{22}m^{-3}$. Alternative synthesis strategies involving deformation processing by intense cold rolling reveal that the primary crystallization can be bypassed. These developments represent a major level of microstructure control that have an impact on the structural performance and stability. The support of the ARO (DAAG55-97-1-0261) is gratefully acknowledged.

11:00 AM
On the Nature of Amorphous Alloys Rapidly Solidified through Vapor Quenching in Immiscible Systems: *Evan Ma*[1]; Jinghao He[1]; Howard Sheng[1]; Paul J. Schilling[2]; [1]Johns Hopkins University, Matls. Sci. & Eng., 3400 N. Charles, Baltimore, MD 21218 USA; [2]University of New Orleans, Mechl. Eng., New Orleans, LA 70145 USA

Many binary systems have a positive free energy of mixing in the solid state and also in the liquid state. For such systems immiscible in equilibrium alloying in the solid state or by liquid quench is difficult. To create new alloys one can start from the vapor phase. However, the rapidly solidified alloys, unlike those in systems with a negative heat of mixing, must survive the large thermodynamic driving force for phase separation. The atomic-level structure actually attained, and the thermodynamic properties of the phases created, are therefore interesting but remain poorly understood. This talk discusses the nature of such amorphous alloys, using Ag-Ni as a model system. Our goal is to understand when and what kind of nonequilibrium phases can be alloyed on atomic scale in such a system with a large positive heat of mixing, and how different the amorphous alloys obtained are when compared with those well known in easy glass-forming systems, such as Ni-Zr and some bulk metallic glasses. To this end, we have carried out a detailed analysis of the atomic-level structures and directly correlated the structural features with the thermodynamic properties measured. Ag-Ni amorphous alloys have been prepared through the vapor quenching route. Evidence of amorphous phase formation has been obtained through a combination of x-ray diffraction (XRD), transmission electron microscopy (TEM), differential scanning calorimetry (DSC), extended x-ray absorption fine structure (EXAFS), and magnetic property measurements. The Ag-Ni amorphous alloys are found in unexpectedly low enthalpy states with low heat of crystallization and crystallization temperatures. EXAFS, combined with reverse Monte Carlo and molecular dynamics (MD) simulations, reveals spinodal-like structures on an extremely fine scale. A common neighbor analysis of the MD configurations indicates significant local icosahedral ordering, which is characteristic of many known amorphous/liquid structures and contributes to the further lowering of the amorphous energy state. Our results demonstrate that it is the nature of the unique atomic level structure that is directly and quantitatively responsible for the thermodynamic properties observed for these unconventional amorphous alloys.

11:20 AM
Concurrent Growth and Coarsening of Spheres: *Christoph Beckermann*[1]; Lorenz Ratke[2]; [1]The University of Iowa, Mechl. Eng., 2412 SC, Iowa City, IA 52242 USA; [2]German Aerospace Research Center, DLR, Inst. for Space Simulation, Cologne 51140 Germany

A theory is developed of concurrent growth and coarsening of a dispersion of spheres. It accounts for the simultaneous exchange of heat by diffusion among the spheres due to their curvature differences and between the spheres and the environment due to an externally imposed heat extraction rate. The results show that the average particle radius asymptotically increases with the cube root of time, which is the same behavior as in separate growth and coarsening. The growth rate constant increases with the heat extraction rate, from the LSW value in pure coarsening to a value that is 1.89 times larger when the heat extraction rate is equal to the sensible heat exchange rate between the spheres in coarsening. For larger heat extraction rates, coarsening has no effect on the average particle growth rate, and the particle number density remains constant.

11:40 AM
Morphological Evolution in Al-4.0wt%Cu Alloys under Diffusive Growth: *Shan Liu*[1]; Shuzu Lu[2]; Rohit K. Trivedi[3]; [1]Iowa State University, Ames Lab., USDOE, 232 Wilhelm Hall, Ames, IA 50011 USA; [2]Michigan Technological University, Dept. Metall. Matl. Eng., 511 M&ME Bldg., 1400 Townsend Dr., Houghton, MI 49931 USA; [3]Iowa State University, Ames Lab., USDOE, 100 Wilhelm Hall, Ames, IA 50010 USA

Directional solidification of Al-Cu alloys has been extensively conducted as a model system to compare with the theories/models. Generally different length scales are correlated with the controllable experimental parameters, (temperature gradient, growth velocity and composition). Most models assume that growth process is solely by diffusion control, however melt convection occurs inevitably in the bulk sample growth process on ground. Numerical simulation indicates that when the sample size is smaller than 1.0 mm, contribution from convection to mass transfer is less than 10% of that from diffusion and the solidification process is fundamentally controlled by diffusion. A technique has been developed to reduce the intensity of convection and grow Al-Cu samples diffusively. The shape of growth tips is analyzed by serial sectioning and compared with the prevailing model predictions. Morphological evolution in thin samples is studied and comparisons are made with the bulk samples where significant convection is present.

General Abstracts: Chemistry & Physics of Materials - I

Sponsored by: TMS
Program Organizers: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Wednesday AM Room: 102
November 7, 2001 Location: Indiana Convention Center

Chairperson: TBA

8:30 AM
Extraction of Zinc Metal from Zinc Ore using Pilot Plant Unit: *E. A. Abdel-Aal*[1]; E. H. El-Shennawy[1]; Z. E. Shukry[2]; [1]Central Metallurgical Research & Development Institute, PO Box 87, Helwan, Cairo, Egypt; [2]The Aluminum Company of Egypt (Egyptalum), Nag-Hammadi, Egypt

Extraction of zinc metal from zinc ore was carried out using a pilot plant of about 17 kg/hr zinc metal capacity. A computer program was developed for calculation of feeding and production rates. The results obtained show that recovery of about 87% of zinc is achieved using 100%-100 mesh ore particle size at a reaction temperature of 50°C for 3 hr total retention time and about 22% solid content. About 2/3 of sulfuric acid used was regenerated during electrolysis of zinc sulfate pregnant liquor. Material and zinc component mass balances for whole process were calculated. For production of 1 ton zinc metal, about 5.62 ton zinc ore, 0.95 ton 98% make-up sulfuric acid and 4.26 m3 wash water are required. In addition, the distributions of impurities were calculated. It was found that most of magnesium (about 96%) dissolves and transfers to zinc sulfate pregnant solution. Novel technique was applied for magnesium sulfate removal.

8:50 AM
Kinetics Study for Batch Leaching Process of Egyptian Zinc Silicate Ore: *E. A. Abdel-Aal*[1]; [1]Central Metallurgical Research & Development Institute, 87 Helwan, Cairo, Egypt

Kinetics results of batch process leaching of Egyptian low-grade zinc silicate ore from Zag-Elbahar area, Eastern Desert (Egypt) with sulfuric acid are presented. Effects of ore particle size, reaction temperature and acid concentration on zinc extraction rate were determined. The results obtained show that leaching of about 96% of zinc is achieved using -200+270 mesh ore particle size at a reaction temperature of 80°C for 180 min reaction time with 10% sulfuric acid concentration. The solid:liquid ratio was maintained constant at 1:20 g/ml. The results of leaching kinetics study indicate that diffusion through the product layer is the rate controlling process during the reaction. The activation energy was determined as about 3.4 kcal/mole which is characteristic for a diffusion controlled process.

9:10 AM
Directional Annealing of Cold-Rolled Copper Single Crystals With and Without SiO2 Particles: *J. Li*[1]; I. Baker[1]; H. J. Frost[1]; [1]Dartmouth College, Thayer Sch. of Eng., Hanover, NH 03755 USA

High-purity (112)[111] copper single crystals with and without fine silica particles were cold rolled to 80% thickness reduction and directionally annealed. For the pure copper, the effect of annealing temperature, hot zone velocity and temperature gradient on the microstructure were investigated. It was found that directional recrystallization did not occur when the velocity of the hot zone was slow or when it exceeded the maximum growth rate of grains, when equiaxed grains developed. When directional recrystallization occurred only the longitudinal grain size increased. Increasing the annealing temperature increased the optimum hot zone velocity for directional recrystallization. The number of twin boundaries per unit area generally decreased with increasing hot zone velocity at a given annealing temperature. Increasing the temperature gradient increased the grain size and decreased the number of twin boundaries per unit area. The silica particles had a marked effect on the kinetics of directional recrystallization.

9:30 AM Break

9:50 AM
Coarsening Kinetics of Gamma Prime Precipitates in Ni-Al Alloys: *Alan C. Lund*[1]; Peter W. Voorhees[1]; [1]Northwestern University, Matls. Sci., 2036 MLSB, 2225 N. Campus Dr., Evanston, IL 60208 USA

In γ-γ' alloys the misfit between the coherent precipitate and matrix phases generates elastic stress. These alloys were used to investigate the effects of elastic stresses on coarsening behavior. The γ' precipitates evolve from spheres, to cuboids, and finally to rod or plate-like morphologies as coarsening proceeds, so quantifying coarsening by measuring the evolution of an average spherical radius is not feasible in these microstructures. To avoid this problem, we measured the evolution of the precipitate-matrix interfacial area per unit volume, S_v. The inverse of this quantity provides a length scale of the microstructure that is independent of particle morphology. We will present results on the temporal exponent of $(S_v)^{-1}$ and on the dependence of the coarsening rate constant on the volume fraction of γ'.

10:10 AM
Substituted Garnets as Magneto-Optic Sensing Materials: *Pragati Mukhopadhyay*[1]; Gautam Mukhopadhyay[2]; Tara Chandra[3]; [1]Indian Institute of Technology, Adv. Ctr. for Rsrch. in Elect., Bombay 400076 India; [2]Indian Institute of Technology, Dept. of Phys., Bombay 400076 India; [3]University of Wollongong, Dept. of Matls. Eng., Wollongong, NSW 2522 Australia

Garnet films have been considered for a long time as good candidates for application in magnetic, microwave and magneto-optic (MO)-devices. In recent years, due to their inherent chemical and structural stability, they have emerged as potential MO materials compared to the more conventional but corrosion/oxidation prone RE-TM alloys. The crystal structure of garnets, with an ease to tailor many of its properties and the magnetic anisotropy present in them, makes them preferable over other materials. There have been efforts to improve the suitability of garnets as sensing materials by enhancing the magneto-optic properties through compositional modification. We report the magneto-optic and crystalline properties of bismuth and cerium substituted iron garnets and compare them with some other compositions.

10:30 AM
A Study of Effective Factors in Entrainment in Solvent Extraction Process: *Mahdi Molaei Nasab*[1]; Shahram Danesh Pajooh[1]; H. R. Hasani[1]; [1]National Iranian Copper Industries Company, Iran

The Sarcheshmeh Copper Complex is located in kerman province in central of Iran. In this factory we have a solvent extraction process, in this unit there are a lot of problem from entrainment. Research and development division tried to solve this problem. Mixer-settler are becoming increasingly used in the solvent extraction industry as a method of product of metals and the application of mixer-settler in copper industry is presented. The development and design of large scale for high volume of copper and low value of entrainment was desired. Theorical and experimental research in aqueous and organic phase in isoterm reactor shows relation of many factors such as pH, agitator(RPM), temperature, aqueous and organic continuity and amount percentage of extracting with entrainment. This problem was created in solvent extraction in Hydrometallurgy process. Results of investigation shows relation of entrainment with mixing, pH, percentage of extractant is direct and with temperature and different density is indirect.

10:50 AM
Investigation of Activation Energy and Thermodynamic Quantities of Methoxy Acetic Acid: *Mandalaparty Bhaskara Ramamurthy*[1]; [1]Multimedia University (Melaka Campus), Fac. of Eng. & Tech., Jalan Ayer Keroh Lama, Melaka 75450 Malaysia

Dielectric and ultrasonic velocity measurements are carried out on dilute solutions of methoxy acetic acid. The activation energy for a pair of solvents is determined by evaluating relaxation times. From ultrasonic velocity measurements certain thermodynamic quantities viz. adiabatic compressibility, Wada's number, Rao's constant etc. are calculated. The results are discussed.

General Abstracts: Non-Ferrous and Powder Metallurgy

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Wednesday AM Room: 206
November 7, 2001 Location: Indiana Convention Center

Session Chair: David L Bourell, The University of Texas at Austin, Matls. Sci. & Eng. Prog., Austin, TX 78705 USA

8:30 AM
Deposition of Compositionally Graded Unitized Structures from Pre-Alloyed Titanium Powders: *Peter Chancellor Collins*[1]; Gopal Babu Viswanathan[1]; David M. Keicher[2]; Richard Grylls[2]; Rajarshi Banerjee[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Optomec, Albuquerque, NM, USA

The concept of creating a unitized structure with localized properties is a very enticing concept to many industries. In the aerospace industry, such a structure would represent savings in weight and cost while improving the properties. A control of the compositional and microstructural variation is required in order to control the variation in properties. Controlled composition profiles have been deposited by direct laser deposition techniques, specifically laser engineered net-shaping (LENS). This technology couples a laser and powder into a dual focal point. Pre-alloyed powders of varying composition were melted under the moving laser beam to produce compositionally graded alloys. Ti alloy powders containing alloying elements such as Al, Sn, Zr and Mo in varying proportions were used to produce two phase (alpha+beta) alloys. Using similar methods, alloy powders containing Ti, Al, Cr and Nb were used to produce intermetallic alloys based on alpha2-Ti3Al and gamma-TiAl phases. These alloy systems were chosen not only for their industrial viability, but also their ability to display different types of phases and microstructures. As intended, in addition to compositional variation, these LENS processed alloys were invariably accompanied by a variety of microstructural features. The microstructural variation that accompanied the change in the composition in these graded alloys was studied in detail. A preliminary attempt has been made to predict these constituent phases by thermodynamic calculations. The results will be presented and discussed.

8:50 AM
Undercooling Behavior of Nanoscale Pb Droplets Encased in Amorphous $Cu_{48}T_{33}Zr_{10}Ni_8Si_1$: *James C. Foley*[1]; Francis C. Laabs[1]; [1]Ames Laboratory, Metall. & Cer., 122 Metals Dvlp., Ames, IA 50011 USA

The undercooling obtained during solidification is an important factor in determining the solidification pathway and related properties of most materials. One of the variables that exhibit a strong influence on the

WEDNESDAY AM

observed undercooling level is the size of the droplets. Arc-melted ingots of $Cu_{48}Ti_{33}Zr_{10}Ni_8Si_1$ was melt-spun with 1 and 10 weight percent 99.99% pure Pb to form a microstructure of amorphous $Cu_{48}Ti_{33}Zr_{10}Ni_8Si_1$ and nanoscale Pb. The undercooling behavior of nanoscale Pb droplets encased in an amorphous $Cu_{48}Ti_{33}Zr_{10}Ni_8Si_1$ ribbon was examined with differential thermal analysis (DSC), transmission electron microscopy (TEM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and chemical analysis. In contrast to research examining the undercooling of nanoscale Pb droplets encased in crystalline Al, the Pb droplets encased in amorphous $Cu_{48}Ti_{33}Zr_{10}Ni_8Si_1$ exhibited minimal (< 2 degrees) undercooling. The results of the alloy characterization techniques will be discussed along with possible mechanisms for the strong nucleation influence of the amorphous solid. The authors gratefully acknowledge the support received from USDOE-BES, Materials Science Division (contract no.W-7405-Eng-82).

9:10 AM

Elevated Temperature Mechanical Behavior of an Al-12.4Fe-1.2V-2.3Si Alloy: *Jeffrey A. Hawk*[1]; [1]US Department of Energy, Off. of Fossil Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA

Rapid solidification processing and powder metallurgy consolidation have led to high strength, high temperature dispersion strengthened aluminum alloys. This investigation examines the room and elevated temperature mechanical behavior of one dispersion strengthened aluminum alloy, an Al-Fe-V-Si alloy with approximately 37% by volume of a hard, complex silicide (-Al12(Fe,V)3Si). These silicide particles possess good microstructural stability up to 425°C, and as such, this alloy has good strength and adequate ductility up to this temperature (ultimate tensile strength (UTS) of 130 MPa at 450°C, and an elongation of 24%). Microstructural stability of the silicide particles leads to high strengths in the alloy after prolonged elevated temperature exposures (i.e., 540 MPa UTS after 100 hours at 500°C, 440 MPa UTS after 100 hours at 550°C, and 320 MPa UTS after 100 hours at 600°C). However, subtle microstructural changes in the location of the particle dispersion during these long term exposures reduces the elongations at fracture to about 1.0% after 100 hours at 600°C. Creep behavior of this material is also good with a Larson-Miller parameter of 30.9.

9:30 AM

Evolution of Dislocation Structures in 5182 Aluminum as a Function of Strain Rate and Temperature: *David Maurice*[1]; Karol K. Schrems[1]; Jeffrey A. Hawk[1]; [1]US Department of Energy, Off. of Fossil Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA

In order to better understand the evolution of dislocation structures in solid solution strengthened Al-Mg alloys (AA5182), a series of experiments were run at room and elevated temperature (525°C), and where the strain rate (0.25 to 50 mm/min crosshead displacement) was varied. Mechanical property data were collected for each series of tests to note any changes in either strength and ductility. The fracture surfaces of the alloy were also examined for changes in fracture morphology. Finally, transmission electron microscopy was conducted on each sample from an area near the fracture surface to document the dislocation structure that had formed as a result of the test conditions. These structures are compared to the initial, non-deformed microstructure.

9:50 AM

Particle Coarsening and Grain Growth in an Al-12.4Fe-1.2V-2.3Si Alloy: *Jeffrey A. Hawk*[1]; [1]US Department of Energy, Off. of Fossil Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA

Elevated temperature, high strength aluminum makes use of a small grain size and a dispersion of small intermetallic dispersoids to stabilize the microstructure. Reasons for the good elevated temperature strength of these materials include the coherency of the high volume fraction of small silicide particles in the aluminum matrix, low solute solubility of the intermetallic forming elements in the matrix, and relatively slow diffusion of these elements in aluminum. These factors lead to a stable microstructure where grain growth is minimal and the particle coarsening rate is low. Grain growth and -Al12(Fe,V)3Si particle coarsening rate are described for an Al-Fe-V-Si alloy with approximately 37% by volume of the strengthening dispersoid. In addition, the overall microstructural stability of the alloy is discussed with respect to long term (up to 1000 hours), high temperature (up to 600°C), isothermal heat treatments as evidenced by changes in hardness with respect to these experimental parameters. Comparisons with other dispersion strengthened aluminum alloys is presented.

10:10 AM Break

10:30 AM

Cast FeAl Alloys Show Improved Weldability: *Aszetta Denise Jordan*[1]; Oswald N.C. Uwakweh[2]; Phil Maziasz[3]; [1]Ratheon, Missile Sys./Eng., 1151 E. Hermans Rd., Tucson, AZ 85706 USA; [2]Consultant, 2396 E. Montana Ave., Cincinnati, OH 45211 USA; [3]Oak Ridge National Laboratory, Metals & Cer., Oak Ridge, TN 37831-6115 USA

FeAl iron-aluminides have outstanding resistance to corrosion and oxidation and are continuously being designed to enhance high temperature strength and room temperature ductility properties. But, can FeAl alloys be welded? And, do the elemental additions that improve the above properties enable them to survive cold cracking after welding? Gleeble simulations of weld cycles were conducted on cast FeAl alloys (Fe- 36 to 40 at. % Al) with additions of Mo, Zr, C and B. In this study, the complex cast FeAl alloy (i.e., Fe-Al-Mo-Zr-C-B) and a series of simpler alloys with each of the critical solutes removed to reach the equivalent binary FeAl alloy have been evaluated. The binary FeAl developed severe cold-cracking, with brittle fracture along the grain boundaries. By contrast, the complex alloy showed no such cracking behavior. Intermediate alloys with only Mo added to the binary FeAl do crack, however, further additions of B and C are resistant to crack formation. This study confirms that B and C together are critical elements for preventing grain-boundary fracture while increasing ductility at room temperature, both of which are needed for resistance to cold cracking during welding.

10:50 AM

The Low-Cycle Fatigue Behavior of Haynes Hastelloy® C-2000 Superalloy: *Robert L. McDaniels*[1]; Lija Chen[1]; Peter K. Liaw[1]; Roger Seeley[2]; Dwaine L. Klarstrom[2]; [1]University of Tennessee, Matls. Sci. & Eng., 434 Dougherty Engineering Bldg., Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The low-cycle fatigue behavior of the new Haynes Hastelloy® C-2000® nickel-chromium-molybdenum superalloy was investigated at total strain ranges from 0.4% to 2.0% and at temperatures ranging from 24°C to 927°C. The test specimens were subjected to fully reversed, push-pull, low-cycle fatigue tests under axial strain range control. The results indicated that both test temperature and total strain range had a significant effect on the low-cycle fatigue properties of the alloy. The alloy exhibited cyclic hardening, cyclic stability, or cyclic softening, depending on the temperature and strain to which the specimens were subjected. Coffin-Manson and Holloman parameters were also computed and plotted. Microstructual analysis of the as-received material and tested specimens was conducted using optical and scanning electron microscopy.

11:10 AM

Fabrication of P/M Processed Super High Strength Aluminum Alloy: *Kozo Osamura*[1]; Hiroki Adachi[1]; Jun Kusui[2]; Kazuhiko Yokoe[2]; [1]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan; [2]Toyo Aluminum K. K., R&D Lab., Hino, Shiga 529-16 Japan

Recently our group demonstrated the development of extremely high strength AlZnMgCu alloys (Mesoalite) by means of powder metallurgy. The air-atomized powder was canned into the aluminum container and then pressed under hydrostatic pressure of 392 MPa. After degassing, the compact piece was hot-extruded at 773K. The extrusion ratio was selected as 10 or 20. Several kinds of alloys including alloy elements of Zn, Mg, Cu, Mn and Ag were investigated. The present technique can provide a large scale product, for instance, the rod with dimension of 60 mm dia and 1000 mm length. As a standard procedure of heat treatment, the solution treatment was carried out at 763 K for 7.2ks and followed by water quenching. The standard condition of aging treatment as T6 was 393 K and 86.4 ks. A selected alloy (MESO20) with composition of Al- 9.5%Zn- 3.0%Mg- 1.5%Cu- 4.0%Mn- 0.04%Ag in mass% recorded tensile strength of 910 MPa and compressive strength of 1032MPa. The present super-high strength aluminum alloys could be achieved by the combination of several strengthening mechanisms; (1) fibre reinforcening, (2) fine grain strengthening and (3) precipitation hardening. Especially nano-scale coherent particles are very effective to achieve the high strength materials.

11:30 AM

Room Temperature Creep and Constant Strain Rate Behavior of Single Colony Ti-6242Si: *Joseph Michael Tatalovich*[1]; Marc Zupan[2]; Michael D. Uchic[3]; Kevin J. Hemker[2]; Michael J. Mills[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]The Johns Hopkins University, Mechl. Eng., Baltimore, MD 21218 USA; [3]Air Force Research Laboratory, Wright Patterson AFB, OH 45433-7817 USA

The constant strain rate and creep deformation behavior of individual alpha/beta colonies of the forgeable titanium aeroengine alloy Ti-6Al-2Sn-4Zr-2Mo-0.1Si (compositions in weight %) has been investigated through microsample testing techniques. The mechanical behavior and deformation mechanisms observed were found to depend strongly on colony orientation. A near-Burgers orientation relationship has been observed for the alpha (hcp) and beta (bcc) phases in these colonies. Scanning Electron Microscopy investigations of the slip line morphology of the deformed samples indicate an inhibition of beta-lath shearing for colonies oriented for basal slip, while frequent shear offsets of the beta phase by slip lines were observed in samples oriented for prism slip. Transmission Electron Microscopy is utilized to explain the behavior of each basal orientation. The observed tension/compression asymmetries will also be discussed.

11:50 AM
Interfacial Microstructure of Poly-Crystalline Alumina and Sapphire Joined using a Spin-On Interlayer: *Liang Zeng*[1]; Martin A. Crimp[1]; Eldon Case[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., E. Lansing, MI 48824-1226 USA

The interfacial microstructure of sapphire joined with poly-crystalline alumina has been evaluated. Joining was performed in a conventional electric furnace with the aid of a spin-on interlayer, SilicafilmTM. A few drops of the silica film were applied to the polished surfaces of the sapphire and poly-crystalline alumina plates and then spun on a high speed substrate spinner. The films were then cured at 200°C for 20 minutes to yield a high purity silica coating approximately 200 nm thick. The sapphire and poly-crystalline alumina were then joined at elevated temperatures under a small load of a 85 gram dead weight which corresponds to 8.5 KPa. Using these interlayers, sapphire and poly-crystalline alumina were successfully joined at temperatures from 1400°C to 1475°C for 3 hours. In contrast, two control samples without silica interlayers failed to join under these conditions. Cross-sectional optical, scanning electron, and transmission electron microscopy were used to examine the interfacial regions of the joints. A variety of interfacial morphologies, including amorphous regions and fine crystalline phases, have been observed. With the increasing joining temperature and time, the thickness of the interfacial reaction layer steadily decreased. This microstructural development will be discussed.

Lead-Free, Lead-Bearing Solders & Alternative Surface Finishes for Electronic Packaging - II: Mechanical Properties, Fatigue, Creep

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee
Program Organizers: C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Srini Chada, Motorola, Department APTC, Fort Lauderdale, FL 33322 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu City 00143 Taiwan; Gautam Ghosh, Northwestern University, Department of Materials Science, Evanston, IL 60208-3108 USA; Prasad Godavarti, FlipChip Technologies, Phoenix, AZ 85034 USA; Zequn Mei, Agilent Technologies, Inc., Palo Alto, CA 94304-1392 USA

Wednesday AM   Room: 202
November 7, 2001   Location: Indiana Convention Center

Session Chairs: K. N. Subramanian, Michigan State University, Dept. of Matls. Sci. & Mech., E. Lansing, MI 48824 USA; Nikhilesh Chawla, Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287 USA

8:30 AM Invited
Microstructural Characterization of Damages in Thermomechanically Fatigued Sn-Ag Based Solder Joints: *K. N. Subramanian*[1]; J. G. Lee[1]; S. Choi[1]; T. R. Bieler[1]; J. P. Lucas[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824-1226 USA

The solder joints experience the interaction of creep and fatigue under reversed shear, termed as thermomechanical fatigue (TMF), under service conditions. It is essential to employ realistic service conditions during TMF testing in order to reveal the true damage that the solder joints undergo during service. In this study, the solder joints were subjected to temperature fluctuations representing commuting-to-work type condition for automotive under-the-hood circuit board applications. Solder joints fabricated with eutectic Sn-Ag, and Sn-Ag solders with Cu and/or Ni were used for this study. Characterization of the damage in these solder joints induced by TMF was carried out after 0, 250, 500, and 1000 cycles. The effects of alloying elements on TMF-induced damage accumulation in the solder joints will be discussed. Work supported by the National Science Foundation under grant NSF DMR-0081796.

9:00 AM
Tensile and Creep Behavior of Lead-Free Solder Alloys: Jason J. Williams[1]; George Piotrowski[1]; Ted Martin[1]; *Nikhilesh Chawla*[1]; Fay Hua[2]; [1]Arizona State University, Cheml. & Matls. Eng., Tempe, AZ 85287 USA; [2]Intel Corporation, 2200 Mission College Blvd., Santa Clara, CA 95052 USA

The tensile and creep behavior of Sn-Ag, Sn-Cu, and Sn-Ag-Cu were examined and compared to that of conventional Pb-Sn alloys. The effect of cooling rate on the microstructure and mechanical behavior of the alloys was examined. Solder-Cu joints were also fabricated. Microstructural evolution of the solder joints was characterized and will be presented. The implications of intermetallic thickness and solder/substrate interactions under tensile and creep conditions will be discussed. Research sponsored by the National Science Foundation, under contract# DMR-0092530, and Intel Corp.

9:20 AM
Residual-Mechanical Behavior of Themomechanically Fatigued Sn-Ag Based Solders: *J. G. Lee*[1]; S. Choi[1]; K. N. Subramanian[1]; T. R. Bieler[1]; J. P. Lucas[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824-1226 USA

The solder joints are subjected to thermal cycling due to the temperature fluctuations encountered in service. Thermomechanical fatigue (TMF) due to the mismatch in coefficients of thermal expansion between solder and substrate gradually degrades the mechanical properties of solder joint during thermal cycling. This study investigates residual-mechanical behavior of solder joints made with eutectic Sn-Ag solder and Sn-Ag solder with Cu and/or Ni. These joints were subject to a thermal cycling from -15°C to 150°C with dwell times of 25 and 330 minutes at high and low temperature extremes, respectively. Tensile and creep deformation testing were conducted on solder joints that underwent 0, 250, 500, and 1000 TMF cycles. Residual-mechanical properties and fracture behaviors of these solder are analyzed on the basis of the composition. Work supported by the National Science Foundation under grant NSF DMR-0081796.

9:40 AM
Deformation Characteristics of Sn-Ag Solder Joints that Experience Stress Relaxation and Reverse Shear Stress: *Christine Alice Lonskey*[1]; A. Telang[1]; K. N. Subramanian[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824-1226 USA

In electronic circuits CTE mismatches present between the components of solder joint create opposing stress states during heating and cooling experienced in service. During the dwell periods at the temperature extremes, stress relaxation/creep takes place. Since it will be difficult to impose reversed shear stress on realistic electronic solder joints by external loading, studies were carried out using 3/8"X3/8"X1" copper blocks joined with about 400 Ým thick Sn-Ag solder. Effects of imposed shear stress along with stress relaxation on the deformation characteristics of the solder were investigated, so as to gain a better understanding of damage accumulation during thermomechanical fatigue. Work supported by National Science Foundation under grant NSF DMR-0081796.

10:00 AM Break

10:20 AM Invited
Mechanical Behavior of Sn-In-Ag Solder Joints: *Fay Hua*[1]; [1]Intel Corporation, SC1-03, 3065 Bowers Ave., Santa Clara, CA 95054 USA

Sn-Ag-Cu alloys are recommended by industry for eutectic Sn-Pb solder replacement. Higher melting point of the Sn-Ag-Cu alloys (~220°C) over eutectic SnPb solder causes problems in the process where multiple step soldering is required. There is a need in finding solders with melting temperature about 200°C. Several Sn-In-Ag solders melt around 200°C. However, very limited mechanical properties were reported. This paper reports the results of shear and creep behavior of the Sn-In-Ag solder joints. Microstructure evaluation of the solder joints is characterized. Solder/substrate interaction are also studied.

10:50 AM
Orientation Imaging of Thermomechanically Fatigued Single Shear Lap Solder: *Adwait Uday Telang*[1]; T. R. Bieler[1]; J. G. Lee[1]; S. Choi[1]; K. N. Subramanian[1]; [1]Michigan State University, Dept. of Matl. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824 USA

Single shear lap specimens with 1 mm^2 area and 80-160 micron thick on copper substrates were thermomechanically cycled from -15 to 150°C for 0, 250, 500, and 1000 cycles. Electron microscopy conducted after 1000 cycles showed shear bands and other surface morphology developed on the originally polished surfaces. To compliment the surface morphology changes, orientation imaging of selected regions of these specimens will be conducted to determine how the microstructure and crystal orientations were altered by thermomechanical cycling. Statistical issues relevant to characterizing the crystallographic texture as well as techniques used for surface preparation to obtain suitable electron backscattered patterns will be discussed. This work is supported by the National Science Foundation under grant NSF DMR-0081796.

11:10 AM
Micromechanical Characterization of Thermomechanically Fatigued Lead-Free Solder Joints: *H. Rhee*[1]; M. Moeller[1]; J. P. Lucas[1]; K.

N. Subramanian[1]; T. R. Bieler[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824-1226 USA

Nanoindentation testing (NIT) was carried out to investigate micromechanical properties of thermomechanically fatigued (TMF) lead-free solder joints. Micromechanical characterization was carried out on solder joints which were thermomechanically fatigued between -15°C and 150°C for 0, 250, 500, and 1000 cycles. The solder joints investigated were fabricated by joining copper substrates with eutectic Sn-Ag solder and Cu containing Sn-Ag solders with or without Ni. Using NIT, micromechanical properties of the various constituent phases of the solder joints were determined. Particular emphasis was focussed on assessing the micromechanical properties of the intermetallic compound (IMC) layer formed at the Cu substrate/solder interfaces. Specifically, micromechanical properties such as hardness, elastic modulus, steady-state creep rate, and stress exponent in creep were determined for constituent phases of the TMF solder joints. Work supported by the National Science Foundation under grant NSF DMR-0081796.

11:30 AM

Low Flux High Temperature Composite Solders Processed by Sintering: *Mark A. Palmer*[1]; [1]Kettering University, Mfg. Eng., 1700 W. Third Ave., Flint, MI 48504-4898 USA

Solder joints have been prepared from metal reinforced alloys based on eutectic Sn-Ag-Bi, Sn-Ag-Cu and Sn-Ag at temperatures less than 215°C. It was found that small amounts of copper, silver or other metal powder reinforcement can further strengthen the joint. It was also found that through this solder state process the amount of flux can be substantially reduced. The mechancial characterization of solder joints prepared by this method will be discussed. This work was supported by the National Science Foundation.

Materials Design Approaches and Experiences - IV

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: Ji-Cheng Zhao, GE Corporate Research & Development, Physical Metallurgy Laboratory, Schenectady, NY 12301 USA; Michael G. Fahrmann, Special Metals Corporation, Technology Department, Huntington, WV 25705-1771 USA; Tresa M. Pollock, University of Michigan, Materials Science and Engineering Department, Ann Arbor, MI 48109-2136 USA

Wednesday AM — Room: 204
November 7, 2001 — Location: Indiana Convention Center

Session Chair: Michael Fahrmann, Special Metals Corporation, Tech. Dept., Huntington, WV 25705-1771 USA

8:30 AM Invited

Computer Modelling of Materials Properties: *Nigel Saunders*[1]; Xiuqing Li[1]; Alfred Peter Miodownik[1]; Jean-Phillipe Schille[1]; [1]Thermotech, Ltd., Surrey Technology Centre, The Surrey Research Park, Guildford, Surrey GU2 7YG UK

Work is being undertaken at Thermotech on the development of a new multi-platform software programme for predicting a wide range of Materials Properties. These properties include thermo-physical and physical properties, mechanical properties, TTT/CCT diagrams etc. and the calculations are being applied to a variety of multi-component alloy types; such as Ni-based superalloys, steels, Ti-alloys, Al-alloys. The new programme is particularly aimed at multi-component alloys used in industrial practice and numerous examples of calculated results for the various properties will be presented with the emphasis being on validation of calculated result against experimental observation.

9:00 AM Invited

Development of Timetal® 834: *Don F. Neal*[1]; [1]Timet UK, Quality & Tech., PO Box 704, Witton, Birmingham, W. Midlands B6 7UR UK

Timetal® 834 has the highest temperature capability of any commercial titanium alloy. It is the culmination of around 50 years of titanium alloy development which has increased temperature capability of from ~300°C (572°F) to ~600°C (1112°F). The paper will describe how the pivotal discovery of the effect of carbon on the phase transformation in Ti, together with an optimised combination of other elements, has maximised creep and fatigue performance. The paper will begin with a short historical review, followed by a consideration of the very large number of possible combinations of alloying elements in multi-component systems. The considerable number of required physical and mechanical properties will also be described. The effect of individual elements on properties, and the need for attention to detail, plus some of the problems encountered, will be described. Finally, the need to combine all of the above with production and commercial viability will be emphasised.

9:30 AM Invited

The Development of a New Welding Product for Joining Silicon-Molybdenum Ductile Iron Exhaust Manifolds to Stainless Steel Converter Housings: *Brian A. Baker*[1]; Gaylord D. Smith[1]; Samuel D. Kiser[2]; [1]Special Metals Corporation, R&D, 3200 Riverside Dr., Huntington, WV 25705 USA; [2]Inco Alloys International Welding Products, Sales, 1401 Burris Rd., Newton, NC 28658 USA

This paper outlines the development of a new welding product for use in welding silicon-molybdenum ductile iron engine exhaust manifolds to ferritic stainless steel catalyst cans. An exisiting Ni-Fe-Mn welding product had been previously used with success in this application. More demanding service conditions, however, began to produce cracking in the heat-affected-zone (HAZ) of the ductile iron manifold and limit service life. This phenomenon has been linked to the presence of aligned secondary graphite within the ductile iron HAZ and stress-accelerated oxidation cracking along the aligned graphite particles. Termo-Calc® software was used to facilitate the development of a Ni-Fe-Mn-Cr-Cb welding product which possesses a more stable microstructure and serves also to enhance the resistance of the ductile iron HAZ to cracking in service.

10:00 AM Invited

Alloy Design Considerations for Braze Filler Metals: *John J. Stephens*[1]; F. Michael Hosking[1]; [1]Sandia National Laboratories, 1833, PO Box 5800, MS0889, Albuquerque, NM 87185-0889 USA

This talk is motivated by the consideration that the elevated temperature mechanical properties of braze alloys used in metal/ceramic brazing are inherently different than those intended for metal/metal joining. Braze alloys used for metal/ceramic brazing are expected to deform readily during the cooldown from the braze cycle to accommodate thermal expansion mismatch between the dissimilar materials joined. Another requirement for braze alloys used in metal/ceramic brazing is that ordering reactions, if present, do not result in extensive alloy strengthening. By contrast, braze alloys used for metal/metal structural applications should have excellent high temperature mechanical properties–both from a strength and ductility viewpoint. These diverging design requirements will be discussed in view of the high temperature mechanical properties of the AWS BAu-4 (82wt.%Au-18Ni) alloy and its active metal alloy (82Au-15.5Ni-0.75Mo-1.75V) counterpart. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Dept. of Energy under Contract DE-AC04-94AL85000.

10:20 AM Break

10:40 AM Invited

The CALPHAD Approach in the Focused Design of Magnesium Alloys: Joachim Groebner[1]; Dmytro Kevorkov[1]; *Rainer Schmid-Fetzer*[1]; [1]TU Clausthal, Institut fuer Metallurgie, Robert-Koch-Str. 42, Clausthal-Zellerfeld D-38678 Germany

In traditional alloy development experimental investigations with many different compositions are performed. The selection criteria for multi-component alloying elements and their compositions become diffuse in the traditional approach. Computational thermochemistry can provide a clear guideline for such selections and helps to avoid long-term experiments with less promising alloys. It enables the calculation of multicomponent phase diagrams and tracking phase distributions and phase compositions of individual alloys during heat treatment and solidification. These are the basic data to understand and control the behavior of any novel or modified Mg-alloy. Thus it is a powerful tool to cut down on cost and time during development of Mg-alloys. We report on recent applications and progress in construction of the necessary thermodynamic database for several promising alloying elements like Al, Li, Si, Mn, Ca, Sc, Y, Zr and Rare Earths, using the Calphad method combined with key experiments.

11:10 AM

Characterization of Superalloy Tertiary Creep by Inverse Modelling: *Martin A. Rist*[1]; Roger C. Reed[1]; [1]University of Cambridge/Rolls-Royce University Technology Centre, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

The design of new alloys for high-temperature engineering applications frequently requires the rapid characterisation of material mechanical behaviour, particularly creep resistance. Such information is ordinarily extracted by conducting a series of time-consuming and costly uniform loading experiments. To expedite material prototyping we have developed a technique that allows estimation of the stress-dependence of material behaviour from a single, short-duration, tensile test performed under non-uniform stress. The approach involves inverse modelling of experiments conducted using a novel tensile testpiece with a concave gauge-length profile. Temporal strain and post-deformation spatial strain distribution are simulated using a well-founded mechanistic creep damage model, and agreement between model results and experimental data is optimised by systematic perturbation of model parameters. This inverse strategy has been validated by examining high temperature tertiary creep in three generations of nickel superalloy single crystal materials, but has wider application to materials characterisation generally.

11:30 AM

Solidification of High Ru+Re Superalloy Single Crystals: *Q. Feng*[1]; S. Tin[1]; A. Gungor[2]; T. M. Pollock[1]; [1]University of Michigan, Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; [2]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., Pittsburgh, PA 15213 USA

Continued improvements in the high temperature capabilities of superalloy single crystals have led to increasingly higher levels of refractory alloying additions, such as Re and Ru. The design of new alloys will require information on the solidification characteristics of alloys with high levels of these alloying additions. In this study, the solidification behavior of several ternary and high order model nickel-base superalloys has been investigated. The model alloys contains very high level of Ru (4~10 wt.%) and Re (4~8 wt.%). Using X-ray diffraction and electron microscopy, a Re-Ru-rich δ phase was identified in as-cast alloys which contained high levels of Re and Al along with low Ru. This phase has hexagonal crystal structure with the space group of $P6_3/mmc$. The δ phase nucleates first during solidification and results in the breakdown of single crystal solidification. Ru additions substantially reduce the temperature at which the δ phase precipitates during solidification. The segregation of refractory elements, such as Re, W and Ta, was not reduced by high levels of Ru additions.

11:50 AM

The Tensile Properties of Disk Superalloys during Supersolvus Quenching Heat Treatments: *Tim Gabb*[1]; John Gayda[1]; Peter Kantzos[3]; [1]NASA Glenn Research Center, 21000 Brookpark Rd., MS 49-3, Cleveland, OH 44135 USA; [3]Ohio Aerospace Institute, 21000 Brookpark Rd., MS 49-7, Cleveland, OH 44135 USA

There is a need to increase the temperature capabilities of superalloy turbine disks. This would allow full utilization of higher temperature combustor and airfoil concepts under development. One approach to meet this goal is to modify the processing and chemistry of advanced alloys, while preserving the ability to use rapid cooling supersolvus heat treatments to achieve coarse grain, fine gamma prime microstructures. An important step in this effort is to understand the key high temperature tensile properties of advanced alloys as they exist during supersolvus heat treatments. This could help in projecting cracking tendencies of disks during quenches from supersolvus heat treatments. The objective of this study was to study the tensile properties of advanced disk superalloys during simulated quenching heat treatments. Specimens were cooled from the solution heat treatment temperatures at controlled rates, interrupted, and immediately tensile tested at various temperatures. The responses and failure modes were compared and related to the quench cracking tendencies of small disk forgings.

Microstructure Modeling and Prediction During Thermomechanical Processing - IV

Sponsored by: Structural Materials Division, Materials Processing & Manufacturing Division, Shaping and Forming Committee, Titanium Committee

Program Organizers: Raghavan Srinivasan, Wright State University, Department of Mechanical & Materials Engineering, Dayton, OH 45435 USA; Armand J. Beaudoin, University of Illinois at Urbana–Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Steve P. Fox, Timet Corporation, Timet Henderson Technical Laboratory, Henderson, NV 89009 USA; Zhe Jin, Reynolds Metals Company, Metallurgy Department, Chester, VA 23836-3122 USA; S. L. Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Wednesday AM Room: 208
November 7, 2001 Location: Indiana Convention Center

Session Chairs: Dan Backman, GE–Aircraft Engines, Lynn, MA 01910-0001 USA; Stephen Fox, Timet Corporation, Timet Henderson Techl. Lab., Henderson, NV 89009 USA

8:30 AM

Strain Hardening of Titanium: Role of Deformation Twinning: *Ayman A. Salem*[1]; Surya R. Kalidindi[1]; Roger D. Doherty[1]; [1]Drexel University, Dept. of Matls. Eng., Philadelphia, PA 19104 USA

The purpose of this study is to investigate the role of deformation twinning in the strain-hardening behavior of high purity, polycrystalline α-titanium in a number of different deformation modes. Constant strain rate tests were conducted on this material in simple compression, plane-strain compression and simple shear, and the true stress (σ)-true strain (ε) responses were documented. From the measured data, the strain hardening rates were numerically computed, normalized with shear modulus (G), and plotted against both normalized stress and ε. These normalized strain hardening plots exhibited three distinct stages of strain hardening that were remarkably similar to those observed in previous studies on low stacking fault energy fcc metals in which deformation twinning has been known to play an important role. The evolution of the microstructure under different deformation modes was documented and the onset of deformation twinning correlated with a sudden increase in strain hardening rate in compression tests.

8:55 AM

Development of Damage Maps for Ti-6Al-4V Processing: *Vasisht Venkatesh*[1]; Stephen P. Fox[1]; [1]TIMET, R&D, PO Box 2128, Henderson, NV 89009 USA

Primary processing of titanium alloys involves several forging, cooling and re-heating stages of the billet, during which time the microstructure evolves along with the process conditions. Occasionally process conditions, i.e., temperature, strain, strain rate and stress state, developed across the section of the billet can lead to surface and/or internal cracking. Optimization of existing process routes can minimize such occurrences resulting in a significant cost reduction, especially for widely used aerospace alloys like Ti-6Al-4V. A methodology for mapping safe and unsafe process regimes utilizing DEFORM 2D and 3D finite element analysis (FEA) codes in conjunction with the Cockroft-Latham (C-L) ductile fracture criteria will be presented. In addition, the effect of starting microstructure on workability limits, and experimental validation of FEA predictions will also be discussed.

9:20 AM

Prediction of Development of Microstructure in Alpha/Beta Ti Alloys using Rules-Based Approaches: Eunha Lee[1]; Sundar Amancherla[1]; Gopal B. Viswanathan[1]; Rajarshi Banerjee[1]; *Hamish L. Fraser*[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Two approaches to the modeling of the development of microstructure in alpha/beta Ti alloys have been employed, one involving phase field models and the second the application of neural networks. In this paper, the latter approach is described. A number of steps have been involved, including the establishing of a convention for describing the various microstructural features, the development of a database for the alloy TIMETAL 550 relating microstructure and phase composition to heat-treatment, and the development of neural networks based on this database. At first, colony and basketweave microstructures corresponding to beta-forged material will be used as initial conditions, but the various microstructural length scales and phase proportions are being varied systematically by prior heat-treatment. In this way, a means of predicting microstructure resulting from a multi-step heat-treatment in this alloy system should be possible. The predictive capability will be validated by experimental, multi-step, heat-treatments. This research is supported by the National Science Foundation, DMR-0080766.

9:45 AM

Grain Growth Phenomena in Ti-15V-3Cr-3Al-3Sn Sheet: *A. Roussel*[2]; I. Duta[1]; H. J. Rack[1]; [1]Clemson University, Cer. & Matls. Eng., 110 Olin Hall, Clemson, SC 29634-0907 USA; [2]Allvac, An Allegheny Technologies Company, 2020 Ashcraft Ave., PO Box 5030, Monroe, NC 28111-5030 USA

This study has examined the grain growth behavior of cold-rolled and fully recrystallized Ti-15V-3Cr-3Al-3Sn sheet. Exposure times between 1 and 32 minutes at four (4) temperatures between 1088 and 1163K were considered. Two regimes of grain growth were observed. Normal grain growth, wherein the grain size distribution was log normal and the mean grain size increased with the square-root of time, was observed at short times and/or low temperatures. At longer times and/or higher temperatures, abnormal grain growth was observed. Under the latter conditions, the mean grain size appeared to be invariant with time while the grain size distribution became increasingly bimodal with increasing time and/or temperature. This presentation will show that this transition in grain growth behavior from normal to abnormal grain growth may be correlated with a change in the distribution of grain boundary mis-orientations. Electron-back scatter diffraction suggested that normal grain growth in Ti-15V-3Cr-3Al-3Sn was associated with a predominance of the high-angle boundaries whereas abnormal grain growth was associated with a broadening of the grain boundary mis-orientation distribution.

10:10 AM Break

10:25 AM
Microstructure Evolution during Equal Channel Angular Extrusion (ECAE) Processing of Titanium Aluminides: *Shankar M.L. Sastry*[1]; Prabhu S. Raghunathan[1]; Rabindra Nath Mahapatra[2]; [1]Washington University, Mechl. Eng., CB 1185, One Brookings Dr., St. Louis, MO 63130 USA; [2]NAVAIRSYSCOM, Matls., Bldg. 2188, Rm. 102, 48066 Shaw Rd., Patuxent River, MD 20670 USA

Gamma-titanium aluminides can be deformed to large strains using equal channel angular extrusion (ECAE) at temperatures below the eutectoid isotherm. Such a method provides the flexibility for producing greater microstructural control than hitherto has been possible. For example alloys with lammellaer gamma+alpha2 microstructure can be deformed to large strains at as low temperature as 1000°C and transformed to fine grained equiaxed microstructure. The ECAE processing consists of extruding a well-lubricated billet through two intersecting channels of identical cross-section. Deformation is achieved by a simple shear in a thin layer at the crossing plane of the channels. The process can be repeated a number of times using the same tool because the billet cross-section remains constant. The microstructural evolution during ECAE processing of single phase gamma two phase gamma+alpha2 titanium aluminides will be discussed.

10:50 AM
The Microstructure Prediction Model of Forged U720LI: *Satoshi Takahashi*[1]; [1]Ishikawajima-Harima Heavy Industries Company, Ltd., Rsrch. Lab., 3-1-15, Toyosu, Koto-ku, Tokyo 135-8732 Japan

In general, the microstructure formed during die forging process strongly influences on the mechanical properties. In order to understand the high temperature deformation behavior of cast/wrought U720LI, the microstructures of the various compressed specimens were investigated. According to experimental results on the dynamic recrystallization by using Zener-Hollomon parameters, there will be the possibility of predicting the microstructures and of developing the model for the optimization of each manufacturing process. Then, we have considered the effects of the initial grain size and each forging condition, namely, total strain, strain rate and temperature at local area, on microstructure. Our model was evaluated through the disk forging under the production scale. This result will be mentioned.

11:15 AM
Mechanisms of Grain Refinement and Superplastic Behavior of Alloy 718: *B. P. Bewlay*[1]; V. A. Valitov[2]; O. A. Kaibyshev[2]; Sh. Kh. Mukhtarov[2]; C. U. Hardwicke[1]; M. F.X. Gigliotti[1]; [1]General Electric Corporate Research and Development, PO Box 8, Schenectady, NY 12301 USA; [2]Institute for Metals Superplasticity Problems, Ufa 450001 Russia

This paper will describe the mechanisms of grain refinement of alloy 718 billet to microcrystalline, submicrocrystalline, and nanocrystalline structures using high-strain thermomechanical processing. Dynamic recrystallization can be employed during high-strain hot deformation to form microcrystalline structure with grain sizes of <5mm. The roles of both dynamic and static recrystallization during grain refinement will be described. A submicrocrystalline structure can also be formed in alloy 718 using high strain plastic deformation in the temperature range 0.67-0.60Tm. During recrystallization the microcrystalline structure transforms to submicrocrystalline structure. Further reduction in the processing temperature to <0.5Tm provides additional refinement of the microstructure to a grain size less than 0.1mm. The influence of the initial grain size on deformation mechanisms during subsequent superplastic deformation of alloy 718 will also be described. Microcrystalline microstructures are very important for alloy 718 because its flow stress is higher than that for other nickel base alloys, and this makes forming complex shapes difficult. This paper will also describe thermomechanical processing regimes for generating microcrystalline structures in large-scale preforms of alloy 718 that were used for superplastic forming.

Modeling the Performance of Engineering Structural Materials - IV: Processing

Sponsored by: Structural Materials Division, Structural Materials Committee
Program Organizers: Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA

Wednesday AM Room: 140
November 7, 2001 Location: Indiana Convention Center

Session Chairs: A. M. Gokhale, Georgia Institute of Technology, Matls. Sci. & Eng., Atlanta, GA 30332-0245 USA; G. M. Michal, Case Western Reserve University, Dept. of Metal & Matl. Sci., Cleveland, OH 44106 USA

8:30 AM Invited
Using Severe Plastic Deformation for the Processing of Structural Metals: *Terence G. Langdon*[1]; Minoru Furukawa[2]; Zenji Horita[3]; [1]University of Southern California, Dept. of Aeros. & Mechl. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA; [2]Fukuoka University of Education, Dept. of Tech., Munakata, Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Eng., Fukuoka 812-8581 Japan

The grain sizes of structural metals may be substantially refined, typically to the submicrometer or nanometer range, by imposing severe plastic deformation in processes such as High-Pressure Torsion (HPT) and Equal-Channel Angular Pressing (ECAP). This paper describes the principles of ECAP and illustrates the application of this procedure to representative alloys. Several factors influence the microstructures attained in ECAP including (i) the channel angle contained within the ECAP die, (ii) the processing route incorporating the rotation of samples between consecutive pressings, (iii) the speed of pressing and (iv) the temperature at which the pressing is conducted. These various factors are discussed and illustrated with experimental examples.

9:00 AM Invited
Structural Sensitivity of the Mechanical Properties in High Deformed Fe-Armco after Equal Channel Angular Pressure: *Mykola Danylenko*[1]; [1]Institute of Problems for Materials Science, 3, Krzhizhanovsky, Kyiv, Ukraine

High degree deformation structure was obtained in Fe-Armco by the equal channel angular pressure method. The effect of the deformation degrees on dislocations and cells structure evolution was analyzed. The structural sensitivity of the mechanical properties in the high-deformed Fe-Armco was studied. The change of mechanical behavior of deformed materials near critical deformation takes place: a) the parabolic strain hardening which was observed at the low and average deformation degrees was changed by the linear stage of deformation at high deformation degrees; b) the growth of the fracture toughness for specimens with cracks introduced into the plane perpendicular to the plane of deformation was shown; c) decreasing of the fracture characteristic for specimens with cracks introduced into the plane parallel to the plane of deformation was observed.

9:30 AM
Equal Channel Angular Processing of Magnesium Alloys: *S. R. Agnew*[1]; G. M. Stoica[2]; L. Chen[2]; T. M. Lillo[3]; J. Macheret[3]; Y. Lu[2]; P. K. Liaw[2]; [1]Oak Ridge National Laboratory, Oak Ridge, TN, USA; [2]University of Tennessee, Knoxville, TN, USA; [3]Idaho National Engineering and Environmental Laboratory, Idaho Falls, ID, USA

Equal channel angular (ECA) processing offers the potential to introduce very large strains into a workpiece without changing its cross-section. Hence, it is an attractive technique for developing homogeneous fine-grained metals with good forming characteristics. Magnesium alloys are ideal candidates to benefit from the ECA process, because they exhibit poor low temperature forming characteristics due to a hexagonal close packed crystal structure. Magnesium has a low melting point, which enables its alloys to be processed isothermally at temperatures of 325°C and below. An assessment of the technique has been made with three commercial magnesium alloys ZK60, WE43 and AZ31 and one experimental alloy Mg-4wt%Li. The ductilities of the former two alloys, which contain Zr, were substantially improved. The latter two alloys, which do not contain Zr, showed little change in their properties for the processing conditions explored. Relative to the as-received material, the ductility of ZK60 was improved by 2 to 3 times (up to 220% elongation) over the entire temperature range investigated (24-450°C). Explanations for the property enhancements are discussed in terms of microstructure observations and deformation mechanisms.

9:55 AM
Deformation Characteristics of Al- and Mg- Alloys Obtained by Equal Channel Angular Processing: Grigoreta Mihaela Stoica[1]; Lijia Chen[1]; Eduard Andrew Payzant[2]; Sean R. Agnew[2]; Yulin Lu[1]; B. Han[3]; T. G. Langdon[3]; Peter K. Liaw[1]; [1]The University of Tennessee, Matl. Sci. & Eng., 323 Dougherty Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., 1 Bethel Rd., Oak Ridge, TN 37831-6115 USA; [3]The University of Southern California, Matls. Sci. & Eng., Los Angeles, CA 90089 USA

Equal-Channel-Angular Processing (ECAP) was used to refine the microstructures of three lightweight structural materials: Mg-alloy ZK 60, Al-alloy 6061, and an Al-matrix-composite 6061+10% vol. Al_2O_3 (10 µm) particles. In order to investigate the impact of severe plastic deformation during ECAP upon microstructures, optical microscopy and X-ray diffraction were employed. The microstructural properties, including grain and sub-grain size, elastic microstrain, and crystallographic texture, are discussed in light of the ECAP deformation history. The changes of mechanical behavior (tensile and cyclic fatigue) were also determined. The study of tensile behavior focuses on ductility enhancement as a function of the number of passes and ECAP route, attempting to correlate the macroscopic behavior with the microstructural evolution. Finally, results obtained on the texture development during high-cyclic fatigue tests of the Al-alloy 6061, and 6061+Al_2O_3 composite are discussed. Acknowledgements: We very much appreciate the financial support of the University of Tennessee (UT) Scholarly Activities Research Incentive Fund (SARIF), and the National Science Foundation (NSF), the Division of Design, Manufacture, and Industrial Innovation, under Grant No. DMI-9724476, the Combined Research-Curriculum Development (CRCD) Program, under EEC-9527527, and the Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, to UT. This research was also financially made possible by the US Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the High Temperature Materials Laboratory (HTML) User Program under contract DE-AC05-96OR22464, managed by UT-Battelle, LLC.

10:20 AM Break

10:40 AM Invited
Gas-Pressure Forming Behavior and Modeling of Superplastic Al 5083 Alloy: *Chol K. Syn*[1]; Michael J. O'Brien[2]; Donald R. Lesuer[1]; Oleg D. Sherby[3]; [1]Lawrence Livermore National Laboratory, Livermore, CA 94556 USA; [2]Aerospace Corporation, Los Angeles, CA, USA; [3]Stanford University, Dept. of Matls. Sci. & Eng., Stanford, CA 94305 USA

Al 5083 alloy disks were gas pressure formed to hemispheres and cones at constant forming pressures with or without back pressure. The dome heights of the hemispheres and cones were measured, and in some cone samples etched with circle grid, local minor and major strains were measured after forming. The effective (von Mises) flow stresses and effective strain rates developed at the top of the dome during the forming were shown to closely follow the flow stress-strain rate relation obtained from the uniaxial strain-rate change tests performed at the same temperature. The local strain measurements on the circle-gridded samples, the data from the tensile tests performed on-site, and other data from literature sources, were used to construct a partial forming limit diagram for superplastic forming of the alloy. The deformation behavior during the forming experiments was also modeled using the finite element code NIKE2D.

11:10 AM
Improved Contact Algorithm for a Sand Surface Element: *Anthony Chang*[1]; Jonathan Dantzig[1]; [1]University of Illinois at Urbana–Champaign, Dept. of Mechl. & Indl. Eng., 1206 W. Green St., Urbana, IL 61801 USA

An improved modeling method is presented for predicting the residual stresses in foundry castings. In this method, the sand is replaced with an equivalent normal force that represents the force applied to the part by the sand mold. The equivalent normal force takes into account the possible loss of contact with the mold due to thermal shrinkage. A special "soft contact" technique is used which accepts both low stiffness sand materials and relatively high stiffness chill materials. Comparisons between conventional full mold numerical solutions, applied boundary force solutions and experimental results are presented.

11:35 AM
Modeling and Measurement of Quenching Residual Stresses in W319: *Bradley J. Robinson*[1]; Jonathan A. Dantzig[1]; [1]University of Illinois (Urbana–Champaign), Dept. of Mechl. & Indl. Eng., 1206 W. Green St., Urbana, IL 61801 USA

A study of the development of residual stresses in aluminum alloy W319 is presented. Rapid tension tests are performed to determine the mechanical response of W319 at a range of temperatures and strain rates. These results, as well as optimization analysis, are used to develop a constitutive model for W319. The constitutive model is implemented as a user material subroutine in ABAQUS. Beam quenching experiments are performed in order to verify the analytical results. Beams with different cutout geometries are used to obtain experimental results for complex stress states and varying amounts of inelastic deformation. Residual stresses are measured using a groove removal technique upon completion of the quenching process. Results from these experiments compare well to the analytical results derived from the constitutive model.

12:00 PM
Alloy Formation Process in Powder Mixtures under High Speed Plastic Deformation: *Yuriy Podrezov*[1]; [1]Institute of Problems for Materials Science, 3, Krzhizhanovsky, Kyiv, Ukraine

The use of traditional methods of powder metallurgy including sintering and long-term homogenizing does not permit to obtain bars with sufficient plasticity because of the presence of molten fragile phases appearing on the particle boundaries in the process of long-term sintering. The main idea of this work is to get the homogeneous Ni-Mo alloys owing to the effect of superhigh-speed diffusion which appears by the optimum modes of hot deformation of heterocomponent powder mixture. In this case the process of alloy formation runs in a short interval of time due to the high activity of dispersed powder components and mechanical activation of the mass transfer process. The investigations showed that in the Ni-Mo powder system this mechanism can be realized under deformation temperature 1450K. We have obtained almost homogeneous alloys with the solid solution structure from Ni-30 % Mo powder mixture. Previous sintering during which the processes of diffusion homogenization start results in considerable deceleration of homogenization in the course of following deformation.

Powder Materials: Current Research & Industrial Practices: Shock Synthesis, Combustion Synthesis and Densification

Sponsored by: Materials Processing & Manufacturing Division, Powder Materials Committee

Program Organizers: Fernand D.S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgical Engineering, Rapid City, SD 57701-3995 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Wednesday AM Room: 209
November 7, 2001 Location: Indiana Convention Center

Session Chairs: Marc A. Meyers, University of California–San Diego, Dept. of Mechl. & Aeros. Eng., San Diego, CA 92093 USA; Medgar Gelenidze, Institute of Mining Mechanics, Tbilisi, Georgia

8:30 AM Keynote
Shock Synthesis and Densification: *Stepan Batsanov*[1]; Fernand D.S. Marquis[2]; [1]National Institute of Standards, Ctr. for High Dynamic Pressures, Mendeleevo, Moscow Region, Russia; [2]South Dakota School of Mines and Technology, Dept. of Matls. & Metlgcl. Eng., Col. of Matls. Sci. & Eng., Rapid City, SD 57701 USA

Chemical interactions on condensed matter may proceed during shock compression in the high-pressure zone or after the relief on account of the residual heat. In the former case one can obtain nontrivial results, and in the latter case we will have an ordinary synthesis. Thus it is very important to estimate the reaction time at the shock compression of a mixture of solids. For the exothermic reactions the temperature measurements in the minimal time can generate data on the kinetics of the chemical interactions. These experiments were carried out (1969-1987) by the thermocouple method for Sn + S and SnS samples under identical conditions in recovery ampoules. In the reacting mixture the temperature in 10-4 s was higher by ~1000 K relatively to the inert substance. The use of optical pyrometers (1986-1994) permitted to decrease the inertia of measurements to 10-7 s and for Sn + S to obtain the temperatures in excess of ca. 1200 K. Similar experiments were carried out for mixtures consisting of Al + Fe_2O_3, Mg + S, Al + S and Ti + S. Each of them displayed heat evolution in 20-50 ns after an explosion, the transformation rate of 30 • 10% and the same dependence on the grain sizes (the finely dispersed material reacts with a higher heat). The study of the shock compressibility of the Sn + S mixture (1969-1986) showed an expansion of the material at P • 15 GPa, probably caused by a sharp release of heat. A similar increase of volume was found in the Sn + Te system at P • 45 GPa (1991) and the Ti + C mixture

WEDNESDAY AM

at P • 15 GPa (2000). Thus the chemical reactions in the solid mixtures under shock compression proceed during a time of •10 –6 s. The practical results of these experiments are the detonation synthesis of diamond and c-BN and the shock decomposition of organic compounds or NH_3BH_3. It is established that the Mach stem is formed with the high thermodynamic parameters in the axial part of the cylindrical ampoule. It is in these parts of the ampoules that chemical reactions occur initially and then the heat evolution can cause the chemical reaction in the periphery part of a cylinder in the after regime. Particles of substances formed under high dynamic pressures are noticeably smaller than those formed at high temperatures. Thus the investigation of the substructure of recovery materials permits the determination of the distribution of shocked and thermal compounds inside a cylindrical ampoule, and hence the distribution of thermodynamic parameters in this system. It has been shown (1988-2001) that the diameter of the Mach stem in the reacting mixture is larger than in an analogous inert matter. Respectively, the splitting plates at the bottom of cylindrical ampoules, being similar to the Mach stem cross-section, are different for the reacting and inert systems. Knowing the weight of the splitting element and its speed (equal to twice the particle velocity in the matter) one can determine the kinetic energy of this element. The increase of the kinetic energy caused by the heat effect of a reaction can be measured with high accuracy (2001). Such measurements have shown that this additional energy corresponds to the completion of a chemical reaction in the Mach stem. Using the shock-wave technique for a compaction of powders has very long history. New aspects here are preheating of samples and realizing the strong plastic deformations, which can be produced by a sectional (cross-similar) distribution of high explosives around a cylindrical ampoule. In this case, the powerful dynamic loading converts the cylinder into a rather thin plate, where in the material is compressed practically to the 100% density. The maximum effect can be achieved by using an ampoule with residual pressure (DSC-method). Another promising way to improve the densification of powders is to increase the initial density of loading (pre-compressing) a powder into a recovery ampoule, by orienting the particles by a strong electromagnetic field.

9:10 AM

Effect of Shock-Activation on Post-Shock Reaction Synthesis of Ti-Based Ternary Carbides, Ti_3SiC_2 and Ti_2AlN: *Jennifer L. Jordan*[1]; Naresh N. Thadhani[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

The effect of shock-compression of titanium, silicon carbide, and graphite and titanium and aluminum nitride powder mixtures on subsequent reaction synthesis and formation of Ti_3SiC_2 and Ti_2AlN was investigated in this study. Ti_3SiC_2 is a novel ceramic with metal-like properties, namely, high electrical conductivity and plastic-like deformation. The powder precursor mixtures were shock-densified at different pressures using 80-mm diameter gas gun and double implosion cylinder techniques. Characterization of the shock-densified compacts showed an intimately mixed state of powders with little or no reaction. The subsequent reaction behavior of the shock-densified compacts was studied via heat treatments and differential thermal analysis (DTA) at varying heating rates. Activation energies were obtained from these DTA studies and correlated with measurements of fraction reacted as a function of time and temperature to determine the reaction mechanism(s) and degree of activation caused by shock compression. This paper will present the results of the reaction mechanisms and the effects of shock compression on the kinetics of reactions leading to the formation of the ternary carbide in the shock-densified precursor powders.

9:40 AM

Investigation of the Microstructures Observed in Shock Synthesized Mo/Si Powder Mixtures and the Mechanisms of their Formation: *F. D.S. Marquis*[1]; K. S. Vandersall[2]; N. N. Thadhani[1]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metlgcl. Eng., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]Georgia Institute of Technology, Sch. of Matls. Sci. & Eng., Atlanta, GA 30332 USA

Previous work has established that in Molybdenum/Silicon powder mixtures it is possible to observe the occurrence of both “shock-induced” and “shock-assisted” chemical reactions leading to the formation of various silicide microstructures. In shock induced reactions the fundamental mechanisms controlling the synthesis occur during the shock pulse rise time (nanoseconds) and/or during the state of the peak pressure (microseconds). Shock assisted reactions can take place due to bulk shock temperature increases and the controlling processes occur following unloading from the peak pressure stage. In this work an investigation of the microstructures observed in shock synthesized Mo + 2Si powder mixtures has been carried out. The paper presents the effects of the shock parameters on the formation of typical microstructures and discusses the appropriate mechanisms of the shock synthesis obtained.

10:10 AM Break

10:30 AM

Processing of TiC-NiTi Composites by Combustion Synthesis and Quasi-Isostatic Pressing: E. R. Strutt[1]; T. Radetic[2]; E. A. Olevsky[3]; *M. A. Meyers*[1]; [1]University of California–San Diego, Dept. of Mechl. & Aeros. Eng., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA; [2]National Center for Electron Microscopy, Lawrence Berkeley Natl. Lab., Berkeley, CA 94720 USA; [3]San Diego State University, Dept. of Mechl. Eng., 5500 Campanile Dr., San Diego, CA 92182-1323 USA

TiC-NiTi composites were produced by an energy efficient technique combining self-propagating high temperature synthesis (SHS) with densification by quasi-isostatic pressing (QIP). The wide compositional range of titanium carbide creates challenges when forming composites from elemental powders. This talk will focus on the effects of initial reactant chemistry and compact size on the product phase content, microstructure, and matrix transformation temperatures. Composites with varying volume fractions of NiTi have been produced, and the effects of ceramic inclusions on the NiTi martensite variant structure will be discussed. Research supported by the US Army Research Office under contracts DAAH04-95-1-0236 and DAAH04-96-1-0376, and by the Director, Office of Science, Office of Basic Energy Sciences, of the US Department of Energy under Contract No. DE-AC03-76SF00098. The transmission electron microscopy work was performed at the National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, University of California, Berkeley, CA 94720.

11:00 AM

Effect of Shock Loading Parameters on the Substructure and Properties of LiF: *Stepan S. Batsanov*[1]; S. M. Gavrilkin[1]; Fernand D.S. Marquis[2]; [1]National Institute of Standards, Ctr. for High Dynamic Pressures, Mendeleevo, Moscow Region, Russia; [2]South Dakota School of Mines and Technology, Dept. of Matls. & Metlgcl. Eng., Col. of Matls. Sci. & Eng., Rapid City, SD 57701 USA

An investigation of the effect of shock loading parameters on the substructure and properties of the LiF was carried out. It was observed that the LiF powders shocked in a cylindrical ampoules have different properties depending on the method for the application high dynamic shock loading action. In this paper we study the substructure, melting, hardness and transparency of the LiF samples shocked by one-time, double, cross-similar and inner explosion. Typical results of these experiments are the compacted nontransparent material, transparent body and solid foam. In addition the paper discusses the evolution of the microstructure in order to develop an understanding of the processes and mechanism of a formation of these materials.

11:30 AM

Distribution of Polimorphous Modifications and Substructure during Shock Loading of PbO: *Fernand D.S. Marquis*[1]; S. M. Gavrilkin[2]; Stepan S. Batsanov[2]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metlgcl. Eng., Col. of Matls. Sci. & Eng., Rapid City, SD 57701 USA; [2]National Institute of Standards, Ctr. for High Dynamic Pressures, Mendeleevo, Moscow Region, Russia

It has been observed that upon shock loading of powders in cylindrical ampoules, the thermodynamic parameters are distributed in rather complex patterns. These patterns consist generally of maximal values along and to an axis and minimal values near and towards the walls. However the laws governing the change of these parameters are not well known. This paper presents results of an investigation using PbO as the object of the shock loading in cylindrical ampoule. The use of PbO leads to the development of quality gradient pictures. This is so because different modifications of this substance have different colors. In addition the study of the microhardness, melting and substructure the shocked PbO lead to the development of processes for the quantification of the thermodynamic characteristics and peculiarities inside of a cylindrical ampoule under the shock wave loading.

Processing/Property Relationships in Polymer-Matrix Composites and Nanocomposites - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee, SPE Polymer Analysis Division

Program Organizers: Linda Schadler, Rensselaer Polytechnic Institute, Materials Science & Engineering Department, Troy, NY 12180-3590 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; Carl Schultheisz, National Institute of Standards and Technology, Polymers Division, Gaithersburg, MD 20899 USA; Sindee Simon, Texas Tech University, Department of Chemical Engineering, Lubbock, TX 79409 USA

Wednesday AM Room: 201
November 7, 2001 Location: Indiana Convention Center

Session Chairs: Sindee Simon, Texas Tech University, Dept. of Cheml. Eng., Lubbock, TX 79409 USA; Carl Cady, Los Alamos National Laboratory, Struct. Property Relations, Los Alamos, NM 87545 USA

8:30 AM Keynote

Low Cost Pathways to Advanced Structural Composite Materials: *J. L. Kardos*[1]; [1]Washington University, Matls. Rsrch. Lab. & Dept. of Cheml. Eng., St. Louis, MO 63130 USA

US infrastructure, particularly highways, bridges, and other earthquake-prone structures, need help badly and quickly. Carbon fiber composites can uniquely do this job, but the costs of these materials and the processes to manufacture them are still too high to be economically attractive. There are three major thrusts now underway to solve this problem. The first two involve lowering the cost of carbon fiber and developing new low-cost manufacturing processes. The third effort, on which this presentation concentrates, involves developing hybrid systems which utilize carbon fiber composites along with much less expensive materials. We will look at two kinds of hybrids; the first involves combining carbon fibers with other much less expensive reinforcements, such as glass and clays, to achieve property/cost ratios that will enable many new applications in the infrastructure area. The second hybrid approach utilizes a high performance carbon/epoxy composite in conjunction with concrete to produce new bridge column and deck designs capable of competing economically with traditional concrete and steel.

9:00 AM

Bone-Shaped Short Fiber Composites: *Yuntian T. Zhu*[1]; Irene J. Beyerlein[1]; Trevor B. Tippetts[2]; [1]Los Alamos National Lab, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]Massachusetts Institute of Technology, Cambridge, MA 02139 USA

Short-fiber composites usually have low strength and toughness. Decades of world-wide effort on modifying the fiber/matrix interface has not been able to solve this problem. In this work, we have used a novel concept, bone-shaped-short (BSS) fibers, to obtain both high strength and high toughness. In BSS-fiber composites, the load is transferred through the mechanical interlocking at the enlarged fiber ends, making it possible to use a weak interface to increase toughness without sacrificing strength. We have investigated polyethylene fiber reinforced polyester matrix composite and performed both composite mechanical tests and single fiber pullout tests. Our results show that the BSS-fiber composites have remarkably higher strength and toughness than composites reinforced with conventional-straight short (CSS) fibers. These experimental results guided both computational and micromechanical modeling at the fiber and composite length scales. For this composite system, we will present results from finite element analyses to study optimum shape and size with respect to interfacial debond initiation and from a micromechanical BSS fiber pullout model. This talk discusses strength, toughness, fractography of this composite, model development, and issues related to BSS-fiber composite design.

9:20 AM

Interfacial Fracture of Thin Epoxy Films: *Neville R. Moody*[1]; David F. Bahr[2]; Michael S. Kent[3]; John A. Emerson[3]; E. David Reedy[3]; [1]Sandia National Laboratories, Livermore, CA 94550 USA; [2]Washington State University, Pullman, WA 99164 USA; [3]Sandia National Laboratories, Albuquerque, NM 87185 USA

Adhesion is a key factor in controlling the susceptibility to interfacial failure of thin polymer films on metallized substrates. Nevertheless, our understanding of interfacial failure in these systems is limited. We have therefore begun a program to determine the fracture susceptibility of one such system, Epon 828/T403 on aluminized glass, as a function of film thickness. Nanoindentation was combined with deposition of highly stressed overlayers to induce delamination and blister formation in these films. Interfacial fracture energies were then obtained from the delaminations and blisters using mechanics-based models modified for multilayer films. The resulting fracture energies decreased systematically with film thickness and approached a lower limit for the thinnest films. However, this limit is significantly higher than the true work of adhesion for this film system suggesting that inelastic or plastic dissipation processes operate even in the thinnest films we can test. This work supported by USDOE Contract DE-AC04-94AL85000.

9:40 AM

Micromechanisms of Near-Surface Plastic Deformation in Polymer Blends: *David Charles Martin*[1]; Houxiang Tang[1]; [1]The University of Michigan, Matls. Sci. & Eng., 2022 H.H. Dow Bldg., Ann Arbor, MI 48109-2136 USA

We have been examining the near surface deformation of polymer blends in order to better elucidate how painted coatings delaminate and surface scratches cause optical whitening. The plastic deformation under surface scratches in automotive polypropylene blends (so called themoplastic polyolefins or TPOs) was studied by optical microscopy, scanning electron microscopy (SEM), and transmission electron microscopy (TEM). It was found that the material plastically deformed by forming shear bands under the scratches. The shear bands initiate from the front of the wavy deformation ridge near the surface and propagate into the bulk of the material with decreasing width and at characteristic tilting angles with respect to the surface. It was observed that the material inside the shear band dilates and that the dilation decreases with the distance from the surface. The morphological features of the shear bands were correlated with the stress field under the scratch. Different factors influencing the modes of deformation were also studied, including the size and shape of the rubbery phase, structural anisotropy, mineral additives, the crystalline structure of the PP matrix and the normal scratching load. The results from this research help to explain the visibility of the surface scratches and have broad implications for designing PP blends with improved surface damage resistance.

10:00 AM Break

10:20 AM

Direct Formation of Silane Coupling Agents on Glass Surfaces: *Jon J. Kellar*[1]; William M. Cross[1]; David A. Boyles[2]; [1]South Dakota School of Mines and Technology, Matls. & Metlgcl. Eng., 501 E. St. Joseph St., Rapid City, SD 57701-3995 USA; [2]South Dakota School of Mines and Technology, Chem. & Cheml. Eng., 501 E. St. Joseph St., Rapid City, SD 57701 USA

Often the Achilles' Heel of polymer matrix composites (PMCs) is the ingress of water to the reinforcement matrix interface. Traditionally, silane coupling agents are adsorbed onto the reinforcement to help prevent debonding. The focus of this research is a significant change in methodology which moves away from traditional coupling agents, bound as they are by a hydrolytically-labile silicon-oxygen linkage to the glass fiber. The first task in the research involved formation of the hydrolytically stable, grafted silane layers. This was accomplished by: 1) hydrogenation of glass substrates: 2) synthesis of aminoalkenes; and 3) reaction of aminoalkenes with the reduced surface via a hydrosilylation reaction which forms the coupling agent directly on the surface of the glass fiber. Next, the grafted silane monolayer was evaluated for hydrolytic stability and compared to traditional silane surface treatments.

10:40 AM

Effects of Thermal Shock on Mechanical Behaviour of Kevlar/Epoxy and Kevlar/Polyester Composites: *Syed T. Hasan*[1]; Bankim C. Ray[2]; [1]Sheffield Hallam University, Sch. of Eng., City Campus, Howard St., Sheffield, S. Yorkshire S1 1WB UK; [2]Regional Engineering College, Dept. of Metall. Eng., Rourkela, India

The aramid kevlar family of fibres has been well accepted as reinforcement for composites and finds wider and newer application in various industrial and aerospace applications. The interfacial bond strength between aramid fibres and epoxy resins is normally lower than what is experienced with carbon fibre composites. This weakness with kevlar necessitates investigation and evaluation of interlaminar shear strength (ILSS) and modulus under some realistic environmental conditions. This work investigates the variation of ILSS and modulus values after being subjected to extreme thermal shock of 160C. Results showed some interesting trends in ILSS variations and modulus values of composites system which helps to understand the damage mechanisms in composite materials.

11:00 AM

The Effect of Voids on the Elastic Properties of Fiber-Tows Reinforced Polymer Composites: *Li Lu*[1]; Rajiv Shivpuri[2]; [1]Visteon Corporation, Interior Sys. CAE, 7900 N. Fountain Park, Apt. 125, Westland, MI

48185 USA; [2]The Ohio State University, Indl. Welding & Sys. Eng., 209 Baker System, 1971 Neil Ave., Columbus, OH 43210 USA

The effect of processing induced voids on the elastic properties of the fiber tow reinforced polymer composites has been investigated using the developed analytical model. The model is based on a multi-level homogenization scheme incorporated with base two-phase micro-mechanics models. Different shapes, sizes and locations of voids are considered. Comparison between theoretical prediction and numerical simulation results shows good consistency between model and simulation in evaluating the effect of voids on elastic properties of composites. The application of the model to predict the effective elastic moduli shows that the stiffness of the composites are much adversely influenced by the presence of manufacturing induced voids.

11:20 AM

Residual Stress Development during Thermoset Cure: Effects of Cure History and Resin Properties: Mataz Alcoutlabi[1]; Gregory B. McKenna[1]; *Sindee L. Simon*[1]; [1]Texas Tech University, Cheml. Eng., MS 4121, Lubbock, TX 79409 USA

A thermo-viscoelastic model that quantitatively describes the evolution of the viscoelastic properties during cure of thermoset materials has been developed and extended to predict residual stresses in a curing resin. We find that, in contrast to the uniaxial case, the stress-free temperature for the triaxially constrained material is not the cure temperature. Furthermore, isotropic stresses that develop during cure can be of the same magnitude as the thermal stresses. An important parameter for predicting and minimizing isotropic residual stresses is found to be the temperature at which the thermoset resin gels during cure as long as vitrification does not occur during cure. In addition to the effects of cure history, we are also evaluating the effects of resin parameters on isotropic residual stresses for systems in which ring-opening reactions are used to reduce shrinkage and residual stresses.

11:40 AM

Fiber-Optic Sensor System for Total Lifetime Monitoring of Polymer Matrix Composites: *Jon J. Kellar*[1]; William M. Cross[1]; Lidvin Kjerengtroen[2]; [1]South Dakota School of Mines and Technology, Matls. & Metlgcl. Eng., 501 E. St. Joseph St., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Mechl. Eng., 501 E. St. Joseph St., Rapid City, SD 57701 USA

The overall objective of this work is the development of a total lifetime, fiber-optic sensor system for polymer matrix composite materials. The specific objectives of the research were to develop and calibrate both the chemical and mechanical sensing behavior of the fiber-optic sensor system in a model, unidirectional, single-ply laminate. Evanescent wave chemical sensing was used to determine the cure extent of the matrix adjacent the optical fibers. Next, the cured model unidirectional, single-ply laminate from each test were used for evaluating the strain response of the system.

Radiation and Thermal Degradation of Polymer Materials

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Nuclear Materials Committee, SPE Polymer Analysis Division

Program Organizers: Robert Sindelar, Westinghouse Savannah River Company, Aiken, SC 29808 USA; Vassilios Galiatsatos, Abbott Laboratories, Ashland, OH 44805 USA; Linda Schadler, RPI, Materials Science & Engineering Department, Troy, NY 12180-3590 USA; Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Wednesday AM Room: 111
November 7, 2001 Location: Indiana Convention Center

Session Chairs: Vassilios Galiatsatos, Abbott Laboratories, Ashland, OH 44805 USA; Robert Sindelar, Westinghouse Savannah River Company, Aiken, SC 29808 USA; Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

8:30 AM

The High Strain-Rate Tensile Response of Pristine and Hydrolytically Aged Estane Elastomer to Plate Impact: *Jerry J. Dick*[1]; James N. Johnson[1]; A. Richard Martinez[1]; [1]Los Alamos National Laboratory, DX-1, MS P952, Los Alamos, NM 87545 USA

Estane discs were made by molten injection followed by hot pressing. They were subjected to plate impact using a light-gas gun. Transmitted wave profiles were measured. The profiles show the response of the material to compression followed by tension. The failure in tension is partial and depends on the amount of aging. The spall strength depends on these factors also. The behavior has been modeled (Ref. 1, 2). 1. J. N. Johnson, J. J. Dick, and R. S. Hixson, "Transient response of three polymers," J. Appl. Phys. 84, 2520 (1998). 2. J. N. Johnson and J. J. Dick, in Shock Compression of Condensed Matter-1999, edited by M. D. Furnish, L. C. Chhabildas, and R. S. Hixson, (American Institute of Physics, Melville, NY, 2000) p. 543.

8:50 AM

Evolution of Hardness in Irradiated Poly(Metyl Methacrylate) at Elevated Temperatures: *Sanboh Lee*[1]; K.-P. Lu[1]; [1]National Tsing Hua University, Dept. of Matls. Sci., 101 Kunag Fu Rd., Sec. 2, Hsinchu, Taiwan 30043 China

Evolution of hardness of gamma-ray irradiated poly(methyl methacrylate)(PMMA) at elevated temperatures has been investigated. The hardness decreases linearly with the concentration of defects for hardness. The change of concentration of defects for hardness follows a first-order annihilation process during isothermal annealing. The reciprocal of time constant satisfies the Arrhenius equation and the corresponding activation energy of the kinetic process dedreases with increasing dose. The hardness of post-annealed PMMA decreases linearly with increasing dose. The results are also compared with the literature.

9:10 AM

Effects of Neutron Exposure on Structural Properties of Regolith Composites: Jerrel Moore[1]; *Jianren Zhou*[1]; Richard Wilkins[1]; Thomas Fogarty[1]; [1]Prairie View A&M University, Ctr. for Appl. Radiation Rsrch., Prairie View, TX 77446 USA

The development of Mars missions for human activity will increasingly require effective utilization of Martian materials in building habitats and working structures, since the Martian atmosphere provides only modest protection from space radiation. One potential approach is to use polymer binders with Martian regolith to form structural elements. Not only can useful composite materials be thus produced to meet structural requirements but also the radiation protection properties are improved. In present work, two composites fabricated using polymers and simulated Martian regolith, that could be synthesized from local Martian materials someday for manned Mars missions, were studied. To validate shielding effectiveness, composites were irradiated with neutron beams up to 800 MeV. Effects of neutron exposures on mechanical and thermal properties of the composites were characterized and studied. Shielding effectiveness of the composites on microelectronic devices was also investigated.

9:30 AM

Mechanistic Insight into the Origin of Phase Separation of Thermally Degraded Segmented Polyester Urethanes via Multinuclear Solid State NMR: *James H. Small*[1]; E. Bruce Orler[1]; Debra A. Wrobleski[1]; [1]Los Alamos National Laboratory, Polymers & Coatings Grp., Matl. Sci. & Tech. Div., MST-7, MS E-549, Los Alamos, NM 87545 USA

In the past ten years, the technical oversight of the Stockpile Stewardship Management Program has been the paramount role of the National Laboratories under the auspices of the Department of Energy (DOE). To accomplish this daunting task, the research direction has focused on aging and degradation in the context of lifetime extension. To conduct a full and accurate evaluation of the aging and degradation pathways of materials commonly found in the stockpile (including mechanistic, kinetic, and thermodynamic studies), a great variety of analytical techniques have been utilized. One powerful technique currently brought to bear to answer many of these questions is nuclear magnetic resonance (NMR). At Los Alamos National Laboratory (LANL), both solution and solid state multinuclear, multidimensional NMR spectroscopy is being used to investigate materials found in the enduring origins of phase separation as a function of thermal degradation. In particular, multinuclear ^{1}H, ^{2}H, ^{13}C, and ^{15}N solid state NMR relaxation and two-dimensional experiments have afforded keen insight into the fundamental origins of phase separation of a commercially available segmented polyester urethane. Solid state NMR has proven to be a valuable tool for molecular-level investigation of structure-property relationships in these multi-component polymeric systems.

9:50 AM

Mechanical Properties of Irradiated Epoxy-Based Protective Coatings: *P. E. Zapp*[1]; P. S. Lam[1]; M. E. Dupont[1]; R. L. Sindelar[1]; [1]Savannah River Technology Center, Aiken, SC 29808 USA

The mechanical properties of epoxy-based coatings for nuclear power plant containment structures were measured and analyzed to predict the response of coatings to a loss-of-coolant accident (LOCA). Adhesive strength, free-film tensile strength, and the resistance to delamination initiated at an intentional non-bond defect were measured under temperature, wetness, and radiation dose conditions (109 rads) representative of a LOCA in a pressurized water reactor. The resistance to delamination of a coating from its substrate is defined here as the material G-value, and represents a novel analysis of resistance to coating failure. The material G-value is compared to an applied G-value, which represents the applied load at a non-bond defect in the coating developed under the thermal transients of the LOCA, to express a criterion for coating failure.

10:10 AM
Effects of Gamma Irradiation on the Performance of Epoxy-Based Protective Coatings in High-Temperature Steam and Water Environments: *M. E. Dupont*[1]; R. L. Sindelar[1]; P. E. Zapp[1]; [1]Savannah River Technology Center, Aiken, SC 29808 USA

Debris formation from the polymeric coatings on the containment of nuclear power plants in the aftermath of a design-basis loss-of-coolant-accident could have a negative effect on the performance of safety systems. Epoxy-based coating systems were irradiation treated to 109 rads at 50°C and exposed to steam and water immersion cycles simulating accident conditions. Coating degradation and debris formation were observed in the irradiated specimens, but not in the non-irradiated companion specimens. The affected region of the irradiated coatings was confined to a near-surface layer, the thickness of which was dependent upon the radiation and dose-rate. These changes appeared to be the result of a synergistic effect of radiation and oxygen permeation during irradiation.

Advocacy for Materials Science and Engineering–*the Washington " scene"*

A Special session sponsored by the Public and Government Affairs Committee of TMS and the Federal Affairs Committee of ASM International

Wednesday PM — Wabash Ballroom I
November 7, 2001 — Location: Indiana Convention Center

Session Chairs: Diran Apelian, Metal Processing Institute, WPI. Worcester, MA; Bruce Boardman, Deere and Company, Moline, IL

Keynote Speakers:

Public Understanding of Engineering: Affecting Change through Strategic Cooperation: Randy Atkins[1]; [1]National Academy of Engineering

As we enter the 21st Century, technology is the driving factor to the economy, health, and quality of life. Yet, society does not understand how technology is developed or applied, much less appreciate its own responsibility in determining technological progress. In a world increasingly dependent upon technology, society is not equipped to make decisions about its own welfare.The engineering community is well aware of the public's lack of understanding and has, in response, devoted a tremendous number of resources in the form of public relations, education, and public affairs efforts. However, with the exception of a few projects, the engineering community tends to splinter by discipline; messages are narrow and discipline-specific; and activities are often redundant and sometimes serve only small communities. It's time for the engineering community to develop an agenda for change. Atkins will speak about successful cooperative and collaborative efforts to increase the public's understanding of engineering.

In the Interest of Science: Getting the Funding Message Through to Washington: Merrilea J. Mayo[1]; [1]The Pennsylvania State University

In the arena of research funding, scientists and engineers have had a long and illustrious history of focusing on the trees and losing sight of the forest. Researchers work long hours to pull together proposals for individual projects, oblivious to the fact that the overall budget for a given agency may be so low as to preclude continued funding for any project, much less theirs. The last decade of declining students and dollars (as a % of GDP) in virtually all physical science disciplines bear testament to this problem. This presentation focuses on the current lobbying landscape for the physical sciences in Washington, and how its structural deficiencies have led to the well-known dichotomy between excellent Congressional support for the life sciences and mediocre support for the physical sciences. ASTRA, a new advocacy organization for the physical sciences, is introduced and described in terms of how it may fill portions of this gap. Additionally, strategies are presented to allow organizations and individuals to successfully lobby Congress on behalf of the physical science and engineering disciplines.

Affordable Metal-Matrix Composites for High Performance Applications - V: Strengthening Mechanisms

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: Awadh B. Pandey, Pratt & Whitney, Liquid Space Propulsion, West Palm Beach, FL 33410-9600 USA; Kevin L. Kendig, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Watson, Pratt & Whitney, East Hartford, CT 06108 USA

Wednesday PM — Room: 139
November 7, 2001 — Location: Indiana Convention Center

Session Chair: John J. Lewandowski, Case Western Reserve University, Dept. of Matls. Sci. & Eng., Cleveland, OH 44106 USA

2:00 PM Invited
Finite Element Modeling of Discontinuous Reinforced Aluminum in 2D and 3D: *Cliff J. Lissenden*[1]; Hui Shen[1]; [1]Pennsylvania State University, Eng. Sci. & Mech., 212 Earth-Engineering Sciences Bldg., University Park, PA 16802 USA

Deformation in discontinuous reinforced aluminum has been simulated using three distinct types of finite element model: a 3D repeating unit cell, a 3D multi-particle model, and 2D multi-particle models. The repeating unit cell model represents a fictitious doubly periodic cubic array of particles. The 3D multi-particle model represents randomly placed and oriented particles. The 2D multi-particle models were obtained from planar sections through the 3D multi-particle model. These models were used to study the tensile stress-strain response in an elastoplastic matrix. The results indicate that the unit cell model predicts less macroscopic strain hardening than the multi-particle models. In addition, the plastic strain distributions predicted by the 2D and 3D multi-particle models are different, with the plastic strain concentrations being more intense in the 2D model than in the 3D model. Thus, it appears necessary to use a multi-particle 3D model to accurately predict material responses that depend on local effects, such as strain-to-failure, fracture toughness, and fatigue life.

2:30 PM
Thermal and Mechanical Properties of Metal Matrix Composites using Microstructure-Based Finite Element Techniques: Rajarshi Saha[1]; Jason J. Williams[1]; *Nikhilesh Chawla*[1]; [1]Arizona State University, Dept. of Chem. & Matls. Eng., PO Box 876006, Tempe, AZ 85287-6006 USA

Conventional Finite Element Methods (FEM) do not account for the complex nature of the microstructure in MMCs (e.g., particle clustering and angularity of the particles) when modeling physical properties. We have employed a two-dimensional object oriented finite element analysis technique (OOF) to obtain a true microstructure-based model for these materials. The effect of reinforcement volume fraction and particle size on thermal and mechanical properties in an Al/SiCp MMC was studied. The elastic modulus and coefficient of thermal expansion of the composites were calculated and correlated quite well with experimentally determined values. The salient features of the microstructures and their effect on stress distributions and properties of the composites will be discussed. Research sponsored by the United States Automotive Materials Partnership (USAMP) and the Office of Naval Research (ONR).

2:50 PM
Discontinuously-Reinforced Titanium Matix Composites by the In-Situ Reaction Synthesis Process: *Radhakrishna B.V. Bhat*[1]; Daniel B. Miracle[2]; [1]Systran Federal Corporation, Dayton, OH 45431-1672 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., Wright-Patterson Air Force Base, OH 45433 USA

The available literature suggests that a combination of Boron & Carbon additions lead to superior properties in the discontinuously reinforced Titanium matrix (DRTi) composites. This paper describes a study carried out to produce DRTi composites having both Boron and Carbon additions and the processing-microstructure-property relationships. The in-situ reaction synthesis of Ti and Ti-6Al-4V alloy matrix composites by the powder metallurgy process involving attrition milling, wet blending and vacuum hot pressing has been adopted. Both pre-alloyed powder produced by gas atomization after addition of Boron and Carbon to the melt, as well as, a mixture of matrix powder, TiB_2 and B_4C powders, have been used as the raw materials. The composites have been produced with three different volume fractions of reinforcement viz. 10, 20 and 40%. The characterization involving both x-ray diffraction and micro-structural evaluation to

WEDNESDAY PM

establish the volume fraction, morphology and composition of the phases and mechanical property evaluation to measure the tensile strength and ductility at room and elevated temperature, the fracture toughness using the notched bend specimens and the conclusions based on these are discussed.

3:10 PM Invited

Ductile Fracture in Particle Reinforced Metallic Composites: *S. Ghosh*[1]; S. Moorthy[1]; [1]The Ohio State University, Dept. of Mechl. Eng., Columbus, OH 43210 USA

Damage initiation and progress to cause ductile failure in particle reinforced composite microstructures with varying distributions, is modeled in this paper. The damage mechanisms include particle and matrix cracking, which are modeled in a Voronoi Cell finite element model (VCFEM) framework. Particles are assumed to be brittle, while the matrix is modeled as ductile with evolving porosity. Particle cracking is modeled by the Mohr's theory of maximum principal stress or the Rankine criterion. Progressive damage by ductile fracture of the metallic matrix material is represented by a pressure dependent elastic-plastic constitutive relation for porous materials. In particular a non-local form of the Tvergaard-Gurson model with void nucleation, growth and coalescence is used for modeling the matrix response and damage. Evolution of damage is monitored through growth of porosity in the microstructure. Regions of critical porosity levels are adaptively enriched to realize the localized strain fields in the evolving matrix crack. Metal matrix composites with different particle volume fractions and particle sizes subjected to different strain levels by uni-axial tension, are studied. All geometric information of each microstructural section are then recorded by an image analysis system. These sections are modeled with VCFEM for damage evolution and compared with the experimental observations. In particular, damage in the microstructure near a structural flaw is compared with experimental observations by modeling a distribution of particles ahead of the flaw in a compact tension test.

3:40 PM Break

3:55 PM Invited

Microstructure, Stability and Mechanical Behavior of Mechanically Alloyed Al-4Mg Alloy Produced on a Production Scale: *Young-Won Kim*[1]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

The microstructures and properties of a mechanically alloyed Al-4wt%Mg alloy, containing fine carbides and oxides, were investigated in successively processed conditions. The alloy was produced through mechanical alloying of elemental powders to a batch amount of 200lbs. The alloy powders were then cold isostatically pressed to cylindrical billets weighing 50lbs. One billet was machined into several smaller billets which were canned and hot-pressed (HP) against a blind die in an extrusion press. These fully dense HP billets were extruded with an extrusion ratio of 9:1. Some of the extruded material was cold rolled to various reductions. All materials were given isochronal, as well as isothermal, annealing treatments, and microstructural variations and stability were recorded using light microscopy and TEM. Tensile properties were evaluated over wide ranges of temperatures (RT to 500°C) and strain rates (0.1 to 10-5 /s). The fatigue crack growth at RT was measured as a function of grain size. This paper focuses on discussing the strengthening mechanism of the mechanically alloyed material, the abnormal grain growth following certain thermomechanical treatments leading to drastic strength loss, and the influence of grain size on both yield strength and crack propagation at RT.

4:25 PM

Mechanical Properties of High Strength 7034 Alloy and MMC: *Anand V. Samant*[1]; Robert Zawierucha[1]; [1]Praxair, Inc., Proc. & Sys. R&D, Matls. Eng. Lab., 175 E. Park Dr., Tonawanda, NY 14150 USA

In the Chemical Process Industry (CPI), the use of materials with high specific strengths can lead to significant cost savings through increased efficiencies in the operation of equipment such as turbomachinery. Aluminum MMCs hold promise in such applications due to their high specific strength and specific stiffness. Currently, investigation is underway on a new high strength Al-Zn alloy, which has been recently given the Aluminum Association designation 7034, and a MMC of the 7034 alloy reinforced with SiC particulate. These materials were chosen because they hold the potential of higher strength and stiffness without significantly compromising ductility and fracture toughness. Mechanical properties of the unreinforced material, such as room and cryogenic temperature strength, fracture toughness, fatigue, and Stress Corrosion Cracking (SCC) behavior, will be presented. Preliminary results for the MMC will also be discussed. Results will be compared to those for other 7000 series alloys reported in the literature.

4:45 PM

Application of the Cruciform Specimen Geometry to Obtain Transverse Interface Property Data in a High Fiber-Volume-Fraction SiC/Titanium Alloy Composite: *C. J. Boehlert*[1]; B. S. Majumdar[2]; D. B. Miracle[3]; [1]Los Alamos National Laboratory, Los Alamos, NM 87544 USA; [2]New Mexico Institute of Mining and Technology, Dept. of Matls. Sci. & Metall., Socorro, NM 87801 USA; [3]Air Force Research Laboratory, Matls. & Mfg. Direct., Wright-Patterson AFB, OH 45433-7817 USA

A combined experimental and computational methodology was used to determine the relevant strength and residual stress parameters in a manufactured, high fiber-volume-fraction multi-ply metal matrix composite (MMC). The method was similar to that previously demonstrated on single-fiber composites, which had extremely low fiber-volume-fraction. Variabilities in residual stresses and debond strengths in high fiber-volume-fraction multi-ply composites, as well as current demands on micro-mechanics based computational prediction and validation of complex composite systems, necessitated the establishment of the test methodology described here. The model material chosen for this investigation was a plasma-processed six-ply, unidirectional Sigma-1240/Ti-6Al-2Sn-4Zr-2Mo(wt.%) MMC containing 32 volume percent continuous fibers. Room-temperature transverse tensile experiments were conducted on cruciform specimens. In addition, rectangular specimens were also evaluated in order to verify their applicability in obtaining valid interfacial property data. Debonding events, evaluated at different positions within a given specimen geometry, were captured by stress-strain curves and metallographic examination. Analytical and finite element stress analysis were conducted to estimate the geometrical stress concentration factors associated with the cruciform geometry. Residual stresses were estimated using etching and computational procedures. For the cruciform specimens, the experimental fiber-matrix debond strength was determined to be 22MPa. Separation occurred within the carbon-rich interfacial layer, consistent with some previous observations on similar systems. Thus the cruciform test methodology described here can be successfully used for transverse interfacial property evaluation of high fiber-volume-fraction composites. For the rectangular specimens, the strain gages at different positions along the specimen width confirmed that the interface crack had initiated from the free edge and propagated inwards. Hence rectangular specimens cannot be used for valid interface strength measurement in multi-ply composites.

5:05 PM

Morphology of Ti-Si Eutectuc Grains: *V. Mazur*[1]; S. Firstov[1]; S. Kapustnikova[1]; [1]Institute for Problems of Materials Science–Kyiv, 3 Krzhizhanovskogo Str., Kyiv 03142 Ukraine

The properties of eutectic alloys are defined by properties of eutectic phases and morphology of eutectic grains. Therefore investigation of morphology of eutectic grain has great significance when making the new eutectic Ti-Si alloys. Usually to give an idea of eutectic grain morphology the plane sections pictures are used. However in this case it is possible to make a false conclusion because plane sections give no conception on spatial structure of crystal. For example it is difficult to say what kind of crystals we observe (round rods or ellipsoid particles) if we have ellipse-like cross-sections of second phase. To obtain a conception on spatial structure of Ti+Ti5Si3 eutectic the methods stereo-morphology are used. They included scanning electron microscopy of skeleton dendrite branches of Ti5Si3 phase of specimen with removed metallic phase, consecutive re-polishing and micro-photographing of the same eutectic grain of given specimen, plane of section was oriented of the main crystallography directions of primary silicide crystal. The eutectic Ti+Ti5Si3 constitutes the product of cooperative growth both of two dendrites: polyhedron and roundish ones. Titanium silicide is nascent and leading phase of eutectic. The eutectic grain inherits the form and facets of primary crystal. Silicide crystal is genetic center of eutectic grain. The formation of eutectic grain includes some stages. At first nucleus of second phase (beta-titanium) arises and grows as planar dendrite at one of silicide crystal facets. Properly eutectic crystallization is realizing as couple, cooperative growth of silicide and metallic phases. It is completing by forming plate-like or rod-like morphology of eutectic. Concrete temperature and concentration arrangement at the crystallization front determines what of two morphologies will be realize in given region. Plate-like eutectic forms when two-phase lamination grows in directions <1000> of Ti5Si3. During further growth in this directions the plates of eutectic phases transform into cellular, ledeburite-like structure where role of matrix plays silicide. During growth in direction <0001> of silicide crystal rod-like morphology realizes. In this type of eutectic structure beta-titanium solid solution serves as matrix phase of eutectic. Of course the mechanical properties of those two types of eutectic will be quite different. Indeed, if a crack was born in same point of eutectic grain it will be braked in metallic matrix more effectively than in silicide one because plasticity of metallic phase is more than silicide. The stereo-morphology model of eutectic grain is proposed. The picture of this model is cited.

5:25 PM

Aging of 7075 Aluminum Alloy Composite Reinforced with Alumina Particles: *Erika Esquivel*[1]; S. K. Varma[1]; [1]The University of Texas at El Paso, Dept. of Metall. & Matls. Eng., El Paso, TX 79968-0520 USA

Metal matrix composites can be attractive materials due to their high strengths and wear resistance. Effect of solutionizing on the aging response of 7075 aluminum alloy containing 0.1 volume fraction of alumina particles (VFAP) has been determined. The aging response of composites has been compared with those with monolith for different solutionizing times. The values of time required to achieve peak hardness at aging temperatures of 180 and 200°C have been qualitatively related to the solutionizing time at a given temperature. Aging curves have been analyzed and compared with results already published for composites and monoliths of 6061 and 2014 aluminum alloys reinforced with similar VFAP. Microstructures evolved during aging and solutionizing will be discussed.

Full Density Powder Processing

Sponsored by: Materials Processing & Manufacturing Division, Powder Materials Committee

Program Organizers: Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Stephen J. Mashl, Bodycote IMT, Inc., Andover, MA 01810 USA

Wednesday PM   Room: 206
November 7, 2001   Location: Indiana Convention Center

Session Chair: TBA

2:00 PM

Microstructure Control of P/M Duplex Stainless Steels through Rapid Cooling within the HIP Vessel: *S. J. Mashl*[1]; J. D. Hall[1]; [1]Bodycote IMT, Inc., 155 River St., Andover, MA 01810 USA

Fully dense powder metallurgy duplex stainless steels are a modern class of alloys offering excellent corrosion resistance and mechanical properties superior to wrought, at a comparatively moderate cost. During the hot isostatic pressing (HIP) of these alloys, the slow cooling rates typical of older vessels promotes the formation of a brittle sigma phase. Recent advances in the development of rapid cool HIP units now make it possible to control phase transformations in some alloy systems. This paper examines the formation of sigma phase as a function of HIP cooling rate and component cross section. The fractions of brittle phase are measured and the mechanical properties of these materials are reported and compared to the properties of a conventional HIPed and heat treated alloy.

2:30 PM

Consolidation of Zr-Based Amorphous Metal Powders with Equal Channel Angular Extrusion: *I. Karaman*[1]; K. T. Hartwig[1]; S. Mathaudhu[1]; I. Anderson[2]; [1]Texas A&M University, Dept. of Mechl. Eng., MS 3123, College Station, TX 77843 USA; [2]Iowa State University, Dept. of Matls. Sci. & Eng., Ames, IA 50011 USA

A severe deformation processing method called equal channel angular extrusion (ECAE) will be presented as a unique powder consolidation technique. Two potential benefits of ECAE over conventional extrusion and other powder consolidation techniques are the uniformity of strain and thus microstructure throughout the cross section of processed material and the conservation of work-piece cross-section between successive extrusions. The present work will demonstrate a successful consolidation of Zr-based amorphous metal powders with ECAE. The amorphous powders of the Zr55Al10Cu30Ni5 alloy are fabricated by high pressure gas atomization (HPGA) in an inert atmosphere. The present alloy is chosen because of its easy glass forming ability with a large supercooled liquid region. The physical properties of as-atomized amorphous powders such as particle size, and critical transformation temperatures will be presented as well as their microstructure. Vacuum encapsulated powders are ECAE processed in the supercooled liquid region. Variations of the extrusion parameters were studied including temperature, hydrostatic pressure, strain level, and strain rate. The mechanical and thermal properties of the resulting amorphous Zr-based alloy billets will be compared with those of the initial powders and those of cast bulk material to verify the effectiveness of consolidation. This work is supported by the DARPA-SAM.

3:00 PM

Direct Laser Deposition of Alloys from Elemental Powder Blends: *Rajarshi Banerjee*[1]; Peter C. Collins[1]; Craig Brice[2]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Lockheed Martin, Dallas, TX, USA

The complexity in design of components used in advanced aerospace and automotive applications is continuously increasing. This has led to the development of near-net shape manufacturing techniques such as laser engineered net-shaping (LENS) which falls in the class of direct laser deposition processes from powder feedstock. In addition to near-net shaping, the LENS technology also allows for the deposition of fully dense alloys. Despite considerable advances in process optimization, there is a rather limited understanding of the role of metallurgical factors in laser deposition of alloys. One such factor is the thermodynamic enthalpy of mixing which plays a significant role in the deposition of alloys from elemental powder blends using LENS. This factor influences the homogeneity as well as the rate of solidification of the alloy and consequently the microstructure and properties of the deposit. The enthalpy of mixing could also serve as a very useful guideline in the design of novel alloys. Examples of Ti-Cr, Ti-Nb, and Ni-Mo alloys deposited from a blend of elemental powders using the LENS process will be discussed in this paper.

3:30 PM

Metallurgy and Laser Processing of Refractory Metal Deposits: *C. Mukira*[1]; M. Jackson[1]; C. Hardwicke[1]; [1]GE Corporate Research & Development, PO Box 8, Schenectady, NY 12301 USA

A systematic study was carried out to establish metallurgical interactions between refractory metal powders (Mo and Cr) and some Ni alloy substrates during the laser welding process. The design-of-experiment (DOE) approach was applied in order to quantify effects of laser welding conditions (e.g., beam power, travel speed and deposition rate) on certain weld features, including–extent of chemical dilution, weld microstructures, and weld defects, that were of interest in the study. Statistical tools, Design for Six Sigma tools (DFSS), were applied to generate transfer functions, correlating the laser conditions and the weld features. The results showed the depth of weld penetration varied from less than 100 µm to greater than 1 mm, depending on laser power density and type of powder. Generally, Mo deposits exhibited greater amounts of weld defects (porosity and cracks) than did the Cr deposits. The weld microstructures grew into large columnar grains during the laser process.

Fundamentals of Solidification - IV

Sponsored by: Materials Processing & Manufacturing Division, Solidification Committee

Program Organizers: John H. Perepezko, University of Wisconsin–Madison, Department of Materials Science & Engineering, Madison, WI 55706 USA; Christoph Beckermann, University of Iowa, Department of Mechanical Engineering, Iowa City, IA 52242-1527 USA; William J. Boettinger, National Institute of Standards & Technology, Gaithersburg, MD 20879 USA

Wednesday PM   Room: White River Ballroom
November 7, 2001   Location: Indiana Convention Center

Session Chair: William J. Boettinger, NIST, Gaithersburg, MD 20879 USA

2:00 PM

Experimental Study and Modelling of the Cell-Dendrite Transition: *R. Anthony Chilton*[1]; John D. Hunt[1]; [1]Oxford University, Dept. of Matls., Parks Rd., Oxford, Oxfordshire OX1 3PY UK

In this study, a succinonitrile alloy was directionally solidified on a temperature gradient stage using a variety of growth velocity profiles. The experimental results show a transition between cellular and dendritic growth morphologies accompanied by a discontinuity in the range of stable array spacings. The two structures are distinguished by their tip shapes rather than the existence/absence of side-arms. A new numerical model for the transition is proposed which gives good agreement with the observed spacing ranges. The numerical results also indicate that the transition between growth morphologies could be precipitated by the break down of the mechanisms limiting the stable range of spacings on the cell/dendrite array. This allows the conditions for the cell-dendrite transition to be predicted numerically.

2:20 PM

The Solid-Liquid Interface for Real: High-Speed, High-Magnification Videos of a GTA Weld in Stainless Steel: *Aaron C. Hall*[1]; [1]Sandia National Laboratories, 1833, PO Box 5800, Albuquerque, NM 81785 USA

Viewing the solid-liquid interface in an engineering metal under real-world solidification conditions is one of the "holy grails" of experimental solidification. This has been accomplished by using a high quality zoom lens and a high-speed CCD camera to video a GTA weld in stainless steel. Both the trailing (solidifying) edge and the leading (melting) edge of the weld pool have been videoed. These videos will be presented and discussed. Secondary dendrite arms can be clearly seen at the trailing edge of the weld pool. Dendrite arm advancement can also be seen and will be discussed. In addition, weld ripple formation has been captured. A sequence of events that consistently accompanies ripple formation has been identified and will be used to present a description of weld ripple formation. Grain boundary melting can be clearly seen at the leading edge of the weld pool. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

2:40 PM
In-Situ Observation of Dendritic Solidification of Sn-13%Bi Alloy by Microradiography: *Bin Li*[1]; Harold D. Brody[1]; [1]University of Connecticut, Inst. of Matls. Sci., Unit 3136, Storrs, CT 06269-3136 USA

A microfocus X-ray source is being used to observe the dendritic solidification in Sn-13%Bi alloy in real time. Dendrite morphology and its evolution can be clearly resolved. Solute distribution around the dendrite tip is being estimated by measuring the contrast difference in the digitized images and the scale of the boundary layer can be resolved. The growth kinetics and the temperature at the solid-liquid interface are also measured during solidification; and the effect of cooling rate on the solidification undercooling is being analyzed.

3:00 PM
Transient Growth of an Equiaxed Dendrite into a Supercooled Melt: *I. Steinbach*[1]; N. Warnken[1]; C. Beckermann[2]; [1]ACCESS e.V., Intzestr. 5, 52072, Aachen, Germany; [2]University of Iowa, Dept. of Mechl. Eng., Iowa City, IA 52242 USA

The transient growth of an equiaxed dendrite of pure succinonitrile is investigated using a mesoscopic simulation model. The model uses an analytical expression for the dendrite tip growth coupled with a numerical solution of the temperature field in the melt away from the crystal. It is applied to simulate the actual configuration of the Isothermal Dendritic Growth Experiment (IDGE). The transient variation of the tip velocity is calculated starting from the initial seed growing out of the stinger up to the final interaction of the dendrite with the wall of the growth chamber. The tip velocity experiences a minimum followed by an increase toward a steady state value. This variation is caused by the seeding procedure used in the experiment, whereas the thermal interactions of the dendrite with the chamber wall become important only later. The predictions are found to agree well with the IDGE data.

3:20 PM Break

3:40 PM
Prediction of Microsegregation in Multi-Component Alloys: *Matthew R.M. Shin*[1]; John D. Hunt[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxfordshire OX1 3PH England

A control volume finite difference model has been developed to simulate microsegregation in multi-component alloys. Assuming axial symmetry, the model can handle any number of components or phases. By solving the diffusion equations in all phases and components, the composition profile is calculated as a function of time and temperature. Interface compositions are obtained by calling library procedures of MTDATA, a thermodynamic database package developed by the National Physical Laboratory, England. The program can treat multi-phase reactions. For peritectic-like reactions, the model assumes a secondary phase nucleates and grows on the primary. Eutectic-like reactions are treated similarly, but the new layer will now consist of the two eutectic phases. The program is written using the Windows environment. A user-friendly front-end provides data input and visual output.

4:00 PM
Solidification Partitioning in Model Ni-Base Superalloy Single Crystals: *Gerhard E. Fuchs*[1]; Fermin J. Fela[1]; [1]University of Florida, MSE Dept., PO Box 116400, 116 Rhines Hall, Gainesville, FL 32611-6400 USA

Solution heat treatments are required to eliminate segregation and eutectic gamma/gamma-prime that forms during solidification of single crystal Ni-base superalloys. The refractory metal content of single crystal alloys has steadily increased with each new generation of alloy, which result in increased heat treatment temperatures and times. Attempts to reduce processing costs by reducing the solution heat treatment temperatures and/or times, are not always successful, since complete chemical homogeneity cannot be attained in some cases. An alternative approach to reduce the heat treatment requirements would be reduce the segregation by reducing the solidification partitioning. Therefore, the effect of alloy composition on the solidification segregation of several model single crystal Ni-base superalloys was examined. Detailed microstructural characterization, compositional analysis and DTA testing was performed to determine the effect of alloy composition on segregation, partitioning ratios and phase change temperatures. The results of this study and their potential impact on future alloy development will be discussed.

4:20 PM
An Experimental and Numerical Study of Microsegregation in Ni-Cr-Mo Alloys: *Shawn A. Cefalu*[1]; Graeme Dickinson[1]; Matthew John M. Krane[1]; [1]Purdue University, Sch. of Matls. Eng., 1289 MSEE Bldg., W. Lafayette, IN 47907 USA

Model predictions of microsegregation in ternary alloys are compared to experiments in the Ni-rich corner of the Ni-Cr-Mo system, which contains several commercial alloys. The model represents a novel application of the Scheil formulation, wherein calculations are performed using the equilibrium phase diagram, assuming the liquid to be in equilibrium only with the solid-liquid interface. The model predicts phase fractions, composition profiles, and the effects of open microscale domains and is designed for ease of use with macroscale solidification models. Solidification experiments were conducted on several alloys which exhibit binary eutectic, ternary peritectic, as well as ternary eutectic solidification. Data were obtained from image analysis, electron microprobe analysis and DTA and these results are compared and contrasted with predictions.

4:40 PM
A Phase Field Model for Phase Transformation in Elastically Stressed Solids: *Cha Pil Ryung*[1]; [1]Seoul National University, Div. of Matls. Sci. & Eng., San 56-1, Shillimdong, Kwanakgu, Seoul 151-742 Korea

With the ability to model the kinetics and the pattern formation for the first order phase transformation, a phase field model has been studied by many scientists. In this work, a new phase field model, which considers the effect of coherent elastic strain on growth and equilibrium state of precipitates in an elastically anisotropic system, is presented. It is shown that the existing phase field models including the effect of coherent elastic strain on the first order phase transition have the limitation of the interface thickness due to the two extra potentials: one is due to chemical energy contribution to the interfacial energy and the other is due to strain energy contribution to the interfacial energy. The presented model has no limitation of the interface thickness by defining the interfacial region as the mixture of precipitate and matrix phases with the same chemical potentials but with the different concentrations and modifying the dependency of the misfit strain on the phase field variable (or concentration field). The model is also shown to satisfy the modified Gibbs-Thompson equation with coherent elastic strain effect automatically and reproduce the equilibrium shape of the precipitate by the competition between elastic strain energy and surface free energy. It is shown by a two-dimensional computer simulation that the infinite-range and highly anisotropic strain-induced interaction results in a shape transition from circle to faceted square with {10} habits and rounded corners at the early stage of growth. Then the divergence of solute atom supply at the corners enhances their growth, which results in a concave morphology. It is also shown by two-dimensional computer simulation that in high precipitate density the precipitates have the cuboidal shape and in low precipitate density they grow dendritic, which was reported by many researches about Ni-based super-alloy.

5:00 PM
Precipitating Behavior of Perovskite Phase in the Ti-Bearing Slags under Special Heat Treatment Condition: *Yuhu Xia*[1]; [1]Northeastern University, 119#, Shenyang, LN 110006 China

The perovskite phase was selected as Ti-rich phase by analysis on chemical composition and mineral component composition of the blast furnace slag bearing titanium. The precipitate process of the perovskite phase in Ti-bearing blast furnace slag was studied by quenching method. The perovskite phase starts to precipitate at 1420 and end at 1200. Through image analysis of the perovskite phase from different heat treatment conditions, it showed that melting temperature and cooling rate affect volume fraction and crystal size of the perovskite phase significantly. Higher melting temperature and lower cooling rate is in favor of precipitation, growth and coarsening of the perovskite phase.

General Abstracts: Alloy Phases

Sponsored by: TMS

Program Organizers: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Wednesday PM Room: 113
November 7, 2001 Location: Indiana Convention Center

Session Chair: Ram B. Bhagat, Pennsylvania State University, Appl. Res. Lab., State College, PA 16804-0030 USA

2:00 PM
On the New-Type Phase in Fe-Ni-C Austenite: *S. Bugaychuk*[1]; V. Lindroos[1]; [1]Laboratory of Physical Metallurgy & Materials Science, PO Box 6200, 02015-HUT, Helsinki, Finland

Prof. D. Rancourt and Co. considered low temperature equilibrium state for Fe-(28-40) at.% Ni alloy as an epitaxially intergrown paramagnetic ε-hexagonal phase and ordered (FeNi, $L1_0$) tetrataenite, regardless of the impossibility to distinguish ε-phase with use of structural diffraction tools. Prof. K. Oda noticed that metallurgists of Japan aware of ε-phase formation in Fe-Ni alloys and investigate it to get to know where it forms in bulk material. The present work confirms the presence of ε-like paramagnetic hexagonal non-close packed metastable phase in Fe-22.4 at.%Ni-5.13 at.%C FCC-alloy at room temperature after long high dose 3 MeV electronic irradiation at 100°C through the TEM. The justification for this phase occurrence is offered and based upon the effect of lowering of the Gibbs Free energy of ε-phase at low temperatures, which is demonstrated through the use of Thermo Calc program.

2:20 PM
In-Situ Observation of Phase Transformation in TRIP Steels at High Temperatures using Synchrotron X-Ray Scattering: *Seung Deug Choi*[1]; [1]Research Institute of Industrial Science & Technology, Metals & Coating Matls. Rsrch. Team, PO Box 135, Pohang 790-600 Korea

TRIP steels are a family of steels offering a unique combination of high strength and ductility. This combination is achieved through an appropriate cooling process of run-out table (ROT) in a hot strip mill. In this paper, a new system has been developed for in-situ observation of phase transformation at high temperature using a synchrotron X-ray scattering method. Changes in powder diffraction patterns from a heated specimen can be measured continuously by scanning a wire detector located behind a slit. Synchrotron X-ray scattering of high time and spatial resolution can give a result of quantitative analysis of a retained austenite and carbon content of the retained austenite. In-situ analysis in continuous cooling process can provide information for a phase transformation model.

2:40 PM
Deformation Analysis of Superplastic Alloy Aluminium Al-8090 using Finite Elements: *Noha Mohamed Hassan*[1]; [1]The American University in Cairo, 19 Amr St., Missaha Sq., Dokki, Cairo, Egypt

The deformation behavior of an Al-Li-Cu alloy (Aluminium 8090) during uniaxial and biaxial stress states were modeled using the nonlinear finite element analysis package ABAQUS 5.8. Two different material models were used for the purpose of comparison and to propose a valid and accurate superplastic material deformation behavior. The two models represents a material model with constant properties (strain rate sensitivity m, and strain hardening exponent n) and a material model with variable properties. The power law strain hardening model in ABAQUS was used in both cases. The results from the numerical models on ABAQUS were compared to experimental results analyzing the free bulging behavior of the material considering the deformation caused by a constant applied pressure and during uniaxial testing published by Chen and Huang (1995). Experimental data were primarily used to acquire the needed material parameters for the model. In addition, they were used to verify the results obtained from the finite element model. The strain rate sensitivity m and strain hardening exponent n are dependable variables on the strain and strain rates. Even under extreme conditions of running the test with constant strain rates, these variables are still varying due to the impossibility of having same strain and strain rate throughout section with time. Mainly due to this fact, the assumption of having constant properties when dealing with superplastic materials does not represent a solid one. Upon comparing the two material models in both stress states whether biaxial or uniaxial, material model with variable properties gives better representation of the true behavior of the material when compared to the actual experimental results.

3:00 PM Break

3:20 PM
Grain Boundary Cementite in Austenite: *Milo V. Kral*[1]; [1]University of Canterbury, Dept. of Mechl. Eng., PO Box 4800, Christchurch, New Zealand

Earlier work showed that during isothermal transformation proeutectoid cementite first precipitates along austenite grain boundaries with what appears to be a predominantly dendritic morphology. Further study using transmission electron microscopy showed that most cementite dendrites are monolithic crystals while other dendrite-like morphologies appear to result from multiple nucleation events. Following along in this vein, the crystallographic directions of primary and secondary dendrite arms in cementite were sought. Previous studies of Widmanstätten cementite precipitates showed a strong correlation between morphology and crystallography. In this case however, the lack of a consistent correlation between crystallographic features and morphological features in the cementite dendrites led to a subsequent experimental study of the orientation relationships between grain boundary cementite and the adjacent austenite grains using electron backscattered diffraction. These results showed that the orientations obtained between cementite and one of the austenite grains often approximated one of the known ORs but rarely was close to the ideal ORs obtained for Widmanstätten precipitates. An approach to modeling both matrix grain boundary-precipitate interfaces will also be presented.

3:40 PM
Formation of an Ultra-Fine Lamellar Microstructure in TiAl Based Alloys: *F. Meisenkothen*[1]; G. Viswanathan[1]; R. Banerjee[1]; H. L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

During the growth of PST TiAl based alloys, a lamellar microstructure forms that consists of parallel laths of the α_2 and γ phases. Ordinarily, these laths have average thicknesses on the order of hundreds of nanometers, and they can form with either γ phase adjacent to γ phase, or with γ phase adjacent to α_2 phase, in a seemingly random fashion. Recent work has found that polycrystalline samples having compositions between Ti-42Al & Ti-48Al can be solution heat treated in the α phase field, and then rapidly quenched, to produce a microstructure that consists entirely of the supersaturated α_2 phase. Upon aging the supersaturated α_2 phase in the α_2+γ phase field. A fully lamellar structure results. However, in contrast to the lamellar PST structure, the laths resulting from the aging treatment have fairly uniform thicknesses on the order of tens of nanometers, and they appear to consist of rigidly alternating γ/α_2 phases. Upon continued aging, the ultrafine lamellar structure coarsens via discontinuous coarsening to yield a coarse non-rigidly alternating lath structure. Given the differences in the morphology between the two types of lath structure, it seems possible that their formation mechanisms may be different. Hot stage experiments will be conducted in the TEM with the objective of studying the in-situ decomposition of the supersaturated α_2 phase. The results of these investigations and their implications on the transformation mechanisms in these alloys will be discussed in this paper.

4:00 PM
The Effect of α_2 Phase Morphology on the Room Temperature Ductility of TiAl Based Alloys: *F. Meisenkothen*[1]; R. Banerjee[1]; G. Viswanathan[1]; H. L. Fraser[1]; [1]Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

The gettering of interstitial contaminants, by the α_2 laths of fully lamellar TiAl based alloys, is responsible for the enhanced slip activity that is observed in the γ laths of these alloys. Unfortunately, the increased interstitial content of the α_2 phase leads to a decrease in the number of active slip systems in that phase, and an attendent reduction in its already low plasticity. The brittle α_2 laths act to constrain the γ phase, and thus reduce the overall ductility of the fully lamellar materials. However, by suitably engineering the microstructure of TiAl based alloys it should be possible to employ the gettering benefits provided by the α_2 phase, while eliminating the constraints that the α_2 phase imposes on the γ phase. To this end, a combination of alloying additions (such as Ta) and heat treatments are being explored with the objective of precipitating discrete α_2 within a γ matrix, thus destroying the lamellar microstructure. In addition to microstructural characterization using SEM and TEM, results from mechanical testing of these alloys will be discussed in this paper.

General Abstracts: Chemistry & Physics of Materials - II

Sponsored by: TMS
Program Organizers: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Wednesday PM   Room: 102
November 7, 2001   Location: Indiana Convention Center

Session Chairs: TBA

2:00 PM
Grain Boundary Effects in High Tc Superconductors: *Chandra Shekhar Pande*[1]; [1]Naval Research Laboratory, Code 6325, 4555 Overlook Ave. S.W., Washington, DC 20375 USA

The goal of this study is to characterize both current transport properties and microstructures in high Tc superconductors in order to optimize material performance. Initial work has focused on BSCCO tapes. Superconducting properties were measured as a function of temperature and applied magnetic field for sections of this material. Additionally, the microstructures were characterized using optical and electron microscopy and x-ray diffraction. These microstructural parameters were then correlated with the superconducting properties in order to rank their relative importance and to predict the combinations which should maximize superconductor performance. Flux Creep in these materials were also studied.

2:20 PM
Cyclic Diffusional Phase Transformations in Metal/Gas Diffusion Couples: *Christopher Schuh*[1]; [1]Northwestern University, Matls. Sci. & Eng. Dept., 2225 N. Campus Dr., Evanston, IL 60208 USA

The diffusional growth of surface layers at metal/gas interfaces has been extensively studied due to its technological importance in, e.g., carburizing, nitriding, and thin film processing. This so-called moving boundary diffusion problem, in which the phase front is a discontinuity in the concentration profile and propagates according to a mass balance condition, has also been treated analytically and numerically by many authors. In this talk, a numerical method is used to examine conditions of cyclic phase transformation in metal/gas diffusion couples, induced by cyclic charging and discharging of the metal host lattice with a gaseous alloying element. The problem is treated by introducing a periodic surface concentration at the gas/metal interface, which gives rise to several phase boundaries that coexist and interact within the specimen. Although the present treatment is theoretical, this problem is expected to have implications in, e.g., hydrogen storage and retrieval or transformation superplastic forming induced by chemical cycling.

2:40 PM
Characterization of Grain Boundary Orientation and Energy in Ni-Cr-Fe Alloys: *Thomas N. Skidmore*[1]; Mary Juhas[1]; Rudy Buchheit[1]; Hamish Fraser[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Cracking of Inconel® 690 samples along boundaries of high, but otherwise uncharacterized, misorientation has prompted an investigation of the relationship between grain boundary energy, orientation and propensity for environmental fracture. The distributions of grain boundary energies have been characterized by atomic force microscopy of thermal grooves and ridges on Ni-Cr-Fe samples subject to elevated temperature annealing. Groove root angle is observed to depend upon boundary misorientation. Boundary misorientation distributions are being compiled using orientation imaging microscopy (OIM). Using information from thermal groove angle measurements and OIM data on misorientation, we seek to characterize fundamental properties of Ni-Cr-Fe alloys and establish relationships between grain boundary character and susceptibility to cracking. Ultimately, we seek to provide fundamental understanding of Ni-Cr-Fe for use in mechanistically based models of crack damage accumulation in Ni-Cr-Fe-based alloy systems.

3:00 PM
Use of Ceramics for Filling Joints in Cell Cathode Blocks: *G. V. Solonin*[1]; M. A. Fridman[1]; V. K. Nikitenko[1]; [1]JSC "Zaporozhye Aluminium Combine" (ZALK), 15 Yuzhnoye Shosse, Zaporozhye 69032 Ukraine

The need to improve performance characteristics of cathode lining, in particular to increase cell life, is driven by the high cell repair costs and the disposal of environmentally harmful wastes formed during the reduction cell relining. This paper presents a method of improving the performance characteristics of the cathode blocks in an aluminium reduction cell. A procedure has been developed and the results of the laboratory tests of ceramic materials used in the cathode joints are given. It has been shown that the resistance of joint materials to the melt attack is determined by the porosity of the materials used. The experimental and industrial test results of using a slip casting procedure for filling the cathode blocks joints are given. The results achieved made it possible to eliminate the use of carbon ramming paste from the cathode relining procedure. It has been demonstrated that after the introduction of the new cell relining procedure, there were no premature cell failures even under long electric power outages. Other positive effects of the procedure presented have also been achieved.

3:20 PM Break

3:40 PM
Structural Transitions in Nb-Based Thin Film Multilayers: *Gregory B. Thompson*[1]; Rajarshi Banerjee[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Structural transitions in thin film multilayers often occur at a length scale of a few monolayers to tens of nanometers. As the dimensions of microelectronic devices approaches this length scale, these structural transitions will become increasingly relevant to predict. By viewing structural transitions as a competition between interfacial and volume components of the free energy, simple biphase stability diagrams can be constructed. These biphase diagrams are plots of the inverse of the bilayer spacing versus the volume fraction of one of the components within the bilayer. Thus, a biphase boundary line separates regions of stable and metastable states. Examples of this technique have been applied to Nb-based multilayers. Interestingly, in the case of Zr/Nb structures, a beta-Zr semi-coherent to coherent transition is observed at smaller bilayer thicknesses relative to the hcp-Zr/bcc-Nb and bcc-Zr/bcc-Nb biphase boundary line. Formation of a novel hcp-Nb multilayer has also been grown in this system. These and other experimental results will be discussed.

4:00 PM
Microstructural Characterization of MBE-Deposited $Ge_{1-x}Mn_x$ Thin Films: *Allen W. Wilson*[1]; George Spanos[1]; Y. D. Park[1]; A. Hanbicki[1]; C. S. Hellberg[1]; S. C. Erwin[1]; B. T. Jonker[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., 4555 Overlook Ave. S.W., Washington, DC 20375 USA

During molecular-beam epitaxial growth, 2 to 7.5% Mn was simultaneously deposited with Ge in an attempt to create metastable single phase magnetic Ge with Mn in solution. Such materials have potential use in semiconductor-based magnetic devices. First principles theory predicts a ferromagnetically ordered ground state at low Mn concentrations at a lattice constant very near that of bulk Ge. Deposition onto a (001) GaAs substrate at a temperature of 300°C produces a microstructure containing Mn rich particles of ~100 nm in a Ge matrix. EDS confirms the composition to be $Mn_{11}Ge_8$. Deposition at 70°C produces single crystal epitaxial film growth, but with Mn-rich precipitates which exhibit a thread-like morphology. Plan view TEM results show the precipitates with ~5 nm diameter and the average long axis parallel to the [001] substrate direction. This work was supported by the Office of Naval Research and the DARPA SpinS program.

4:20 PM
Synthesis and Thermodynamic Properties of Zirconium Pyrovanadate Doped with Zirconia and Vanadium(V) Oxide: *Xianran Xing*[1]; Zhenqi Zhu[1]; Xue Qi[1]; [1]University of Science & Technology–Beijing, Dept. of Phys. Chem., Xueyuan Rd. 30, Beijing 100083 China

The main phase ZrV2O7 material, doped with zirconia and vanadium(V) oxide, was synthesized by solid state reaction and sol-gel methods. X-ray power diffraction patterns show that it is cubic structure. Thermal mechanic analysis measurements present a zero-thermal expansion of this material above 150°C. As well the heat capacity dependent on temperature, determined by differential scanning calorimetry, keeps in constant almost in the same temperature range. The relation between unusual thermal expansion and abnormal heat capacity is discussed with quantum mechanics. Keywords: ZrV2O7, zero-thermal expansion, heat capacity, Grüneisen parameter.

4:40 PM
Growth and Characterisation of Gallium INdium Antimonide Thin Films for Infrared Detectors: *Partha Bir Barman*[1]; [1]Multimedia University, Fac. of Eng. & Tech., Jalan Ayer Keroh Lama, 75450 Melaka, Malaysia

Growth of GaxIn1-xSb(x = 0.1 to 0.5) thin films were carried out using Hot wall epitaxy system on high purity quartz glasses and high resistive Silicon as substrates. The source material for the growth of film was grown in the laboratory using vertical Bridgmen method in microprocessor controlled high temperature furnace. The grown films were characterized using grazing angle X-ray diffraction analysis and found to be polycrystalline in nature. XPS studies on the films showed non-stoichiometry compared to the bulk crystals. Band gap measurements using FTIR confirms the formation of energy gap in IR region, but the value does not exactly tally with the expected value of x. SEM studies on the films confirm the formation of pits on the surface which attributes to the non-stoichiometry in the films. Electrical characterization of the film viz. Vander-Pauw resistivity, Hall-mobility, surface carrier concentration, etc. measurement is under progress to study the interplay between the microstructure and electronic properties of polycrystalline semiconducting thin films. Finally Au/GaxIn1-xSb Schottky diodes were fabricated on the grown films and electrically characterised for IR detectors. The results were found to be satisfactory compared to grown films by other expensive and sophisticated techniques viz. MBE, MOCVD, etc.

General Abstracts: Ferrous Metallurgy

Sponsored by: TMS

Program Organizers: TMS, Warrendale, PA 15086 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Wednesday PM — Room: 203
November 7, 2001 — Location: Indiana Convention Center

Session Chair: Eric M. Taleff, The University of Texas at Austin, Matls. Sci. & Eng. Prog., Austin, TX 78705 USA

2:00 PM

Modelling the Overall Austenite Decomposition Kinetics: *Fateh Fazeli*[1]; Matthias Militzer[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., #309-6350 Stores Rd., Vancouver, BC VC1Z4 Canada

A model is developed to describe the overall austenite decomposition kinetics in low carbon steels. The model considers both interface and carbon diffusion-controlled ferrite formation. Solute drag effects of substitutional elements are explicitly incorporated. The inclusion of pearlite and bainite formation into the model will be discussed. Model predictions are validated with experimental data for Fe-C-Mn alloys.

2:20 PM

Mechanical and Magnetic Properties of Heat Treated Hiperco Alloys: *Ben Hailer*[1]; Stephen L. Kampe[1]; William T. Reynolds[1]; [1]Virginia Tech, Matls. Sci. & Eng., 213 Holden Hall, Blacksburg, VA 24061-0237 USA

Iron-cobalt-vanadium (approximately 49Fe-49Co-2V atom%) alloys are soft magnetic materials that can be processed to have high magnetic saturation, relatively high yield strength, and moderate core loss. This combination of properties makes these alloys well-suited for high-stress applications such as switched reluctance motor/generator cores. This study quantitatively measures the changes in strength, ductility, magnetic permeability, coercivity, remnant induction, and core loss with annealing times varying from 10 min. up to 2 hours and temperatures from 640 to 800°C. It has been found that annealing at temperatures below 720°C produces small changes in properties. However, recrystallization occurs when annealing is conducted for approximately one hour between 720 and 740°C, this leads to lower strength, ductility, and core loss. Other magnetic parameters were observed to improve with longer times and higher temperatures. These changes are a direct result of changes observed in the microstructure.

2:40 PM

The Austempering Kinetics, Mechanical Properties, and Microstructure in High Nickel Alloyed Ductile Iron: *David J. Heal*[1]; *Tara Chandra*[1]; [1]University of Wollongong, Eng. Fac., Northfields Ave., Wollongong, NSW 2522 Australia

High temperature dilatometry has been used to study the effect of nickel on the austempering behaviour in alloyed ductile iron of two different compositions. Material A contains 3.69%C, 2.22%Si, 0.27%Mn, 0.23%Mo, 0.05wt%Mg, 1.15%Cu, & 0.93%Ni while material B has a similar composition except much higher nickel content of 2.24wt%. Both alloys were austenitised at 920°C and then austempered at 375 and 325°C for varying times to study the relationship between microstructural development, mechanical properties and austempering kinetics. The unreacted austenite volume (UAV) as a function of austempering time was used to determine the austempering kinetics. The austempering kinetics for material B was substantially retarded as compared to material A, as a result of much higher nickel content. It was found that the kinetics was sluggish at 375°C compared to 325°C for these alloys. The microstructural study indicated that the second stage reaction occurred in the eutectic cell while the first stage reaction is incomplete in the intercellular regions in both alloys at austempering temperature of 375°C.

3:00 PM

A Study of Sulfur Behaviors in Austenite Stainless Steels at High Temperature: *Kim Hie Jin*[1]; Lee Yun Yong[2]; [1]Hanyang University, Dept. of Metall. & Matls. Sci. Eng., Sadong, Ansan, Kyunggido 425-791 S. Korea; [2]Pohang Iron & Steel Co., Ltd., Stainless Steel Rsrch. Grp. Techl. Rsrch. Labs., PO Box 36, Pohang, Kyungbuk 790-785 S. Korea

It is known that austenite stainless steels are susceptible to hot cracking although they exhibit excellent mechanical properties at high temperature. It is considered that the hot cracking may result from the precipitation or segregation of sulfur. However, there exists little information on the exact mechanism. The sulfur behaviors have been studied in 310S stainless steels using hot tensile test. For the hot tensile tests, samples held at 1523K for 300 sec, and cooled to 1323K with the various cooling rate of 10~50 K/sec. Thereafter, samples was deformed after holding 10~600 sec. As a result, the values of R. A. increased as the holding time increased. However, the variations of the cooling rate resulted in almost no significant change of the values of R. A. Utilizing in-situ AES, it was observed that sulfur initially segregated in grain boundaries. As the holding time increased, sulfides were found to be formed.

3:20 PM Break

3:40 PM

The Influences of Microstructure and Nitrogen Alloying on Hot Ductility of 304 Austenite Stainless Steels: *Kim Hie Jin*[1]; Kwon So Young[1]; Lee Yun Yong[2]; [1]Hanyang University, Dept. of Metall. & Matls. Sci. Eng., Sadong, Ansan, Kyunggido 425-791 S. Korea; [2]Pohang Iron & Steel Company, Ltd., Stainless Steel Rsrch. Grp. Tech. Rsrch. Labs., PO Box 36, Pohang, Kyungbuk 790-785 S. Korea

An investigation has been made on the effect of nitrogen content on the hot ductility of austenite stainless steels containing 18Cr-9Ni-1.5Si at 1~3000 hrs in the temperature range from 873K to 1173K. The nitrogen added 800 and 1400 ppm and Nb, W, Ti varied. The NbN, TiN, W and M23C6 carbide were recognized in all specimens regardless of their heat treating condition. However, Cr2N and TiN were recognized by long time aging in the nitrogen containing specimen. It was found that increasing nitrogen content decreased the hot ductility below the temperature of 1173K due to the decrease of stacking fault energy. The addition of nitrogen into austenite stainless steel markedly retarded the coarsening of precipitates and promoted homogeneous distribution of precipitates during isothermal aging. The orientation relationships between the matrix and the precipitates formed aging treatment were also elucidated.

4:00 PM

Determination of the Size Distributions of R-Phase and Monoclinic Phase by using Energy Filtered Transmission Electron Microscopy: *Aytekin Hitit*[1]; Warren M. Garrison[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Two types of precipitates, R-phase and monoclinic phase, were identified in a 12Cr/12Co/5Mo/4.5-6Ni martensitic precipitation strengthened stainless. It is found that the average size of R-phase particles is greater than that of monoclinic phase at the tempering temperatures at and above 525°C. EDX results showed that there is some overlap between the size distributions of the phases. In other words, some of the larger monoclinic phase particles are larger than some of the smaller R-phase particles. In order to determine the size distributions of the phases separately, dark field images were obtained by using the spots from each precipitates. However, because the spots obtained from the precipitates are often too close to each other, dark field images contained both types of precipitates. Iron, molybdenum and chromium contents of the phases and the matrix are quite different. Therefore, Energy Filtered TEM (EFTEM) imaging technique seems to be promising to determine the size distributions of the phases. In this study, size distributions of the phase will be determined as a function of tempering temperature by EFTEM method.

4:20 PM

Optimization of Geometric Profile of Well Block and Upper Nozzle in Continuous Casting Tundish: *Jung-Eui Lee*[1]; *Wan Wook Huh*[1]; *Hyung Tae Chung*[2]; *Taewook Kang*[2]; [1]POSCO, Kwangyang Iron & Steelmaking Rsrch. Labs., Tech. Rsrch. Labs, 699, Kwangyang, Cheonnam 545-090 Korea; [2]RIST, Strip Casting Proj. Team, No. 32, Hyoja-dong, Nam-ku, Pohang, Kyungbuk 790-330 Korea

Geometric profile of well block and upper nozzle for continuous casting tundish at Kwangyang Works, POSCO was designed to avoid flow separation, which causes nozzle clogging and degradation of CR coil quality. Flow pattern was visualized using polyethylene sphere and hydrogen bubble in 1/2-scaled water model. Numerical analyses were performed to more clearly understand the geometric effects of the nozzle on the inclusion behavior and on the flow separation. From the results, the stepwise flow passage was revised into a smooth one to reduce the local separation of the flow remarkably. Plant tests proved that the new design was effective to prevention of the inclusion accumulation in the upper part of upper nozzle.

4:40 PM
Corrosion Mechanism of MgO-C Base Refractory in Melt of Smelting Reduction with Iron Bath: *Qingcai Liu*[1]; Joseph W. Newkirk[1]; Jing Lin[2]; Dengfu Chen[3]; [1]University of Missouri–Rolla, Dept. of Metlgcl. Eng., McNutt Hall, Rolla, MO 65401 USA; [2]Chongqing University–District B, Dept. of Sci. & Tech., 185 Sapinba N. St., Chongqing 400045 China; [3]Chongqing University, Sch. of Matls. Sci. & Eng., 174 Sapinba St., Chongqing 400044 China

The corrosion mechanism of MgO-C base refractory in the melts of smelting reduction with iron bath was studied using rotary immersion and quasi-static immersion tests. The microstructures and composition of the refractory were investigated by SEM and X-Ray Diffraction. The influence of the additions TiC and ZrO_2 in MgO-C base refractory on the corrosion behavior of the refractory is in evidence. The corrosion rate of the refractory is decreased greatly with the additions of ZrO_2 and TiC and is increased as FeO concentration of melt increases and stirring strength of molten bath increases. The temperature of the molten bath is one of the important factors effecting the corrosion behavior of refractory. Additions of TiC and ZrO_2 in a MgO-C refractory improves the anti-corrosion ability of the refractory. The corrosion mechanism of MgO-C base refractory is due to the interaction between the refractory constituents and melt to form a deteriorated layer.

General Abstracts: Mechanical Metallurgy and Composite Materials

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Wednesday PM Room: 115
November 7, 2001 Location: Indiana Convention Center

Session Chairs: Amit K. Ghosh, University of Michigan, Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109-2136 USA; Enrique V. Barrera, Rice University, Mechl. Eng. & Matls. Sci., Houston, TX 77005 USA

2:00 PM
A Simplified Solution for the Stress Field of a Misfitting Particle: *Qizhen Li*[1]; *Peter M. Anderson*[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

The critical stress to push a dislocation loop through an array of obstacles is a fundamental quantity that depends on the detailed elastic interaction between the loop and the particle. We first present a simple, analytic method to obtain the stress field from a misfitting elastic particle. That solution reproduces earlier reported solutions but is simpler to implement. This result is then used to study the propagation of a dislocation loop past arrays of misfitting particles that are characterized by a misfit strain as well as particle size and spacing distributions. The loop propagation is modeled by a cellular automaton approach in which the slip plane is discretized into small rectangular sections which can shear provided the energy of the material and loads is reduced. The results suggest features of the particle distribution which control the strength of a precipitate-reinforced plane to shearing.

2:20 PM
The Effect of Interfacial Bonding on the Critical Stress to Transmit a Screw Dislocation: Zhiyong Li[1]; *Peter M. Anderson*[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

The critical stress to transmit a dislocation across an interface is a fundamental quantity that controls, in part, the resistance of many crystalline materials to plastic deformation. Our analysis considers a Peierls analysis of the transmission of a screw dislocation across an interface. The properties of bonds across the incoming and outgoing slip planes, as well as the interface, can be varied in order to study the effect on the critical macroscopic shear stress to transmit the dislocation. One of the most dramatic effects occurs when the unstable stacking fault energy associated with slip of the interface is reduced. In that case, the critical stress to transmit can increase by several hundred percent, due to the delocalization of the dislocation core into the interface. We also discuss the associated changes in activation energy for transmission, and use this information to discuss three-dimensional loop configurations for dislocation transmission.

2:40 PM
Effect of Chamfered Narrow Mold on Corner Cracking of Continuous Casting Carbon Steel: *Woong Ho Bang*[1]; Kyu Hwan Oh[1]; Joo Choi[2]; Chang Hee Yim[2]; [1]Seoul National University, Sch. of Matls. Sci. & Eng., San 56-1, Shinrim-dong, Kwanak-ku, Seoul 151-742 Korea; [2]POSCO, Iron & Steel Rsrch. Team, Tech. Rsrch. Lab., PO Box 36, 1 Koedong-dong, Pohang, Kyungbuk 790-785 Korea

During continuous casting, fast cooling of slab corner in mold leads to ductility trough and high thermal stress of corner region, and finally causes corner cracking. To reduce corner cracking, uniform cooling of slab in mold is required, and the condition can be obtained by modification of mold geometry. FDM was used to calculate LIT~ZDT zone which is main temperature range of crack generation of carbon steels, and 2-dimensional thermal-stress coupled FEM was used to evaluate the effect of chamfered narrow mold on corner cracking. Durability of continuous casting mold is mainly determined by plastic strain and wear which are main factors of mold damage during operation. Wear parameter of common and chamfered mold is calculated with 2-dimensional FEM to compare wear condition according to mold shape. 3-dimensional FEM program, ABAQUS is used to analyze thermal stress and strain condition of narrow mold during operation.

3:00 PM
Scale-Up Effects for Equal Channel Angular Extrusion: *Robert E. Barber*[1]; K. Ted Hartwig[1]; [1]Texas A&M University, Mechl. Eng., College Station, TX 77843-3123 USA

Equal Channel Angular Extrusion (ECAE) is an innovative process for introducing severe plastic deformation (SPD) into bulk material. Characteristics of the process include producing uniform strain and having substantial control over the development of product microstructure including texture. Other benefits are relatively low extrusion pressure and the opportunity for easy scale-up. Two notable results of multipass ECAE are that extremely fine microstructures and very high strengths can be developed in bulk material by this process. Results will be reported on how the mechanical properties, strain uniformity, and punch pressure varies for pure copper as the workpiece cross-sectional area increases from 161 to 2581 mm^2.

3:20 PM
Experimental and Numerical Analysis of Heat Transfer and Residual Stress Distribution during Bead-On-Plate GMAW Welding: *Xuan Hung Ha*[1]; In-Wan Bang[1]; Kyu Hwan Oh[1]; [1]Seoul National University, Sch. of Matls. Sci. & Eng., San 56-1 Shinrim-dong, Kwanak-ku, Seoul 151-742 Korea

The experimental and numerical study has been conducted on the bead-on-plate GMAW welding of pipeline steel. The temperature, HAZ hardness and residual stress distributions of the bead-on-plate welding have been observed according to the welding conditions. Experimental measurements on the residual stress distributions were carried out using center hole drilling and X-ray diffraction techniques. 3-dimensional finite element analysis has been conducted to predict the residual stresses in the bead-on-plate welding. The thermal and mechanical analyses were performed with the commercial finite element program, ABAQUS. The temperature histories and the residual stress distributions of surface and through-thickness directions were measured and predicted. The predicted results were found to be in good agreement with the experimental results.

3:40 PM Break

4:00 PM
Creep Behavior for In-Situ Single Crystal Oxide Ceramic Eutectic Composites at Ultra-High Temperature: *Yoshihisa Harada*[1]; Takayuki Suzuki[1]; Kazumi Hirano[1]; Yoshiharu Waku[2]; [1]National Institute of Advanced Industrial Science and Technology, Inst. for Mechl. Sys. Eng., Tsukuba E., Namiki 1-2, Ibaraki 305-8564 Japan; [2]Japan Ultra-High Temperature Materials Research Institute, Proj. Dept., Okiube 573-3, Ube, Yamaguchi 755-0001 Japan

In-situ single crystal oxide ceramic eutectic composites such as Al2O3/YAG, Al2O3/GAP and Al2O3/EAG, are expected to be one of the ultra-high temperature structural materials in the field of power generator industry and aerospace industry, because of their low density, good oxidation resistance and potentially useful high strength at ultra-high temperature. Little research has however been performed on the mechanical properties of these composites at ultra-high temperature. In this study, systematic creep study was undertaken for in-situ Al2O3/YAG eutectic composites. Creep tests were conducted in the temperature range from 1773 to 1973 K under a constant compressive or tensile stress ranging from 70 to 200 MPa. Creep behavior for in-situ Al2O3/YAG eutectic composites was examined by the influences of temperature, stress, and orientation dependency. Also, creep behavior in tension was examined in moisture-rich environments.

4:20 PM
Cyclic Plasticity of Single Crystal Nickel at Various Plastic Strain Amplitudes: Constricted Hysteresis Loops: *Yan Jia*[1]; David Morrison[1]; John Moosbrugger[1]; [1]Clarkson University, Mech. & Aeros. Eng., PO Box 5727, Potsdam, NY 13699 USA

The cyclic plasticity of single crystal nickel was studied by performing fully reversed tension-compression fatigue tests at constant plastic strain amplitudes of 1.0x10-3, 2.5x10-4 and 1.0x10-4 at room temperature. The cyclic plasticity behavior within a hysteresis loop was analyzed by measuring the curvature of the loop which is directly related to the probability density function, $f(\sigma)$. In the Masing model of kinematic hardening, this probability density function can be related to the frequency distribution of material volume element yield stresses. This study revealed a distinct constriction in the stress-plastic strain hysteresis loops. The constrictions were much more pronounced as plastic strain amplitude decreased. Results indicated that this constrictive behavior was a manifestation of the magnetostrictive effect in nickel and was also influenced by dislocation structure and internal stress condition. This research was supported by the National Science Foundation under grant CMS 9821021.

4:40 PM
Competing Mechanisms and Modeling of Deformation in Austenitic Stainless Steel Single Crystals With and Without Nitrogen: *Ibrahim Karaman*[1]; H. Sehitoglu[2]; H. J. Maier[3]; Y. I. Chumlyakov[4]; [1]Texas A&M University, Mechl. Eng., College Station, TX 77843 USA; [2]University of Illinois at Urbana–Champaign, Mechl. & Indl. Eng., Urbana, IL 61801 USA; [3]University of Paderborn, Lehrstuhl f. Werkstoffkunde, Paderborn D-33095 Germany; [4]Siberian Physical-Technical Institute, Tomsk 634050 Russia

The stress-strain behavior of low stacking fault energy AISI 316L austenitic stainless steel (SS) (Fe, 17 Cr, 12 Ni, 2 Mn, and 0.75 Si in wt.) single crystals was studied for selected crystallographic orientations ([111], [001], and [123]) under tension. Nitrogen (0.4 wt%) was added to the [111], [001] and [011] crystals. The monotonic deformation of 316L SS was presented with and without nitrogen. The overall stress-strain response was strongly dependent on the crystallographic orientation. Transmission electron microscopy demonstrated for the first time that twinning was present in the [111] orientation of the nitrogen free 316L SS at very low strains (3%) and in the [123] and [001] orientations at moderate strains (~10%). Twinning boundaries led to a very high strain hardening coefficient by restraining the dislocation mean free path. The nitrogen addition at the present level caused following significant changes in the stress strain response: 1) a considerable increase in the critical resolved shear stresses leading to a deviation from Schmid Law; 2) suppression of twinning although planar slip was evident; 3) change in the primary deformation mechanisms; and 4) a decrease in strain hardening coefficients. Most of these differences stemmed from the non-monotonous change in the stacking fault energy with nitrogen concentration and the role of short-range order. A unique strain hardening approach was introduced into a viscoplastic self-consistent (VPSC) formulation. The strain hardening formulation incorporates length scales associated with spacing between twin lamellae (or grain size and dislocation cell size) as well as statistical dislocation storage and the dynamic recovery. The simulations correctly predicted the stress strain response of both nitrogen free and nitrogen alloyed 316L SS single crystals.

5:00 PM
Nondestructive Evaluation (NDE) and Fatigue Behavior of Nextel/Blackglas Composites: *Jeongguk Kim*[1]; Peter K. Liaw[1]; Hsin Wang[2]; [1]University of Tennessee, Dept. of MSE, 323 Dougherty, Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Cer., High Temp. Matls. Lab., Oak Ridge, TN 37831 USA

High-cycle fatigue behavior of Nextel 312 fiber reinforced Blackglas ceramic matrix composites (CMCs) was investigated with the aid of nondestructive evaluation (NDE) techniques. Several NDE methods, such as ultrasonic testing (UT), infrared (IR) thermography, and acoustic emission (AE) techniques, were employed for this investigation. Prior to fatigue testing, UT and IR thermography were used to characterize the initial defect distribution of CMC samples, i.e., developing ultrasonic C-scans and thermal diffusivity maps, respectively. During fatigue testing, AE sensors and an IR camera were used for in-situ monitoring of progressive damages of CMC samples. A stress versus cycles to failure (S-N) curve has been provided to predict the lifetime of CMC samples as functions of initial defects and progressive damages. UT and IR thermography were conducted on fractured samples after fatigue testing to compare progressive damages with the initial defects. Microstructural characterization using scanning electron microscopy (SEM) was performed to investigate fracture mechanisms of Nextel/Blackglas samples. In this paper, NDE techniques were used to facilitate a better understanding of fracture mechanisms of Nextel/Blackglas during high-cycle fatigue.

5:20 PM
Unified Approach to Fatigue Crack Growth I-General Principles: *Kuntimaddi Sadananda*[1]; A. K. Vasudevan[2]; Roland L. Holtz[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., 4500 Overlook Ave., Washington, DC 22152 USA; [2]Office of Naval Research, 800 N. Quincy St., Arlington, VA 22217 USA

General principles under the Unified Approach to fatigue crack growth developed by the authors will be presented. Fatigue is shown to be a two-load parameter problem involving amplitude and maximum load. For fatigue crack growth this manifests as two driving forces, *K and Kmax and two thresholds *K*th and K*max,th that must be met simultaneously for crack to grow. Consideration of these two parameters is generally sufficient to uniquely describe the crack growth behavior without the need of crack closure. The approach is not an alternative to crack closure concept as some critiques like to consider but fundamental to crack growth. If crack closure does exist then it should be considered as an additional factor. All the phenomena that have been hitherto attributed to crack closure can be effectively described by the Unified Approach. This includes apparent anomalous behavior of short cracks, overload and underload effects, tension-compression, compression-compression fatigue, etc.

5:40 PM
Unified Approach to Fatigue Crack Growth II-Environmental Effects: *Kuntimaddi Sadananda*[1]; Ronald L. Holtz[1]; A. K. Vasudevan[2]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., 4500 Overlook Ave., Washington, DC 20375 USA; [2]Office of Naval Research, 800 N. Quincy St., Arlington, VA 22217 USA

The Unified Approach to Fatigue Crack Growth is extended to analyze the environmental effects on crack growth. Since the Unified Approach involves two driving forces, of which one is Kmax, environmental effects naturally can be included as they are time and Kmax dependent. We show here how the crack growth rates as a function of load ratio can be used to develop crack growth trajectories. These involve *K and Kmax that define the relative roles of the two parameters as function of crack growth rates. The trajectories change with the nature of the environmental effects on crack growth. We have developed a simple classification to characterize these effects based on *K-Kmax trajectory map. In the process we show the Unified Approach provides a powerful tool to characterize the environmental effects.

General Abstracts: Polymers

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Wednesday PM   Room: 111
November 7, 2001   Location: Indiana Convention Center

Session Chair: George T. (Rusty) Gray, III, Los Alamos National Lab, Dynamic Properties Team, Los Alamos, NM 87544 USA

2:00 PM
Effect of Strain-Rate and Temperature on the Mechanical Properties of Plastic-Bonded Explosive Binder Materials: *Carl M. Cady*[1]; William R. Blumenthal[1]; George T. Gray[1]; Deanne J. Idar[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, DX-DO, MS C920, Los Alamos, NM 87545 USA

Recently, interest has been shown concerning the mechanical response of plastic-bonded explosives (PBX) to enable the development of predictive materials models describing the mechanical behavior of these composites. Accordingly, detailed information about the constituents is crucial. Compression measurements were conducted on three explosive binders as a function of temperature and strain rate. The visco-elastic recovery of the binders is seen to dominate the mechanical behavior at temperatures above the glass transition temperature (Tg). The binders exhibited increasing elastic loading moduli, E, with increasing strain rate or decreasing temperature, which is similar to other polymeric materials. Also, the Tg shifts to higher temperatures as the strain rate is increased. The binders are shown to be sensitive to strain rate and temperature below Tg. The binders exhibit a yield behavior, followed by an increasing maximum flow stress, sm, or the strain-at-maximum stress, em, which occurs when the binder first yields for increasing strain rate or decreasing temperature.

2:20 PM

Molecular Model of Macromolecular Chain Rupture in Loaded Oriented Crystalline Polymer: *Ulmas Gafurov*[1]; [1]Institute of Nuclear Physics, Ulugbek, Tashkent 702132, Uzbekistan

Molecular model of thermo-fluctuation slippage and thermo-fluctuation breakage connection of stressed macromolecular chain in creep process of a loaded linear high-oriented amorphous-crystalline polymer type of polyethylene is suggested. It is used the Frenkel-Kontorova's crowdion model. The slippage of a fastened passage polymer chain happens by formation and localization of this type dislocation in polymer crystallite. We considered conditions under which thermofluctuational breakage of a chemical bond of a stretched linear atomic macromolecular chain takes place. The favorable condition to chain rupture are formed for stressed section of macromolecule fixed on polymer crystallite surface or in entanglements. The activation energy of the thermofluctuation rupture of an amorpous section is calculated. It is taken into account complex interaction between slippage and rupture of polymer chains. The activation energy of the flexible-chain polymer creep and fracture processes is defined by the activation energy of macromolecular slippage process in the conditions.

2:40 PM

Variation of Local Loads in Deformation Molecular Processes in an Oriented Flexible-Chain Crystalline Polymer: *Ulmas Gafurov*[1]; [1]Institute of Nuclear Physics, Ulugbek, Tashkent 702132, Uzbekistan

On the base of measurable molecular and supermolecular structural and deformation parameters was estimated the distribution of the local loads (correspondingly overstrain coefficients) on the passing macromolecular amorphous sections and redistribution ones with conformational regroupings and macromolecular slippage in deformation process of an oriented flexible-chain crystal-line polymer. The biphase model of a linear high-oriented crystalline polymer of fibrillary microstructure with alternating amorphous and crystalline regions type of linear polyethylene is considered. For the simplification of our calculations we shall guess that passing straightened sections are intermolecularly independent or weakly connected with each other. Also we shall believe that the deformation of the highly oriented polymer is primarily defined by stretching of the amorphous sections being inside fibril. But since straightened regions can be considerably loaded then in stressed polymer sample one should take into account the stretching of the chains in the crystalline regions. For its and at constructions of the molecular chain slippage is used of the Frenkel-Kontorova's crowdion model. We considered redistribution of local loads at conformation regroupings and slippage of passage of macromolecular chain and additional polymer lengthening. Because of steric conditions and discreet character for rotary-isomeric transformations it is demanded some preliminary elastic lengthening of passing sections in order to realize their of conformation reogroupings. Limits of loads of deformation within which conformation reconstructions takes place characterize dispersions on lengths and conformation structure of passing amorphous sections. Using different conformation models of kink-isomers of amorphous sections we have calculated the number of rotary-isomeric transformation corresponding to given lengthening. One can find by number computation relationship between molecular structural parameters for the passing chains and elasticity modulus of the polymer sample.

3:00 PM

Crosslinking Methods for Enhanced Tribological Properties of Ultrahigh-Molecular-Weight Polyethylene for Total Joint Arthroplasty: *Mark L. Morrison*[1]; Raymond A. Buchanan[1]; Roberto S. Benson[1]; Peter K. Liaw[1]; Carl J. McHargue[1]; Warren O. Haggard[2]; Kim Sevo[2]; Kelly Richelsoph[2]; Jason D. Fowlkes[1]; Anthony J. Pedraza[1]; [1]University of Tennessee, Dept. of Matls. Sci. & Eng., 434 Dougherty Engineering Bldg., Knoxville, TN 37996-2200 USA; [2]Wright Medical Technology, 5677 Airline Rd., Arlington, TN 38002 USA

One of the primary life-limiting factors for modern total joint arthroplasty components is ultrahigh-molecular-weight polyethylene (UHMWPE) wear-debris-induced periprosthetic osteolysis, which leads to aseptic implant loosening and, ultimately, the need for revision surgery. Therefore, substantially reducing the wear rate of UHMWPE orthopaedic implant components is critical to extending the in vivo lifetime of these implants. This work is an effort to compare the wear performance of a novel radiation treatment, UV radiation, to that of unmodified and gamma-irradiated UHMWPE. UHMWPE extruded rod stock was exposed to gamma radiation (7.5 Mrads) and ultra-violet (UV) radiation (l=248 nm) at various doses to induce crosslinking within the polymer. Pin-on-disk wear tests were performed for a simulated 6 years of in vivo use and the amount of wear was quantified via profilometry. The wear resistance of these samples was compared to that of compression-molded and unmodified, extruded material. Wear tests were performed with UHMWPE disks against a Co-Cr-Mo (ASTM F799) pin at a contact stress of 3.45 MPa. Diluted bovine blood serum with additions of sodium azide (NaN3) and EDTA was utilized as the lubricant to simulate synovial fluid. Results obtained to date demonstrate that UV irradiation of UHMWPE does improve the wear resistance. The UV radiation dosages utilized in this evaluation produced improved wear rates similar to those witnessed for the gamma-irradiated samples.

General Abstracts: Surface Engineering

Sponsored by: TMS

Program Organizers: TMS, Warrendale, PA 15086 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Wednesday PM Room: 108
November 7, 2001 Location: Indiana Convention Center

Session Chair: Jyotirmoy Mazumder, University of Michigan, Mechl. Eng., Ann Arbor, MI 48109-2125 USA

2:00 PM

Orientation Imaging Microscopy of Plutonium-Gallium Alloys: *Carl Joseph Boehlert*[1]; Roland Kim Schulze[1]; Jeremy N. Mitchell[1]; Thomas G. Zocco[1]; [1]Los Alamos National Laboratory, NMT-16, MS G721, Los Alamos, NM 87545 USA

Microstructural characterization of plutonium-gallium (Pu-Ga) alloys by orientation imaging microscopy (OIM) is nontrivial due primarily to the difficulty in handling this extremely toxic material as well as the rapid surface oxidation rate associated with Pu. Recent work at Los Alamos National Laboratory has shown that by minimizing the surface oxide layer, successful capture of electron backscattered diffraction patterns (EBSPs) of the underlying metal is possible. This work highlights the experimental techniques used and the microstructural features of the Pu-Ga alloy system. The experimental techniques included standard metallographic practices performed inside gloveboxes and/or other enclosures to prevent the spread of radioactive contamination. A scanning auger microprobe (SAM), equipped with an ion-sputter gun, was used to characterize the level of cleanliness and remove chemical impurities concentrated in the surface layers. After ion sputtering, a specially designed vacuum transfer device was used to minimize oxidation during the sample transfer from the SAM to the SEM. EBSPs of the delta-phase (face-centered-cubic structure) were captured.

2:20 PM

The Principle and Commercial Applications of Highly Efficient Regenerative Industrial Furnaces: *Hou Changlian*[1]; Liu Rixin[1]; [1]Beijing North–Island Energy Technology Company, Ltd., Beijing 100029 China

The Principle and commercial applications of highly efficient regenerative industrial furnaces are introduced. The high-efficiency regenerative industrial furnace (NIF) invented by Beijing North-Island Energy Technology Co., Ltd. combines the high-efficiency regenerative heat recovery system with the furnace. The fuel gas and air are pre-heated over 1100°. The exhausted gas temperature is less than 150°. The heat efficiency of NIF is over 70%. The single blast furnace gas can be used to hot-rolling reheating furnace. 21 sets of NIF have been used in China steel company. The application results show that the NIF is greatly benefit to energy-saving, consumption-decreasing and environment protection.

2:40 PM

An Analysis of Stress Waves in 12Cr Steel, Stellite 6B and TiN by Liquid Impact Loading: *Whungwhoe Kim*[1]; Minku Lee[1]; [1]KAERI, Nuclear Matl. Dvlp. Grp., Yousung-gu, Ducjin dong, Teajon 305-353 Korea

This research placed emphasis on the computer simulated stress distribution on the surface and in the bulk of the materials subjected to a water jet impact which might result in erosion damage. The erosion damage was predicted by evaluating the spatial and temporal stress wave distribution generated by water impact pressure on 12 Cr steel and Stellite 6B as steam turbine materials and TiN as a hard coating material. There were two distinctive stress wave behaviors. Firstly, the large tensile stress at the surface was developed by the Rayleigh wave component which appeared between the water drop and the Rayleigh wave front. After the Rayleigh wave detached from the water drop, the materials were in the tensile stress state which could be related to fracture initiation. Secondly, the largest tensile stress in the bulk was near the surface due to the Rayleigh wave generated at the surface and it decreased due to the enlargement of the wave front as the radial distance increased. The Rayleigh wave shape broadened due to the difference between the contact point velocity and the wave front velocity, while its value decayed exponentially in the depth direction. Also, there may be a tendency to produce a circumferential crack by órr near the surface and a lateral crack by ózz in the sub-surface.

3:00 PM
Molecular Dynamics Study of Amorphous Au Cluster Deposits on Gold (001) Surfaces: *Hoseok Nam*[1]; Seung-Cheol Lee[1]; Nong Moon Hwang[1]; Jong-Kyu Yoon[1]; [1]Seoul National University, Sch. of Matls. Sci. & Eng., San 56-1 Shinrim-2, Dong Kwanak-ku, Seoul 151-742 Korea

Soft landing of gold clusters on a gold (001) surface at room temperature was studied using molecular dynamics. The slowing down energy range was 0-1 eV/atom, which is characteristic of low energy cluster beam deposition (LECBD) and the potential of classical molecular dynamics is based on the embedded atom method. Amorphous clusters that were quenched from 1300 K were simulated together with crystalline clusters to see the effects of cluster structure on the deposition behavior. Partial epitaxy of the gold cluster was found in both the cases but the mechanism of atomic rearrangement was somewhat different with each other. The structure and the radial pair correlation functions are used to study the details of the epitaxial properties.

3:20 PM
Studies on Thick W2C Ceramic Coating Fabricated by Self-Propagating High Temperature Synthesis Casting Route: *Pei Qing La*[1]; Qunji Xue[1]; Weimin Liu[1]; [1]Lanzhoy Insititute of Chemical Physics, State Key Lab. of Solid Lubrication, Lanzhou, Gansu 730000 China

Thick W2C coating was tried to prepare on a carbon steel substrate by self-propagating high temperature synthesis (SHS) casting route for exploring the potential of the SHS casting to fabricate thick ceramic coatings. The prepared coating was characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) with energy dispersive spectrum (EDS). Hardness, toughness and wear rate of the coating were evaluated. The results showed that the coating was a composite that consisted of W2C and W phase and had high purity and fine microstructure. Microstructure, hardness and Fe and W content of the steel substrate and the coating regions near the interface varied with the distance to the interface and thus a metallurgical bonding interface was formed. Hardness and toughness of the bulk coating were high and the coating had good wear resistance. It indicated that thick W2C based coating with fine microstructure and good properties can be prepared by the SHS casting. By analyzing the thick W2C based coating developing mechanisms, it was concluded that thick ceramic coatings with good properties could be fabricated on the carbon steel by the SHS casting if the ceramic materials can be produced and melted by a large exothermic chemical reaction and there were diffusions of the steel substrate and coating elements across the interface.

Lead-Free, Lead-Bearing Solders & Alternative Surface Finishes for Electronic Packaging - III: Interfacial Reactions, Intermetallics

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Srini Chada, Motorola, Department APTC, Fort Lauderdale, FL 33322 USA; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu City 00143 Taiwan; Gautam Ghosh, Northwestern University, Department of Materials Science, Evanston, IL 60208-3108 USA; Prasad Godavarti, FlipChip Technologies, Phoenix, AZ 85034 USA; Zequn Mei, Agilent Technologies, Inc., Palo Alto, CA 94304-1392 USA

Wednesday PM   Room: 202
November 7, 2001   Location: Indiana Convention Center

Session Chairs: C. Robert Kao, National Central University, Dept. of Cheml. & Matls. Eng., Chungli City, Taiwan; Sinn-Wen Chen, National Tsing-Hua University, Dept. of Cheml. Eng., Hsin-Chu 00143 Taiwan

2:00 PM Invited
Kinetics of Redeposition of Au-Containing Intermetallics at Solder/UBM Interface during Aging Treatment: *Yong-Seog Kim*[1]; Jong-Hyun Lee[1]; Yong-Ho Lee[1]; Dong Hyuk Shin[2]; [1]Hongik University, Matls. Sci. & Eng., 72-1 Sangsu Dong Mapo Ku, Seoul 121-791 Korea; [2]Hanyan University, Matls. Sci. & Eng., Sa 1 Dong, Ansan Si, Kyunggi Do 425-791 Korea

Redeposition of Au-containing intermetallics on Au/Ni/Cu UBM pads during aging treatment has been one of the main concerns in electronic packages for a possibility of Au-embrittlement in solder interconnections. The redeposition rates of the Au-containing intermetallics in eutectic Sn-Pb and Sn-Ag solders were measured experimentally and analyzed theoretically. The experimental results indicated that thickness of the redeposited layer increases in a power law type behavior and its time exponent varies from 0.4 and 0.5 depending on the types of solder and thickness of Au layer. The redeposition kinetics were analyzed theoretically assuming various rate controlling steps, such as decomposition of AuSn4, diffusion of Au through solder matrix, redeposition reaction of the Au-containing intermetallic, and diffusion of Ni through the redeposited layer. The model assuming the diffusion of Au through the solder matrix as the rate controlling step agreed well with most of the experimental results.

2:30 PM
Effects upon Interfacial Reactions by Electric Currents of Alternating Directions: *Sinn-Wen Chen*[1]; *Mei-Yau Du*[1]; *Chih-Ming Chen*[1]; [1]National Tsing-Hua University, Dept. of Cheml. Eng., #101 Kuang-Fu Rd., Sec. 2, Hsin-Chu 300 Taiwan

Electromigration refers to the phenomenon that the passage of electric currents induces the movement of metallic atoms in metals. The electromigration effects caused by the passage through of a 500A/cm^2 electric current of alternating directions upon the interfacial reactions in the Sn-0.7wt%Cu/Ni couples were examined. The results were compared with the interfacial reactions in the same-type of couples reacted under various experimental conditions, such as without passage through of electric currents and with passage through of a 500A/cm^2 electric current of a fixed direction. The thickness of the reaction layers varied with experimental conditions. Ni_3Sn_4 phase was found in all these couples; however, Cu_6Sn_5 phase was only found in the couples with passage through of an electric current of a fixed direction. It is concluded that the electromigration effect changed the atomic fluxes but not nucleation behavior in the Sn-0.7wt%Cu/Ni couples, based on the different phenomena observed in these couples.

2:55 PM
Solderability Testing of 95.5Sn-3.9Ag-0.6Cu Solder on OFHC Copper and Au-Ni Plated Kovar: *Edwin Paul Lopez*[1]; Paul T. Vianco[1]; Jerome A. Rejent[1]; [1]Sandia National Laboratories, 1832, MS-0888, PO Box 5800, Albuquerque, NM 87185 USA

The solderability of 95.5Sn-3.9Ag-0.6Cu solder on OFHC copper and Au-Ni plated Kovar was examined as a function of solder flux and temperature. Three solder fluxes including a rosin (R), rosin mildly activated (RMA) and a low solids flux were studied for three different test temperatures. The meniscometer test method was used to measure the meniscus rise of a solder up the side of a coupon test sample. The wetting balance test determined the weight of solder within the meniscus. Together, the meniscus height and weight were used to calculate the contact angle, which served as the solderability metric. The wetting rate and wetting time parameters were also documented. These test results were used to compare the solderability performance of the Sn-Ag-Cu lead-free solder against that of the more traditional Sn-Pb alloy as a means of establishing best practices in the development of commercial soldering processes. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Dept. of Energy under Contract DE-AC04-94AL85000.

3:20 PM
The Reaction Between SnAgCu Lead-Free Solders and Ni: C. E. Ho[1]; W. T. Chen[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Cheml. & Matls. Eng., Chungli City 32054 Taiwan

The reactions between the SnAgCu lead-free solders of various concentrations and the Ni substrate were studied. The SnAgCu solder family are the most promising lead-free solders to replace the PbSn eutectic solder, while Ni is the key element used in many different lead-free surface finishes, such as the Au/Ni and Pd/Ni surface finishes. In this study, several different SnAgCu compositions were used. Reaction between Ni and SnAgCu in the molten state as well as in the solid state were examined. In this talk, we will show that the SnAgCu composition has a profound effect on the reaction.

3:45 PM Break

4:05 PM Invited
Intermetallic Growth in Pb Free Solder/Metallization Diffusion Couples: *Eric J. Cotts*[1]; Richard R. Chromik[1]; Lubov Zavalij[1]; Anis Zribi[1]; [1]SUNY Binghamton, Dept. of Phys. & Astron., PO Box 6016, Binghamton, NY 13902 USA

The evolution of solder alloys at Pb free solder/Cu and Pb free solder/Ni interfaces is examined. We study the formation of binary and ternary phases at the solder/metal interface. Phase selection is compared to the well known behavior of Pb-Sn solder/metal systems. For example, at $Sn_{94.35}Ag_{3.8}Cu_{1.85}Ni$ interfaces, Cu was observed to combine with Ni and Sn to form a ternary phase, $(Cu,Ni)_6Sn_5$, instead of the Ni_3Sn_4 phase observed in PbSn/Ni diffusion couples upon reflow. The effect of the formation of solder alloys upon the composition and microstructure of the solder itself is considered. Examination is made of the effect of the variation in reflow parameters and heat treatment times and temperatures on the evolution of the solder joint microstructure.

4:35 PM

The Interface Phases of Sn-Pb Solder and 42 Ni-Fe Alloy: *Shih-Chin Chang*[1]; Wei-Xuan Yu[1]; Hsi-Sen Chang[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., Hsinchu 30043 Taiwan

The morphology and the development of the intermetallic phases at the Sn-Pb/Alloy 42 interface with different Pb content were studied. With a technique of taper grinding, re-mounting and pull opening, the interface intermetallic compound could be observed. An interface layer in a thickness of 3 µm was observed at the interface between the solder and the Alloy 42. On the alloy 42 is a continuous layer of FeSn2. On the continuous FeSn2 layer many very long, thin and narrow particles were observed to densely scattered in just one single layer. The dimensions of these particles are about 100 x 5 x 1 µm and they have the appearance of the sweet-flag-leaves (SFL). The sweet-flag-leaf (SFL) phase has been identified by X-ray diffractometer as the Ni3Sn4. Because the thickness of them is smaller than one µm and they lie on the interface in just one single layer, though the SFL particles are very manifest on the opened interface, they are generally not visible from the cross section (side view) and little observation of them has been reported before. A mechanism is proposed to explain the formation and growth of the SFL phases. It explains the shape of the SFL phases and why they form only in a single layer.

5:00 PM

Phase Transformation Mechanisms in Cu-Ag-Sn/Metallization Electronic Joints: *Anis Zribi*[1]; Eric J. Cotts[1]; [1]SUNY Binghamton, Dept. of Phys. & Astron., PO Box 6016, Binghamton, NY 13902 USA

We investigated the growth of intermetallic compounds in $Cu_{1.85}Ag_{3.8}Sn_{94.35}$/Cu and $Cu_{1.85}Ag_{3.8}Sn_{94.35}$/Au/Ni solder joints. Upon reflow of the solder, Cu_6Sn_5 formed at the interface of solder/Cu joints while $(Cu,Ni)_6Sn_5$ grew at the interface of solder/Au/Ni joints. During thermal annealing at 150°C, Cu_6Sn_5 continues thickening, while $(Cu,Ni)_6Sn_5$ transforms gradually to another ternary compound $(Ni,Cu)_3Sn_4$ after 16 h of annealing. We monitored the growth rate of interfacial alloys and the evolution of the morphology and distribution of bulk and interfacial phases with thermal aging. Consideration of interdiffusion arguments and the stoichiometry of the different compounds brought more insight into the possible formation mechanisms of these alloys. The fast diffusion of Cu in Sn is held responsible for the preferential growth of Cu-rich intermetallics upon reflow, while the depletion of Cu from the solder is suspected to be the reason behind the transformation of $(Cu,Ni)_6Sn_5$ to $(Ni,Cu)_3Sn_4$.

Materials Design Approaches and Experiences - V

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: Ji-Cheng Zhao, GE Corporate Research & Development, Physical Metallurgy Laboratory, Schenectady, NY 12301 USA; Michael G. Fahrmann, Special Metals Corporation, Technology Department, Huntington, WV 25705-1771 USA; Tresa M. Pollock, University of Michigan, Materials Science and Engineering Department, Ann Arbor, MI 48109-2136 USA

Wednesday PM Room: 204
November 7, 2001 Location: Indiana Convention Center

Session Chair: Hiroshi Harada, National Research Institute of Metals, Tsukuba, Ibaraki 305-0047 Japan

2:00 PM Invited

Simulation of Diffusional Transformations and Phase Precipitation in Materials: *John A.L. Agren*[1]; [1]KTH, Matls. Sci. & Eng., Stockholm SE-100 44 Sweden

The theoretical analysis of diffusional phase transformations and phase precipitation has an old tradition in materials science. Approaches based on the solution of a diffusion problem with thermodynamically based boundary conditions, e.g. local equilibrium or paraequilibrium, are very powerful tools because they make only use of bulk properties of the phases involved. The tremendous development in computational thermodynamics over the last decade makes it now feasible to consider quite complex multicomponent materials, e.g. high-alloy steels, cemented carbides, coatings, etc. Experiences of sharp-inteface approaches, e.g. DICTRA, and how they can be modified to take into account also deviation from local equilibrium by surface tension effects, solute drag and finite interface mobility, as well as some recent applications will be discussed. Diffuse interface approaches, e.g. phase field, and some recent applications will be discussed. Recent experiences based on stochastic differential equations will be be discussed and compared with traditional approaches.

2:30 PM Invited

Phase Field Microelasticity Model of Microstructure Dynamics in Phase Transformations and Plastic Deformation: *Armen G. Khachaturyan*[1]; Yongmei M. Jin[1]; Yu U. Wang[1]; [1]Rutgers, The State University of New Jersey, Dept. of Cer. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854-8065 USA

Structural inhomogeneities caused by phase transformations or plastic deformation generate long-range strain field resulting in interaction dependencies on microstructure. This interaction plays a dominant role in the microstructure evolution. The Phase Field Microelasticity theory and its application to microstructure evolution in decomposition of intermetallics and to the dislocation dynamics are discussed. Examples of computer simulations of the microstructure and dislocation dynamics are presented.

3:00 PM Invited

Development of Rene 88DT for Aircraft Engine Disk Applications: *Dan D. Krueger*[1]; [1]General Electric Aircraft Engines, MD H85, 1 Neumann Way, Cincinnati, OH 45215 USA

Rene 88DT is a powder metallurgy nickel-base superalloy that was developed by General Electric Aircraft Engines during the mid-to-late 1980's for use in aircraft gas turbine engine disk applications. The alloy provides a significant improvement in the balance of tensile, creep and fatigue damage tolerance compared to other high performance disk alloys, such as Rene 95. This paper describes the work done to design and evaluate experimental alloy compositions, and select a preferred alloy through subsequent scale-up and full scale processing trials. The technology to overcome challenges in grain size control, heat treatment, and achieving a successful balance between property levels and producibility is also discussed.

3:30 PM

Growth Kinetics of Gamma Prime Precipitates in P/M Superalloys: *Jian Mao*[1]; Keh-Minn Chang[1]; David Ulrich Furrer[2]; [1]West Virginia University, Dept. of Mechl. & Aeros. Eng., PO Box 6106, Morgantown, WV 26505 USA; [2]Ladish Co., Inc., R&D, 5481 S. Packard Ave., Cudahy, WI 53110 USA

In order to better understand the precipitation of cooling g' and its growth as a function of temperature during a quench process, interrupted cooling tests were conducted in both Rene88DT and U720LI alloys. The tests are comprised of several continuous cooling tests, each of which was interrupted at a different intermediate temperature. The results show that the cooling precipitates grew continuously as the temperature was decreased to 650°C. Empirical models were derived to describe the growth of the g' precipitates against the interrupt temperature at a fixed cooling rate. The size of the g' precipitates in Rene88DT alloy is found much smaller than that in U720LI alloy. The strength responses to the interrupt temperature were also studied in both alloys. It indicated that as the g' grows with the decrease of the interrupt temperature, the strength of the alloys decreases. This decrease is further identified to be non-monotonic. A multi-stage burst (nucleation) of g' precipitates is believed to be accountable for the strength behavior.

3:50 PM Break

4:10 PM Invited

A Review of U720LI Alloy and Process Development: *David Ulrich Furrer*[1]; [1]Ladish Company, Inc., PO Box 8902, Cudahy, WI 53110-8902 USA

Udimet 720LI is a superalloy of considerable interest to many turbine engine manufacturers for its mechanical property capability and relative open market accessibility, due to the fact that a material supplier and not any one OEM originally developed it. A review of the literature has uncovered considerable development work aimed at optimizing and evolving this material for new or higher performance applications. A large number of researchers have contributed to the understanding of U720 and U720LI. Small focussed efforts have given rise to evolutionary changes in the chemistry, processing and application of U720LI, as a result of over twenty plus years of research and development.

4:40 PM Invited

The Development of HAYNES® 230® Alloy: *Dwaine L. Klarstrom*[1]; [1]Haynes International, Inc., Eng. & Tech., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

HAYNES® 230® alloy is a material that is currently used in many high temperature applications such as combustion cans, transition ducts, flameholders, honey-comb seals, and thermocouple sheaths in gas turbine engines, and for a variety of other components in the chemical processing, industrial heating and heat treating industries. The alloy was designed to offer the best combination of oxidation resistance, high temperature creep-rupture strength, fatigue resistance and thermal stability in a nickel-based alloy. The design rationale for each of these properties is presented and discussed along with supporting test results.

Mechanical Behavior & Constitutive Modeling of Polymers, Fiber- & Particulate-Reinforced Polymer Composites & Polymer-Based Nanocomposites - II: Polymer Nanocomposites

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials, SPE Polymer Analysis Division, Jt. Composite Materials Committee
Program Organizers: George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Linda Schadler, Rensselaer Polytechnic Institute, Materials Science & Engineering Department, Troy, NY 12180-3590 USA; Carl Schultheisz, National Institute of Standards and Technology, Polymers Division, Gaithersburg, MD 20899 USA

Wednesday PM Room: 201
November 7, 2001 Location: Indiana Convention Center

Session Chairs: Carl Schultheisz, National Institute of Standards and Technology, Polymers Div., Gaithersburg, MD 20899 USA; Jon Kellar, South Dakota School of Mines and Technology, Dept. of Matls. & Metlgcl. Eng., Rapid City, SD 57701-3995 USA

2:00 PM
Time Dependent Strain/Stress Redistribution in Continuous Graphite Fiber/Epoxy Model Composites with Fiber Breaks, Grad Student Poster Contest: *C. H. Zhou*[1]; Linda S. Schadler[1]; I. J. Beyerlein[2]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Eng., 15th St., Troy, NY 12180 USA; [2]Los Alamos National Laboratory, Los Alamos, MS G755, NM 87545 USA

Time dependent micromechanics in graphite fiber/epoxy composites at room temperature was investigated with micro Raman spectroscopy (MRS) and modeled by a viscous break interaction (VBI) technique based on the shear-lag concept. Creep tests were performed on the unfilled epoxy matrix and the data was fit to a power law function, which provided the modeling parameters used as input for the composite model. The model predictions showed good agreement with the experimental strain/stress profiles obtained with MRS. It was found that the local matrix shear creep around fiber breaks not only led to a growing load recovery length along the broken fiber, but also broadened the over-loaded length in the neighboring intact fibers. This increased the probability of additional fiber breaks with time. When the distance between two breaks along different fibers was close enough, strong interactions resulted in high local shear stress and facilitated the interfacial debonding between the breaks in the composites.

2:20 PM
The High Strain-Rate Response of a Plasticized Elastomer to Plate Impact: *Jerry J. Dick*[1]; James N. Johnson[1]; A. Richard Martinez[1]; [1]Los Alamos National Laboratory, DX-1, MS P952, Los Alamos, NM 87545 USA

Plasticized Estane used as an explosive binder was subjected to plate impact using a high-velocity impact facility. The response in compression and tension were measured and compared to that of pure Estane. A Maxwell model is used to describe the compressive behavior (Ref.1). For tensile loading the arrangement of impactor and sample was such that rarefactions from the rear surface of the impactor and the free surface of the sample collide in the interior of the sample. This sends the material into tension at that location. The motion of the binder free surface is monitored. It indicates that the tensile strength and failure mode of the binder depend on the amount of precompression of the sample. The damage evolves in time, but spall separation is incomplete. 1. J. N. Johnson, J. J. Dick, and R. S. Hixson, “Transient response of three polymers,” J. Appl. Phys. 84, 2520 (1998).

2:40 PM
Effect of Plasticizer Concentration, Temperature and Strain Rate on the Mechanical Properties of HMX Based Plastic Bonded Explosives: *Darla Graff Thompson*[1]; Deanne J. Idar[1]; George T. Gray[2]; William R. Blumenthal[2]; Carl D. Cady[2]; Edward L. Roemer[1]; Walter J. Wright[1]; [1]Los Alamos National Laboratory, DX-2, MS C920, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

Compressive and tensile measurements were obtained on three different formulations of plastic-bonded explosives (PBX) which contained varying weight percentages of HMX and a BDNPF-A nitroplasticizer (NP) in the polymeric binder. The baseline material, PBX 9501, contains 2.5% NP by weight, 2.5% (wt/wt) Estane 5703, a polyester-polyurethane, and 0.1 % (wt/wt) Irganox 1010; the remaining 94.9% (wt/wt) is the explosive, HMX. Two other PBX materials were formulated, containing 0.95% and 0.69% (wt/wt) NP, respectively; the NP weight decreases were countered by increased weight percents of HMX (96.45% and 96.71%, respectively). These materials were tested with dynamic and quasi-static compression as a function of temperature and strain rate. Quasi-static tension tests were also performed as a function of temperature and strain rate. Results show significant differences due to differences in NP content, as well as demonstrating temperature and strain rate dependent behavior.

3:00 PM
Characterization of Polymeric Composites with Low CTE Ceramic Particulate Fillers: P. U. Sonje[1]; *K. N. Subramanian*[1]; A. Lee[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824-1226 USA

Use of particulate fillers is a potential approach to develop isotropic low CTE polymeric composite materials for electronic circuit board applications. Low CTE inorganic fillers such as cordierite, beta quartz, alkaline and alkaline earth zirconium phosphates were incorporated into EPON 8280 and Epicure 3223 system. Although such composites did show a decrease in CTE, the overall effect was not clear. In order to understand the behavior of various fillers in this epoxy system and to evaluate the nature of interfaces, fillers were coated with surface active agents like silane coupling agents. Fracture behavior and mechanical properties of these composites are studied to understand the nature of interfacial bonding and its effect on CTE reduction.

3:20 PM Break

3:40 PM
Experimental Studies on Three Point Bending of Syntactic Foam Core Sandwich Structured Composites: *Nikhil Gupta*[1]; Eyassu Woldesenbet[1]; [1]Louisiana State University, Mechl. Eng. Dept., 2508, CEBA Bldg., Baton Rouge, LA 70803 USA

Sandwich structures consisting of light weight, relatively weak core and thin but stiff skins display superior flexural properties. Use of syntactic foams as core materials gives several distinct advantages over traditionally used core materials, especially in aerospace applications due to their superior compressive properties, low density, low radar detectability and low moisture absorption features among others. Owing to the difference in structure of syntactic foams vis-à-vis the honeycomb and other such largely used core materials, their response is considerably different during mechanical testing. The present work aims at studying the behavior of sandwich structures containing syntactic foam as core under three point bending conditions. The study is carried out with specimens having both large and short span-length/thickness ratios giving rise to the conditions where true bending and shear stresses prevail respectively. Scanning electron microscopy of specimens is done to examine the post-test fracture features of the material.

4:00 PM
Effect of Jute Fiber Reinforcement on the Tribological Properties of Phenolics Based Composites: *A. S.M. A. Haseeb*[1]; Abdul Gafur[1]; Mosharrat Hasan[1]; [1]Bangladesh University of Engineering and Technology (BUET), Dept. of Matls. & Metall. Eng., Dhaka 1000 Bangladesh

Although glass and other synthetic fibres are commonly used for the reinforcement of polymers, natural fibres like jute can offer certain advantages in that they are environmentally friendly, easily available and cheap. Replacement of synthetic fibres by the natural ones in polymer matrix composites has therefore been of great interest in recent years. The present work aims at investigating the tribological properties of phenolics based composites reinforced by jute fibre. Composites with randomly oriented short jute fibres, with and without fibre pre-treatment, have been prepared by hot pressing. The wear behaviour of the composites has been studied under various conditions of load, speed and sliding distance in a horizontal pin-on-disc type wear apparatus. Cylindrical composite pins were pressed against grey cast iron counter body using a constant normal load. The wear rates of the composite samples were calculated from the weight loss measurements. Worn surfaces were examined under an optical and a scanning electron microscope. Considerable improvement in the wear resistance has been observed as a result of reinforcement by jute fibre. It has been found that the wear resistance of the composite increases with the increase of jute content. Attempt has been made to understand the wear mechanism of the natural fibre reinforced composites.

4:20 PM

Composite Sliding Bearing Based on Metal-Polymer–Soft Metal-Ceramics Composition and New Manufacturing Technology: *Maxim Kireytsev*[1]; A. Federavichus[1]; [1]National Academy of Sciences of Belarus, Lab. of Tribology & Mech., Inst. of Mach. Reliability, Minsk, Belarus

Experimental evidence of the susceptibility of composites based on anodic oxide ceramics to mechanical and wear degradation under cyclic and static loading has been well documented in a number of studies. This has serious implications for the practical use of these materials in mechanically loaded components like hard loaded bearings and friction units. This paper addresses several topics concerning fatigue of composite coatings based on anodic oxide ceramics and chrome carbides. To improve operating properties and decrease disadvantages our team design and tests new composite sliding bearing and manufacturing technology. The bearing sequentially arranged main layers are steel base, polymer layer, aluminum or its alloy layer and oxide ceramic layer.

Microstructure Modeling and Prediction During Thermomechanical Processing - V

Sponsored by: Structural Materials Division, Materials Processing & Manufacturing Division, Shaping and Forming Committee, Titanium Committee

Program Organizers: Raghavan Srinivasan, Wright State University, Department of Mechanical & Materials Engineering, Dayton, OH 45435 USA; Armand J. Beaudoin, University of Illinois at Urbana–Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Steve P. Fox, Timet Corporation, Timet Henderson Technical Laboratory, Henderson, NV 89009 USA; Zhe Jin, Reynolds Metals Company, Metallurgy Department, Chester, VA 23836-3122 USA; S. L. Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Wednesday PM — Room: 208
November 7, 2001 — Location: Indiana Convention Center

Session Chairs: Henry Rack, Clemson University, Sch. of Cheml. & Matls. Sci., Clemson, SC 29634-0921 USA; Hamid Garmestani, Florida State University, Mechl. Eng., Tallahassee, FL 32306 USA

2:00 PM

Kinetics in Stress Relaxation of AA 5182: *Armand J. Beaudoin*[1]; Lihua Zhu[1]; Stuart MacEwen[2]; [1]University of Illinois, 1206 W. Green St., MC-244, Urbana, IL 61801 USA; [2]Alcan International, Ltd., Kingston R&D Ctr., PO Box 8400, Kingston K7L4L9, Ontario, Canada

An examination of kinetics is made for stress relation of AA 5182 sheet. A state of bending, initially elastic, is maintained experimentally for a period of monts. Plastic deformation develops with strain rates on the order of 10^8. Finite simulations are applied to interpretation of experimental data, necessary because of the inherent non-uniformity in the bending test. This leads to development of a kinetic law. Effects of residual stress in the initial stock material are examined. Implications for the application of solution strengthened aluminum alloys are presented.

2:25 PM

Effect of Processing Variables on Nucleation and Growth during Recrystallization of Aluminum: *Malesela Jones Papo*[1]; Burton R. Patterson[1]; Hasso Weiland[2]; [1]University of Alabama at Birmingham, Matls. & Mechl. Eng., Rm. 254, BEC, Birmingham, AL 35294 USA; [2]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069 USA

Stereological methods have been used to study the effects of processing variables on the rates of nucleation and growth during recrystallization of 5052 aluminum. Variables examined included preheat temperature, hot vs. cold rolling and chromium level. Growth rate was determined by the Cahn-Hagel method and nucleation rate was computed directly from the time rate of increase in number of new grains per unit volume of unrecrystallized material. Growth rates were found to either remain constant or decrease with time. Nucleation rates were found to increase with time for cold rolled material and decrease with time for hot rolled material. Contiguity measurements typically showed clustering of the recrystallizing grains. It was found that the effects of preheat temperature and Cr level on recrystallization kinetics were primarily due to solid solution rather than dispersoid effects.

2:50 PM

Role of Segregation and Precipitates on Interfacial Strengthening Mechanisms in SiC Reinforced Aluminium Alloy when Subjected to Thermomechanical Processing: *Syed T. Hasan*[1]; John H. Beynon[2]; Roy G. Faulkner[3]; [1]Sheffield Hallam University, Sch. of Eng., City Campus, Howard St., Sheffield, S. Yorkshire S1 1WB UK; [2]University of Sheffield, Dept. of Mechl. Eng., Mappin St., Sheffield S1 3JD UK; [3]Loughborough University of Technology, IPTME, Loughborough LE11 3TU UK

The satisfactory performance of metal matrix composites depends critically on their integrity, the heart of which is the quality of the matrix-reinforcement interface. The nature of the interface depends in turn on the processing of the metal matrix composite component. At the micro-level the development of local gradients around the reinforcement, as the metal matrix attempts to deform during processing, can be very different to the nominal conditions and play a crucial role in important microstructural events such as segregation and precipitation at the matrix-reinforcement interface. These events dominate the cohesive strength and subsequent mechanical properties of the interface. The compositional variations at the matrix-reinforcement interface of a metal matrix composite are reported, which highlights the interfacial strengthening mechanisms in metal matrix composites during thermomechanical processing. A method of calculation has been applied to predict the interfacial fracture strength of aluminium and SiC interface, in the presence of magnesium segregation.

3:15 PM

Applications of Powder Sintering to Selective Laser Sintering: Martin Wohlert[1]; Nicole Harlan[1]; *David L. Bourell*[1]; [1]University of Texas at Austin, TX Matls. Inst., MC C2200, Austin, TX 78712 USA

Selective Laser Sintering (SLS) is a Solid Freeform Fabrication process in which a part is built quickly from powders without the use of part-specific tooling. Production of metallic, ceramic and composite parts often requires some form of pre-processing or post-process sintering to achieve full density. Powder Densification Maps represent a potent tool for optimizing the pre- and post-processing parameters. Such maps are computational representations of part density as affected by time, temperature, pressure and materials properties. Critical to the formulation of densification maps is an understanding of time-dependent plasticity. This presentation will summarize SLS developments at The University of Texas at Austin with emphasis on the utility of powder densification mapping of powder pre-processing and part post-processing of Ti-6Al-4V, Alloy 625, zirconia and copper.

3:40 PM Break

3:55 PM

Atomic Structural Models for Amorphous Metals: *Dan Miracle*[1]; Oleg Senkov[2]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., AFRL/MLLMD, Wright-Patterson AFB, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH, USA

Amorphous metal alloys based on Al, Ti, Mg and other elements hold significant promise as structural materials for aerospace systems. These materials not only offer unusual functional properties in the fully amorphous condition, but also provide a path to unique microstructures (nanocrystalline, quasicrystalline and partially crystalline) with exceptional properties. While rapid solidification (>104°C/sec) is a typical requirement for the production of a fully amorphous product, a very small number of systems have been identified which can be produced in the fully amorphous condition at cooling rates as low as 1/100°C/sec. At present, there is no quantitative and structurally specific model which can explain the dramatic difference in glass forming ability of these bulk glass formers and the vast majority of alloys which require very high cooling rates. This presentation will discuss recent efforts to identify and quantify the structural features of amorphous metallic alloys at the atomic level.

4:20 PM

Fine-Grained Sheet Produced by Twin-Roll Casting of Aluminum Eutectic Alloys: *Tracey Jayne Cockfield*[1]; John D. Hunt[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxfordshire OX1 3PH UK

Twin-roll casting is an established industrial continuous casting process for the production of aluminium sheet. Problems in controlling the downstream grain size of conventionally processed sheet using commercial DC casting alloys are encountered. This may result in poor ductility and formability. Current work explores a method of processing eutectic-based alloys to produce fine-grained sheet. An equiaxed grain structure of 15 microns or less has been produced using Al-Ni alloys of hypo- and fully eutectic compositions. Heat treatments carried out prior to cold rolling are employed to spheroidise and coarsen the eutectic, which can then act as recrystallisation nuclei during a final anneal. The role of particle characteristics in recrystallisation is discussed, including the effects of size, distribution through the strip and coarsening kinetics. The method is being investigated in other alloy systems and shows promise for design of twin-roll casting alloys.

4:45 PM

Weld Visualization for Investigating Solidification: *Aaron C. Hall*[1]; Charles V. Robino[1]; John Brooks[2]; [1]Sandia National Laboratories, 1835, PO Box 5800, MS 889, Albuquerque, NM 87185 USA; [2]Sandia National Laboratories, 8724, PO Box 969, MS 9402, Livermore, CA 94551-0969 USA

Using a high quality zoom lens and a -CCD camera the solid-liquid interface in a stainless steel gas tungsten arc (GTA) weld has been filmed at high magnification. At low magnification, the dendritic structure of the solid-liquid interface can be clearly seen, as can characteristic ripples caused by pool instabilities. At higher magnification, secondary dendrite arms can be seen sticking through the liquid surface of the weld pool. Using a computer-generated autocorrelation the average fractal spacings (primary and secondary dendrite arm spacings) recorded in the video frame can be measured. High-speed video-microscopy allows large amounts of quantitative information about the solid-liquid interface to be accessed under actual welding conditions. This technique has tremendous potential for fundamental solidification science, computer model verification, weld process development, and real time process control. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

Modeling the Performance of Engineering Structural Materials - V: Friction and Tribology

Sponsored by: Structural Materials Division, Structural Materials Committee
Program Organizers: Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA

Wednesday PM — Room: 140
November 7, 2001 — Location: Indiana Convention Center

Session Chairs: T. J. Langdon, University of Southern California, Dept. of Mechl. Eng., Los Angeles, CA 90089-1453 USA; T. S. Srivatsan, University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

2:00 PM
The Effects of Phosphate Treatments on the Surface Friction of Galvannealed Sheet Steels: *D. J. Paik*[1]; G. M. Michal[1]; [1]Case Western Reserve University, The Case Sch. of Eng., Dept. of Matls. Sci. & Eng., Cleveland, OH 44106-7204 USA

Application of a phosphate coating to the surface of a metal prior to a forming operation has been known to reduce surface friction. Recently phosphate compounds have been applied to the surface of galvanneal coatings on highly formable steel sheet substrates. The resulting changes in surface friction have been evaluated through a series of cup drawing tests. Forming loads as a function of punch displacement were continuously monitored for cups drawn with punch velocities up to 300 mm/sec using a newly designed high speed hydraulic press. A model for the cup drawing process was formulated and used to extract the effective values for the coefficient of surface friction from the load/displacement data. Mechanisms by which phosphate can reduce surface friction will be discussed.

2:25 PM
Friction and Wear Properties of CVD-Coated Diamond: *Ali Soleman Alwatban*[1]; [1]Riyadh Technology College, Mechl. Tech. Dept., PO Box 53699, Riyadh 11593 Saudi Arabia

In this paper, the room temperature frictional properties of a CVD coated diamond were studied when softer metallic sliders were used. The experiments were conducted at relatively low sliding speeds (7mm/min), in a vacuum of 0.0001 mbr after having cleaned the specimen surfaces by outgassing them for about 30 min at 800°C. Single and multi-traversals were employed for track distance of 5mm in order to investigate the effect of the traversal number on the coefficient of friction. As a result of increasing the number of traversals an exceptional phenomenon where observed when a significant wear of the coated diamond caused by the mild steel sliders.

2:50 PM
Composite Coatings Based on Anodic Hard Oxide Ceramics and Chrome Carbide: *V. Basenuk*[1]; M. Kireytsev[1]; [1]National Academy of Sciences of Belarus, Lab. of Tribology & Mech., Inst. of Mach. Reliability, Minsk, Belarus

Experimental evidence of the susceptibility of composites based on anodic oxide ceramics to mechanical and wear degradation under cyclic and static loading has been well documented in a number of studies. This has serious implications for the practical use of these materials in mechanically loaded components like hard loaded bearings and friction units. This paper addresses several topics concerning fatigue of composite coatings based on anodic oxide ceramics and chrome carbides. In this paper some of the above observations have been confirmed by in situ experiments performed in a scanning electron microscope, so as to exclude the possibility that they are environmentally controlled. More importantly, micromechanical models using arrays of internal or surface cracks have been developed which provide plausible answers to the questions arising from the above three observations. The models also reveal the role of overloading in crack arrest, which may well be exploited in the safe design of transformation-toughened ceramics against fatigue.

3:15 PM Break

3:35 PM
Tribological Parameters of Thermal Flame Sprayed Coatings: *M. Belotzerkovski*[1]; V. Basenuk[1]; M. Kireytsev[1]; [1]National Academy of Sciences of Belarus, Lab. of Tribology & Mech., Inst. of Mach. Reliability, Minsk, Belarus

A number of researches indicate a great importance of thermal sprayed coatings for wear resistance. However some valuable properties such as self-lubrication ability of thermal sprayed coatings not often recognized. The results of tribomechanic properties (friction coefficient, wear rates) of bronze-based thermal flame sprayed coatings are shown. The abilities to self-lubricate of these coatings are studied. The principle of the tribometer is a "pin-on-disk" system. The coefficient of friction µ between the two materials tested can be calculated with the classical relation of Coulomb: µ=Ft/Fn, where Ft is the tangential force, Ft is the normal force. Also µ can be calculated with the following relation: µ=2Mfr/ND, where Mfr is the friction momentum, N is the normal force, D is the disk diameter. In this work, five different materials for thermal flame coatings used to make coatings on the disk samples are investigated.

4:00 PM
Study of Load Rating of the CrC-Al2O3-Al Composite Coating: *V. Basenuk*[1]; M. Kireytsev[1]; [1]National Academy of Sciences of Belarus, Lab. of Tribology & Mech., Inst. of Mach. Reliability, Minsk, Belarus

Oxide ceramics hard coatings on all types of aluminum and its alloys formed by the recently developed method of the electrochemical micro-arc oxidizing are well known and recognized over the world. These coatings have a number of the improved properties such as no hazardous wastes, ecologically sound, high microhardness and wear resistance at lubricated contact and others. It attracts the increasing commercial concern of many companies and customers. To extend an area of application of these coatings the investigation of its mechanical properties is necessary to realize. Microhardness, porosity, microstructure, adhesion and micro-structure from micro samples were studied and were used to investigate the load rating of the composite such as the Al-Al2O3 and the top-developed Al-Al2O3-CrC.

4:25 PM
Investigation of Tribological Properties of the CrC-Al2O3-Al: *M. Kireytsev*[1]; V. Basenuk[1]; [1]National Academy of Sciences of Belarus, Lab. of Tribology & Mech., Inst. of Mach. Reliability, Minsk, Belarus

In practical engineering it is well known, that anodic hard oxide coatings on all types of aluminum and its alloys formed by the method of micro arc oxidizing have high friction coefficient and wear rate at dry friction. Nowadays growth of new technologies and know-how allow to develop new coatings based on anodic hard oxide ceramics. As research reports shown new coatings improve process of friction significantly. It attracts high commercial interest of many companies and customers. In this work, composite coatings based on the anodic hard oxide aluminum formed on 2024 aluminum by micro arc oxidizing process are investigated. On the friction surface of the disk samples made from 2024 aluminum the oxide ceramic hard layer Al2O3 is formed with the micro arc oxidizing process. The layer has up to 10 % of pores opened on the inner surface of the sample. The diameter of the pores is up to 3,5 microns. The thickness of the anodic hard alumina is 50…300 microns. The carbide chromium layer was formed on the oxide aluminum layer by the pyrolysis method. The thickness of the CrC layer is 10…50 microns. The photo of the Al-Al2O3-CrC composite coating is shown at figure. The parameters of the anodic oxide layer are studied.

4:50 PM
Mechanical Properties of a High Carbon, High Silicon and High Manganese Steel: *Susil K. Putatunda*[1]; [1]Wayne State University, Dept. of Cheml. Eng. & Matls. Sci., Detroit, MI 48202 USA

A new high carbon (1%), high silicon (3.0%) and high manganese (2.0%) steel has been developed. This steel was synthesized using the concepts of Austempered Ductile Cast Iron (ADI) technology. The influence of austempering process on the microstructure and mechanical properties of this steel was examined. The influence of austempering process on the fracture toughness of this steel was also studied. Test results show that the austempering process has resulted in significant improvement in mechanical properties and fracture toughness of this steel over the conventional heat treatment process; i.e., quenching and tempering.

Powder Materials: Current Research & Industrial Practices: Structure/Properties/Processing Relationships

Sponsored by: Materials Processing & Manufacturing Division, Powder Materials Committee

Program Organizers: Fernand D.S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgical Engineering, Rapid City, SD 57701-3995 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Wednesday PM Room: 209
November 7, 2001 Location: Indiana Convention Center

Session Chairs: Karl P. Staudhammer, Los Alamos National Laboratory, Nucl. Matls. Tech. Div., Los Alamos, NM 87545 USA; Akaki B. Peikrishvili, Institute of Mining Mechanics, Tbilisi 380086 Georgia

2:00 PM
High-Strain-Rate Behavior of Microstructurally-Biased Two-Phase TiAL Ceramics: *Greg B. Kennedy*[1]; [1]Georgia Institute of Technology, Matls. Sci. & Eng., 771 Ferst Dr., Love Bldg., Atlanta, GA 30332-0245 USA

The high-strain-rate behavior of two-phase TiB2+Al2O3 ceramics with biased microstructures is investigated. Quantitative microscopy was performed to characterize the microstructural bias on the basis of phase size and curvature (used as a measure of TiB2 phase connectivity around Al2O3). Dynamic compression and tension (spall) properties were measured, using plate-impact experiments. The measurements used VISAR interferometry and piezoelectric PVDF stress gauges. Finite element simulations of fracture were carried out to describe the actual phase morphologies and arbitrary fracture patterns in the microstructures. The results of the quantitative microstructural characterization and high-strain-rate experiments are discussed.

2:25 PM
Consolidation of Metallic Powders by Equal Channel Angular Extrusion: *K. T. Hartwig*[1]; *S. N. Mathaudhu*[1]; [1]Texas A&M University, Dept. of Mechl. Eng., College Station, TX 77840 USA

The results of studies to determine the effectiveness of equal channel angular extrusion for consolidating aluminum 6061 and WC plus Co powders will be reported. Measurements of density, hardness, tensile strength and ductility were taken on specimens processed through 90° tooling. Above 98% theoretical density is obtained after one extrusion pass regardless of processing temperature for the 6061 aluminum and full consolidation is realized after only two passes. Tensile test results on as-extruded 6061 Al show that strength increases with the number of extrusions to levels above those of wrought material. ECAE is shown to be a viable method for consolidating powdered metallic alloys.

2:50 PM
Structural Investigations on Iron-Copper Alloys Sintered by Microwave and Conventional Heating: *Hogyu Kim*[1]; Dinesh K. Agrawal[2]; Paul R. Howell[3]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Eng., 226 Steidle Bldg., University Park, PA 16801 USA; [2]Pennsylvania State University, 207 Materials Research Lab., University Park, PA 16802-4801 USA; [3]Pennsylvania State University, Dept. of Matls. Sci. & Eng., 206 Steidle Bldg., University Park, PA 16801 USA

Microwave and conventional heating has been utilized to sinter Fe-C, Fe-Cu and Fe-Cu-C alloys to investigate and compare the microstructures of sintered samples, and to determine the effect of the microwave radiation on various phase transformations. The preliminary investigation of Fe-Cu alloy samples showed that samples sintered in the microwave furnace had less porosity and higher density as compared with samples sintered in a conventional furnace. However, the results of electron microprobe analysis showed no noticeable differences in the diffusion profiles of Cu in the conventionally and microwave sintered samples. A scanning electron microscope study on both microwave and conventionally sintered samples revealed that Cu precipitates in ferrite matrix were rod-like particles with terminating hemispherical caps, which are the equilibrium morphology of Cu precipitates in ferrite. To understand both the sintering behavior and phase transformations during annealing, the kinetics of sintering/diffusion and some aspects of transient liquid phase sintering are being examined in depth, and systematic investigations on both microwave and conventionally sintered samples are being conducted using various microscopic and analytical techniques: some results of the investigations will be presented.

3:15 PM
Variation in Mechanical Properties of P/M Parts: *Joseph W. Newkirk*[1]; [1]University of Missouri–Rolla, Metlgcl. Eng., 1870 Miner Cir., Rolla, MO 65409 USA

The final mechanical properties of P/M parts are strongly effected by the processing parameters. Processing controls porosity, composition and microstructure. Variations within the processing sequence lead to significant property variations. Variations in strength values are often accounted for by the publication of typical values and minimum values. Other mechanical properties important for critical applications, such as toughness, are even more sensitive to processing. This paper will discuss the use of Weibull statistics to analyze the properties of P/M parts and suggest new ways to determine property variablity for design application. Examples of different properties and materials will be used to illustrate the variation. The effect of processing parameters in controlling the variabilty will be discussed. Weibull parameters are suggested as being more suitable for designers than the current system.

3:40 PM Break

3:55 PM
Explosive Fabrication of Ni-C Composite Materials: Structure/Properties Relationships: *Akaki B. Peikrishvili*[1]; Laszlo J. Kecskes[2]; Fernand D.S. Marquis[3]; Levan A. Japaridze[1]; Nikoloz Chikhradze[1]; [1]Institute of Mining Mechanics, Tbilisi 380086 Georgia; [2]US Army Research Laboratory, Weapons & Matls. Rsrch. Direct., Aberdeen Proving Ground, Aberdeen, MD 21005 USA; [3]South Dakota School of Mines and Technology, Dept. of Matls. & Metlgcl. Eng., Col. of Matls. Sci. & Eng., Rapid City, SD 57701 USA

Hot explosive compaction technology was used in order to fabricate Ni-C composites with high content of C and up to near theoretical density. The clad graphite powders (by Ni) and hot explosive compaction technique was used. The compacting temperature was set at various values up to 1000°C, while the intensity of shock loading using cylindrical geometry was set under 10 GPa. This investigation shows that the use of clad powders from Ni graphite compositions enables us to consolidate them with higher value of density in contrast to mechanical mixtures at same intensity of loading. The simultaneous application of high temperatures and explosive consolidation of coated powders in hot conditions is an effective process to obtain cylindrivcal samples (rods and tubes) near theoretical densities with content of graphite up to 50%(vol). The structure and properties (density, hardness and strength) of the samples obtained could be controlled by the compacting temperature and the intensity of loading. The application high temperatures enables us to obtain high density long-body cylindrical samples with correct geometry and appropriate microstructure. This paper will discuss the effect of the parameters described above on the structure, properties and structure/properties relationships.

4:20 PM
Stress Tensor Calculations under Axisymmetric Dynamic Compression of Porous Materials: *Nikoloz Chikhradze*[1]; Ilia Lomidze[1]; Fernand D.S. Marquis[2]; Karl P. Staudhammer[3]; Levan A. Japaridze[1]; Akaki B. Peikrishvili[1]; [1]Institute of Mining Mechanics, Tbilisi 380086 Georgia; [2]South Dakota School of Mines and Technology, Dept. of Matls. & Metlgcl. Eng., Col. of Matls. Sci. & Eng., Rapid City, SD 57701 USA; [3]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

This work deals with calculations of the components of the stress tensor when the pressure impulse of cylindrical symmetry is being uniformly spread along the surface of the infinite cylindrical elastic body. The objective of these calculations is to asses with a sufficient approximation the stress-deformed state in samples during their axisymmetric low intensity shock wave loading. The necessity of such an assessment is grounded on the wide utilization and practical merits of axisymmetric schemes used for material's explosive treatment. The main assumptions made at the initial stage of the calculation are: elasticity and isotropy of the medium as well as the constancy of the sound speed and Lame elasticity constants. In addition we consider the medium as well as boundary conditions of cylindrical symmetry. Subsequently, the removal of some assumptions during the investigation process makes possible to take into account effects engendered by boundary conditions' asymmetry and changes of sound speeds and Lame constants. These changes are caused by irreversible thermal transformations going on in the medium. In these calculations we use well known methods for solving differential equations, such as the Fourier method, functions of Bessel, Neumann, Hankel, equation of Helmholtz, etc. At the axial symmetry, result of calculations are presented as a set of simple equations where the arguments are components of the stress tensor and the solution of this set, for the concrete case, gives all the components of the stress tensor.

4:45 PM
Synthesis of Al-Fe-Si Intermetallics by Mechanical Alloying: *C. Suryanarayana*[1]; E. Ivanov[2]; [1]University of Central Florida, Dept. of Mechl., Matls. & Aeros. Eng., Orlando, FL 32816-2450 USA; [2]TOSOH SMD, Inc., 3600 Ganz Rd., Grove City, OH 43123 USA

Mechanical alloying is a high energy powder metallurgy process involving repeated cold welding, fracturing, and rewelding of powder particles. This technique can synthesize a variety of alloy phases including amorphous alloys, quasicrystalline phases, and intermetallics. In this investigation, two intermetallics-Al8Fe2Si and Al4FeSi2-were prepared by mechanical alloying starting from blended elemental powder mixtures in a Fritsch Pulverisette mill. The milling speed and the grinding medium were changed as two important process variables, which helps in establishing the optimum milling conditions. Results of this investigation obtained by X-raydiffraction, DTA, and scanning electron microscopy will be presented.

Surface Composite Coatings (Session Dedicated to: Dr. Robert Reeber, ARO [Retired]) - I: Surface Composite Coatings - Characterization

Sponsored by: Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: John C. Bilello, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; Ronald Gibala, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA

Wednesday PM Room: 112
November 7, 2001 Location: Indiana Convention Center

Session Chair: John C. Bilello, University of Michigan, Ctr. for Nanomatls. Sci., Ann Arbor, MI 48109-2136 USA

2:00 PM Invited
Army Research Office Support of Materials Research: *John T. Prater*[1]; [1]Army Research Office, Matls. Scis. Div., PO Box 12211, Durham, NC 27709-2211 USA

The Army Research Office (ARO) celebrated its 50th anniversary in June 2001. For fifty years ARO has been a staunch supporter of basic research in this country. An overview of the current ARO mission and research activities will be presented. This will include a summary of the major research topics currently being supported by ARO, particularly within the Materials Sciences Division, and a forecast for future directions in funding. Finally, there will be a listing of upcoming program solicitations that are of potential interest to the materials community, and a brief discussion of the proposal selection process.

2:20 PM Invited
High-Resolution Synchrotron X-Ray Scattering Studies of the Nanostructure of Surface Composite Coatings: *John C. Bilello*[1]; Benjamin L. French[1]; Matthew J. Daniels[1]; [1]University of Michigan, Ctr. for Nanomatls. Scis., Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109-2136 USA

In recent years the scale of interest in designing, processing and using technical coatings has become dramatically smaller and what was once called "microstructure" is now more appropriately designated as "nanostructure". Building up these nanostructures via multilayer architectures allows one to create a variety of nanocomposite surface coatings with potentially enhanced properties. The complexity, and extremely small length scale of such nanocomposites, requires the development of new tools for precisely characterizing their morphology. The advent of high-energy/high-resolution synchrotron radiation facilities created a unique probe to address the problem. The application of synchrotron scattering experiments, namely, grazing incidence x-ray scattering (GIXS), and of real-time direct diffraction topography/radiography imaging, as tools for analyzing the nanostructure of surface composite coatings will be presented. Results will be discussed in terms of the potential for developing new classes of quasicrystalline, metal/amorphous, and amorphous/amorphous coatings. Support acknowledged under ARO under grant(s) DAAG 55-98-1-0382 (plus others), USAF under subcontracts from TAT, Annapolis, MD and DoE (for SSRL beamtime).

2:45 PM
Composition of Compound Films by Ion Scattering during 1mTorr Growth: *J. Albert Schultz*[1]; [1]Ionwerks, Inc., 2472 Bolsover 255, Houston, TX 77005 USA

Several new ion beam analysis techniques suitable for real time insitu composition monitoring at pressures up to several mTorr have emerged from our laboratory and collaborators over the last fifteen years. Pulsed Argon ion beams of 15 keV energy impinge a surface at grazing incidence. Time of flight (TOF) techniques are used to simultaneously measure the energy loss of Argon scattering from individual surface atoms as well as the energy and identity of surface atoms which are directly recoiled into a forward direction. Direct recoils can be used to identify all elements—including surface hydrogen and its isotopes. Directly recoiled ions can be collected in TOF mass spectrometers to achieve a well resolved mass spectrum of all surface elements and their isotopes. Examples will be given of surface stoichiometery control during deposition of compound superconductors, semiconductors and wide bandgap nitrides in the pressure ranges from ultrahigh vacuum up to 1 mTorr.

3:10 PM
In Situ Real-Time Studies of GaN Epitaxial Growth: *I. S.T. Tsong*[1]; [1]Arizona State University, Dept. of Phys., Tempe, AZ 85287-1504 USA

To understand the fundamental issues surrounding thin-film epitaxy, it is important to follow the nucleation and growth processes in situ and in real-time. The epitaxial growth of GaN films by molecular beam epitaxy (MBE) and metal-organic chemical vapor deposition (MOCVD) were monitored in situ and in real-time by low-energy electron microscopy (LEEM), scanning tunneling microscopy (STM) and multiple-beam optical stress sensor (MOSS). The coalescence of the GaN layer and the atomic structure of the surface reconstruction of the layer were observed by LEEM and STM, while the stress evolution in the layer was measured by MOSS for a complete characterization of the GaN surface during growth.

3:35 PM Break

3:55 PM
Relation between Intrinsic and Total Toughness: *Vijay Gupta*[1]; Xueming Wang[1]; [1]UCLA, Dept. Mechl. & Aeros. Eng., Los Angeles, CA 90095 USA

The relationship between intrinsic and total toughness is important for robust design of joints, and to understand size effects in bonding dissimilar material structures. Such a relationship is sought in our work through use of novel experimentation. Results for stainless steel/E-glass composite, and Al/epoxy bonds will be presented. The intrinsic toughness is measured by implanting well-characterized interfacial flaws and loading them using a laser-induced stress wave in the substrate. The wave loading of the crack is quantified using a dynamic fracture mechanics-based simulation to calculate the critical energy release rate values upon crack advance. The total toughness is measured using a double cantilever beam experiment, equipped with a cryogenic cell. The intrinsic interface toughness is varied through use of self-assembled monolayers on substrate surfaces and also by changing the substrate surface roughness.

4:20 PM
High Resolution Medium Energy Ion Beam Analysis of Ultra-Thin Oxides: *Robert A. Weller*[1]; Bridget R. Rogers[1]; [1]Vanderbilt University, Nashville, TN 37235 USA

The characterization of ultra-thin films, those thinner than approximately 20 nm such as state-of-the-art gate dielectrics in MOS transistors, is one of the most challenging problems of modern ion-beam analysis. In this presentation, we explore the strengths and limitations of time-of-flight medium energy backscattering as a tool for elemental depth profiling and describe the physical processes that determine the sensitivity of the technique and the depth resolution of the data. Some of the factors that degrade the resolution can be mitigated by proper spectrometer design, while others are fundamental and set the ultimate limits of the technique. For 100 keV He+ ions, data suggest that the ultimate resolution is approximately 1 keV, with the corresponding elemental depth resolution at the sample surface dependent upon scattering geometry. Recent results of analyses of thin, high-dielectric constant films produced for semiconductor applications by chemical vapor deposition will be presented. This work was supported in part by the US Army Research Office under grants, DAAL 03-92-G-0037, DAAH 04-94-G-0148, DAAH 04-95-1-0565, and DAAD 19-99-1-0283.

4:45 PM
Electron Scattering from Polymer-Based Interfaces and Nanoparticles: *Matthew R. Libera*[1]; [1]Stevens Institute of Technology, Hoboken, NJ 07030 USA

Traditional methods for studying polymer morphology in the transmission electron microscope (TEM) principally rely on differential heavy-element staining to induce amplitude contrast. This approach has enjoyed only incremental improvement over many decades of practice, and it is increasingly unsuited for the quantitative determination of materials morphology with the necessary spatial resolution demanded by a broad range of technologically forward-looking applications. This presentation describes two new electron-optical approaches–one based on transmission electron holography and the other based on spatially resolved electron energy-loss spectroscopy (eels)–to quantitatively determine local structure, composition, and, in some cases, 3-D topology of polymeric nanoparticles and interfaces. After introducing the basis for these new approaches, this talk describes measurements of structure at spatial resolutions of order 1-10 nm including: (i) measurement of the interphase in epoxy-matrix composites; (ii) quantitative determination of fast-secondary electron trajectories in polymeric thin films; and (iii) the nature of biologically-derived protein-based nanocontainers.

5:10 PM
Acoustic Resonances and Brillouin Scattering in Laminar Structures: *R. Sooryakumar*[1]; [1]The Ohio State University, Dept. of Phys., Columbus, OH 43210 USA

High-resolution Brillouin spectroscopy, an inelastic light scattering technique based on Fabry-Perot interferometry methods, has in recent years emerged as a powerful probe of the elastic properties of small volume samples such as thin films, coatings and laminar structures. Central to this success is the observation of acoustic excitations such as Rayleigh, Sezawa and Lamb waves as well as longitudinal, shear and standing wave resonances whose properties depend sensitively on the elastic properties of the film and the mechanical boundary conditions at the surface and interfaces. Experimental results highlighting the first observation of longitudinal guided resonance in hard coatings, evidence of acoustic barriers in GaN-AlN based hetero-structures and unusual standing wave resonances in supported films will be presented. These results are discussed in the framework of the elastodynamic Green's function that provides insight into fundamental properties of these excitations and the elastic properties of the supported structures.

Affordable Metal-Matrix Composites for High Performance Applications - IV: Fatigue, Fracture and Creep of MMCs - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: Awadh B. Pandey, Pratt & Whitney, Liquid Space Propulsion, West Palm Beach, FL 33410-9600 USA; Kevin L. Kendig, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Thomas J. Watson, Pratt & Whitney, East Hartford, CT 06108 USA

Thursday AM Room: 139
November 8, 2001 Location: Indiana Convention Center

Session Chair: Cliff J. Lissenden, Pennsylvania State University, University Park, PA 16802 USA

8:30 AM Discussion (Abstract Moved to Another Day)

9:00 AM
In-Situ Investigation of the Strain Distribution in AL-MMC Torsion Samples using High Energy Synchrotron Radiation: *René Valéry Martins*[1]; Ulrich Lienert[2]; Lawrence Margulies[3]; Anke Pyzalla[4]; [1]European Synchrotron Radiation Facility, 6, rue Jules Horowitz, BP 220, Grenoble, Cedex 09 F-38043 France; [2]APS, Argonne Natl. Lab., Adv. Photon Source, 9700 S. Cass Ave., Argonne, IL 60439 USA; [3]Riso National Laboratory, Roskilde DK-4000 Denmark; [4]Hahn-Meitner-Institut Berlin, Glienicker Str. 100, Berlin D-14109 Germany

Solid torsion samples, made of a two phase AlSi alloy, are largely plastically deformed to different shear strain maxima. The diameter of the samples is 5mm. A low speed deformation in uniform direction is carried out at room temperature and elevated temperatures. The strain during the deformation as well as the residual strain after the deformation are studied using high-energy synchrotron radiation. The diffraction measurements are carried out on the beamline ID11 at the ESRF. The utilisation of high-energy X-rays permits the nondestructive measurement of the local strain and texture within bulk materials. The local gauge volume is about 5x5x300 micrometer. The complete radial dependent strain tensors are measured for different phases and reflections. Large differences between the strain components of different phases and reflections are observed.

9:20 AM
Effect of Overloading on the Fatigue Crack Growth Behaviour of Cross-Ply Ti-6Al-4V/SCS-6 Laminate: *A. Yasmin*[1]; P. Bowen[1]; [1]University of Birmingham, Sch. of Metall. & Matls./IRC in Matls. for High Perfor. Applications, Edgbaston, Birmingham B15 2TT UK

The effect of single overload cycles between constant load range cycling on the fatigue crack growth resistance of single edge notched [90/0]2s laminate have been investigated. It is found that although specimens without overloading show crack arrest at initial stress intensity factor range (Kini) of 14 MPam and the corresponding Crack Arrest/Catastrophic Failure (CA/CF) transition is at Kini of 14-16 MPam, the specimens subject to periodic single overload cycles during constant load range cycling at Kini of 14 MPam or lower after every 104 or 105 cycles often show catastrophic failure. Furthermore, the higher the frequency of overloading cycles, the lower the fatigue crack growth resistance of the laminate. The number of fibre failures during overloading cycles has been determined by acoustic emission and it is found that either acceleration or retardation of fatigue crack growth rate after any overloading cycle depends on the number of intact bridging fibres in the crack wake of the laminate.

9:40 AM
Fracture and Fatigue Studies on Al-Be Composites: *J. Larose*[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, The Case School of Eng., Dept. of Matls. Sci. & Eng., Cleveland, OH 44106 USA

The fracture and fatigue behavior of Al-Be composites are being determined under a variety of test conditions. Fracture toughness is being determined on both notched and fatigue precracked specimens, while fatigue crack growth behavior is being measured at different R-ratios. The effects of test conditions (e.g. R-ratio, test temperature) on the fracture behavior will be summarized in addition to both optical and SEM examination of the fracture path and fracture surfaces.

10:00 AM Break

10:15 AM Invited
Plastic-Strain Fatigue Life and Cyclic Response of a SiC Particulate Reinforced Aluminum Composite: N. L. Han[1]; Z. G. Wang[2]; *J.-M. Yang*[1]; [1]University of California–Los Angeles, Dept. of Matls. Sci. & Eng., Los Angeles, CA 90095-1595 USA; [2]Chinese Academy of Science, State Key Lab. for Fatigue & Fracture of Matls., Shenyang 110015 China

The low-cycle fatigue lives and cyclic stress response characteristics of SiC particulate reinforced aluminum composites and its unreinforced counterpart were investigated at room and elevated temperature. The as-extruded specimens were cyclically deformed with fully-reversed loading under a range of constant plastic-strain amplitudes. The test results showed that the composites exhibited inferior fatigue endurance as compared to that of the unreinforced matrix. Also the cyclic response characteristics of the reinforced and unreinforced materials were different. In most of the fatigue life, the unreinforced aluminum mainly exhibited cyclic hardening, while the composite exhibited cyclic softening. Transmission election microscopy was used to examine the dislocation substructure developed during cyclic deformation at room and elevated temperature.

10:45 AM
The Cyclic Fatigue and Fracture Behavior of SiC Particulate-Reinforced 7034 Aluminum Alloy Metal-Matrix Composite: *Meslet Al-Hajri*[1]; T. S. Srivatsan[1]; X. Gao[1]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

A study was undertaken with the objective of rationalizing the influence of discontinuous ceramic particulate reinforcements on the cyclic fatigue response of 7034 aluminum alloy metal-matrix composites. The cyclic strain amplitude-controlled and stress amplitude-controlled fatigue properties and fracture characteristics of the 7034 aluminum alloy metal matrix discontinuously-reinforced with silicon carbide particulates will be highlighted. Specimens of the discontinuously reinforced aluminum alloy composite were cyclically deformed over a range of cyclic stress amplitudes, at two different load ratios. The influence of test temperature and load ratio on stress amplitude versus fatigue life stress response will be highlighted and discussed. In this presentation, the intrinsic mechanisms controlling the stress response and fatigue fracture characteristics will be discussed in light of composite microstructural effects, deformation characteristics of the composite constituents, cyclic stress amplitude, load ratio and test temperature.

11:05 AM
In-Situ Observations of Crack Opening Displacements (COD) and The Effect of Increased Interfacial Strength on Fibre Bridging in a Meta-Stable Titanium Composite: *F. Kaya*[1]; J. Liu[1]; P. Bowen[1]; [1]The University of Birmingham, Sch. of Metall. & Matls./IRC in Matls. for High Perfor. Applications, Birmingham B15 2TT UK

Compressive residual stresses play an important role in fibre bridging in composite materials. The mechanism of crack bridging includes debonding and sliding of high strength fibres. Hence, the magnitude of the initial debonding stresses and sliding length are important parameters in achieving effective bridging tractions. The initial debonding length of fibre/matrix interface is expected to be determined by the maximum applied stress. During in-situ observations, for similar crack lengths, the crack opening displacement (COD) (which may be proportional to the initial debonding and sliding length) of a testpiece under a higher applied load is, of course, larger than that of a testpiece under a lower applied load, as shown in figure

1. For a given stress intensity factor range, the fatigue crack growth resistance of titanium metal matrix composites is affected by the initial maximum applied stress intensity factor, K max, ini. As K max, ini increases the fatigue crack growth resistance of titanium based composite decreases. Eventual crack arrest, or specimen failure is determined for a given testpiece geometry by the number of intact fibres bridging the crack. As fibre debonding is encouraged by weak interfacial strength, composites containing weak interfaces are often preferred. However, for a given number of cycles, the length of the fatigue crack is much shorter and the fatigue crack growth rate is slower (as the crack opening displacements may be smaller) in composites with strong interfaces, provided fibres do not fail in significant numbers. Thus, recent mathematical modelling on the crack bridging suggests that increasing interfacial strength may actually be beneficial, providing the strength of the bridging fibres are sufficient to sustain increased load transfer due to increased interfacial shear strength. In this work the effects of increased interfacial shear strength on the fibre bridging and the crack opening displacements of fatigue cracks were investigated. The interfacial strength of meta-stable titanium matrix composite (Ti 21-S/ SCS-6) was increased via peak aging. The secondary precipitation, which was formed during aging of composite at 540°C for 8 hours leads to an increase in Young's modulus of the matrix and also to a volume expansion in the titanium matrix, thus increasing the compressive residual stresses around the fibres. Titanium based composite specimens which were in as-received and peak aged conditions were then tested in tension-tension loading using load ratio of R= 0.5 and frequency of 10 Hz, in order to attain a fully bridged fatigue crack. During fatigue testing, fibre fracture within each composite specimen was monitored simultaneously using acoustic emission to determine the number of broken fibres. Once fully bridged fatigue cracks were achieved, a fibre-probing technique was employed in order to determine the exact number and precise location of fractured fibres, within each composite test specimen. Finally, composite specimens were loaded monotonically and the crack opening displacements of bridged cracks were measured in-situ using a loading stage within in an Hitachi 4000 S FEG SEM. The crack opening displacements of composite specimens were also measured at 300°C. The results are discussed in terms of the reduced modulus of the meta-stable titanium matrix at such an elevated temperature and a reduction of the thermal mismatch between the SCS-6 fibres and the titanium matrix.

11:25 AM
Mechanical Property Evaluation of SiCp/AZ91D Magnesium Composites Prepared by a Novel Rotation-Cylinder Method: *Shae-Kwang Kim*[1]; Young-Jig Kim[1]; [1]Sungkyunkwan University, Sch. of Metall. & Matl. Eng., 300 Chunchun-dong, Jangan-gu, Suwon, Gyungi-go 440-746 Korea

A large number and variety of metal matrix composite (MMC) materials have been developed and the properties of interest include strength, stiffness and/or wear resistance. Low density is important for uses in the transport industry and in electronics applications where low inertia is required for rapidly moving parts. Magnesium alloys composites with particulate reinforcements are attractive candidate materials in these fields. Although the use of ceramic particulate instead of fibrous reinforcements has help to reduce the over-all material cost, however, for those applications, the cost related to the processes to prepare composite materials should also be considered. A novel Rotation-Cylinder method (RCM) has been developed for preparation of particulate reinforced MMC materials, with the aim of rapid incorporation and homogeneous distribution of reinforcement particulates in an ambient atmosphere. RCM claims to significantly reduce the time required for incorporation and particulate agglomerations by the characteristic U-shaped stabile melt surface with the Rankine vortex. Sound magnesium alloys composites can be produced in conjunction with subsequent forming processes, such as permanent mold casting, investment casting, squeeze casting and thixoforming. This paper presents the results of the mechanical properties of this low cost SiCp/AZ91D magnesium composite with some indications about the characteristic features of RCM.

Materials Design Approaches and Experiences - VI

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: Ji-Cheng Zhao, GE Corporate Research & Development, Physical Metallurgy Laboratory, Schenectady, NY 12301 USA; Michael G. Fahrmann, Special Metals Corporation, Technology Department, Huntington, WV 25705-1771 USA; Tresa M. Pollock, University of Michigan, Materials Science and Engineering Department, Ann Arbor, MI 48109-2136 USA

Thursday AM — Room: 204
November 8, 2001 — Location: Indiana Convention Center

Session Chair: John Agren, KTH, Stockholm, Sweden

8:30 AM
A Systematic Framework for Rational Materials Formulation and Design: *Venkat Venkatasubramanian*[1]; James Caruthers[1]; Santhoji Katare[1]; Priyan Patkar[1]; Prasenjeet Ghosh[1]; [1]Purdue University, Cheml. Eng., 1283 CHME Bldg., W. Lafayette, IN 47907 USA

Systematic development of new materials for specific engineering applications with optimal thermophysical, mechanical and/or biological properties is a key technical challenge with obvious commercial applications. Since traditional approaches use laborious and expensive trial-and-error procedures, there is considerable incentive in developing computer-assisted approaches to solve the problem. While one would like to address all aspects of design from a rigorous mechanistic consideration, the complexity of industrial design situations plagued by sparse and noisy data, limited fundamental understanding and stringent time and resource constraints do not often allow a totally fundamental approach. Therefore, a hybrid approach using first-principles modeling in tandem with expert rules and artificial intelligence tools seems most promising. To this effect, we will present a novel framework that integrates fundamental modeling, neural-networks and genetic algorithms for the materials-design problem. The applicability of this framework will be demonstrated on two widely different industrial product-design problems: fuel-additives design and sulfur-vulcanized elastomers.

9:00 AM Invited
Exploring Complex Material Systems using Combinatorial Materials Chips: *Xiao-Dong Xiang*[1]; [1]SRI International, 333 Ravenswood Ave., Menlo Park, CA 94025 USA

Conventional approach to mapping phase diagrams or exploring new materials is to make and characterize samples of discrete composition one at a time. Since 1995, in an effort to speed up this process, we have fabricated "combinatorial materials chips" (CMCs) in a format of "discrete material chip" and "continuous phase diagrams" (CPDs) using thin film deposition of elemental precursors through "combinatorial masks" or linear shutters. Thousands of distinct compounds or an entire continuous ternary phase-diagram can be formed in epitaxial thin film format on a small substrate. Various physical properties, including electrical impedance, optical, magnetic and structural properties, of these compounds are then mapped using various imaging instruments. We are routinely applying this approach to explore and optimize existing function materials and to study materials phase diagrams.

9:30 AM Invited
Simulation of Forging and Thermal Mechanical Processing: *Gangshu Shen*[1]; [1]Ladish Company, Inc., Adv. Matls. & Proc. Tech., 5481 S. Packard Ave., Cudahy, WI 53110 USA

Aerospace forging process development relies heavily on computer modeling. Process modeling provides a scientific tool for rapid evaluation of process changes for process optimization. Microstructure modeling is now the major emphasis in advanced forging process development. Improved processes, equipment and computer modeling tools are building on each other to rapidly advance the manner in which aerospace forging processes are designed and implemented, resulting in reduced development times, improved metal utilization, reduced component costs and enhanced mechanical properties.

10:00 AM Invited
Using Atomistic Modeling for Materials Design: *Michael Baskes*[1]; [1]Los Alamos National Laboratory, Struct./Prop. Relations Grp., G755, Los Alamos, NM 87545 USA

Advances in computers and atomistic modeling have made the realistic simulation of materials behavior possible. Two decades ago, modeling of materials at the atomic level used simple pair potentials. These potentials did not provide an accurate description of the elastic properties of materials or of the formation of free surfaces, a phenomenon critical in the fracture process. This talk will review the evolution of the Embedded Atom Method (EAM), a modern theory of metallic cohesion that was developed to overcome the limitations of pair potentials. The EAM includes many body effects that are necessary to describe such processes as bond weakening (or strengthening) by impurities. An extension of the EAM that is capable of describing bonding in all materials, the Modified Embedded Atom Method (MEAM), will also be discussed. Recently experiments have shown that the yield stress of materials depends upon the size of the sample for very small samples. Clearly an understanding of this phenomenon is needed if we are to build micro-devices. One interpretation of these experiments is that the phenomenon is caused by strain gradients. Atomistic calculations will be presented that show a size effect is present in small single crystals of Ni containing from 100 atoms to 100 million atoms under simple shear, a strain state that does not have an imposed strain gradient. These atomistic simulations reveal that dislocations nucleating at free surfaces are critical to causing microyield and macroyield in pristine material. The atomistic simulations and experimental data show that differences in applied strain rate, temperature, stacking faults (several FCC materials have been examined), deformation mode, and crystal orientation on yield and plasticity are small compared to the size scale effect. Development of high temperature materials is an important topic in materials design. Being able to predict the mechanical properties of these materials is now becoming possible. As an example of the applicability of atomistic methods to materials with complex bonding and complex crystal structure, we have studied the core structure and mobility of dislocations in the intermetallic material, MoSi2. A summary of our predictions of the dynamic behavior of dislocations in this material will be given. Processing is a critical component of materials design. For example, modeling of casting has advanced to a very sophisticated (and useful) stage. However, the advanced casting modeler requires accurate materials properties, e.g., melting point, viscosity, and diffusivity of liquid metals and their alloys, as input to his codes. Atomistic modeling can play an important role in obtaining some of this missing data. Utilizing molecular dynamics techniques, we present calculations of the melting point, diffusivity, and viscosity of several technologically important metals and their alloys (i.e. aluminum, nickel, and aluminum-nickel alloys). This work was supported at Los Alamos National Laboratory by the USDOE under contract W-7405-ENG-36.

10:30 AM Invited
Managing Corrosion Engineering Information for Metals in Hot Corrosive Gases: *Randy C. John*[1]; Arthur D. Pelton[2]; W. T. Thompson[3]; A. L. Young[4]; I. G. Wright[5]; T. M. Besman[5]; [1]Shell Global Solutions U.S., PO Box 1380, Houston, TX 77251 USA; [2]CRCT, 447 Berwick, Montreal, Quebec H3R 1Z8 Canada; [3]Royal Military College of Canada, Kingston, Ontario, K7K 5LO Canada; [4]Humberside Solutions, Ltd., Toronto, Ontario M6N 4X7 Canada; [5]Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA

The task of providing reliable information on corrosion of metals and alloys in high-temperature gases is difficult, due to the diversity of corrosive environments, and lack of standardization for corrosion data. The ASSET information system is being developed to address many of these difficulties. The present system now contains information on about 80 commercial alloys, 4,900 corrosion data measurements, and 6.4 million exposure hours. The system can be used to predict metal thickness losses by several corrosion mechanisms at high-temperatures as functions of gas composition, temperature, time, and alloy type. Use of the information system will be illustrated for corrosion in oxidizing, sulfidizing, sulfidizing/ oxidizing, and carburizing conditions.

Surface Composite Coatings (Session Dedicated to: Dr. Robert Reeber, ARO [Retired]) - II: Surface Composite Coatings - Processing

Sponsored by: Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: John C. Bilello, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; Ronald Gibala, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA

Thursday AM	Room: 112
November 8, 2001	Location: Indiana Convention Center

Session Chair: Carolyn R. Aita, University of Wisconsin–Milwaukee, Adv. Coating Experimental Lab., Milwaukee, WI 53201 USA

8:30 AM Invited
Ceramic Nanolaminate Coatings: *Carolyn R. Aita*[1]; [1]University of Wisconsin–Milwaukee, Adv. Coatings Experimental Lab., PO Box 784, Milwaukee, WI 53201 USA

In recent years, nanostructured ceramic coatings have emerged as technologically important materials chiefly because they can be tailored to exhibit unique properties and behavior not achievable in bulk. This paper addresses the design and synthesis of tailored structures in which two or more ceramics are combined in a multilayered nanocomposite: a nanolaminate. The sputter-deposited zirconia-alumina, zirconia-yttria, and zirconia-titania nanolaminates will be used as model systems to discuss intralayer phase selection, interface characteristics of as-grown coatings, and architectural stability of under extreme conditions of thermal and laser annealing. Lastly, we will show how these nanolaminates can be used as components in multifunctional "smart" coatings for mechanical and corrosion protection for biomedical applications. Support acknowledged under ARO Grants DAAH04-93-G-0238, DAAH04-95-1-0242, DAAG44-97-0097, AFOSR Grant F49620-95-1-0467, and NSF Grant 9988892.

9:00 AM
Laser and Plasma Deposition of Light Element Films on Structural Materials: *H. W. White*[1]; J. E. Chamberlain[1]; J. Wragg[1]; F. Shadipour[1]; Shen Zhu[1]; [1]University of Missouri, Dept. of Phys. & Astron., Columbia, MO 65211 USA

Carbon and nitrogen based protective films have been grown on structural materials using laser and plasma synthesis techniques. The combination IR-laser-assisted electron-cyclotron resonance plasma-enhanced chemical vapor deposition method was used to deposit Si-C-N and C-N films at low temperatures (*650°C) on steel substrates. ECR PECVD was used to grow diamond-like carbon films at low temperatures (*400°C) on Fe-based alloy substrates without diamond seeding or use of a template layer. The plasma species absorbed during nucleation of diamond grown on steel by ECR PECVD was investigated by in situ IRRAS. Supported in part by the Army Research Office, Research Triangle Park, NC, Grant Nos: DAAH04-94-0305 and DAAG55-97-1-0142.

9:25 AM
Evolution of Texture in Refractory Metal Films: *Steven M. Yalisove*[1]; [1]University of Michigan, Dept. of Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

The evolution of both in plane and out of plane texture in sputter deposited refractory metal films can be controlled with great precision using the correct geometry and processing conditions. The impact of this control is seen in the kinds of stress relief that a "quasi" single crystal film can accommodate via plasticity. Results will be shown which demonstrate this control and a model will be presented to explain the data.

9:50 AM
Carbo-Nitride Nanomaterials, Thin Films and Solids: *Valery N. Khabashesku*[1]; John L. Margrave[1]; [1]Rice University, Dept. of Chem., Houston, TX 77005-1892 USA

The novel carbon materials, such as heterodiamonds, built from carbon atoms covalently bonded to a heteroatoms (B, N, Si or P), are expected to find various applications due to their outstanding hardness, wear resistance, thermal conductivity and wide bandgap semiconducting properties. We have recently developed new synthetic methods for preparation of carbonitride materials which include cryogenic deposition of CNx thin films using atomic nitrogen beams, powder syntheses of bulk quantities of sp2-bonded solids with the C3N4 and B3C3N7 stoichiometry, and solvothermal synthesis of a previously unknown spherical carbon nitride nanopowder. These amorphous materials have been studied as precursors in high pressure-high temperature experiments to yield crystalline and hard C3N4 phases. The results of this research as well as of the similar studies of the single-wall carbon nanotubes demonstrating the formation of novel carbon materials, nanodiamonds and nanographite, will be presented and discussed.

10:15 AM Break

10:45 AM
Alloys and Compounds in the C-Si-Ge-Sn System: *J. Kouvetakis*[1]; J. W. Mayer[1]; [1]Arizona State University, Dept. of Chem., Ctr. for Solid State Sci., Tempe, AZ 85287 USA

Synthesis of new group IV semiconductors, which are intended for Si based bandgap engineering and lattice matching will be described. These materials are created heteroepitaxially on Si and include diamond cubic alloys and strain stabilized ordered phases in the C-Si-Ge-Sn system. Examples of new compound semiconductors include the Si4C,Ge4C, and (Si2Ge)Cx compositions with diamond-cubic structures and the Si3GeC4 phase with a sphalerite-like structure. Random alloy systems include silicon-germanium-carbon solid solutions, monocrystalline and nanostructured (quantum dots and wires) Ge1-xCx hybrids of Ge and diamond and related Si1-xCx materials. Current work aimed towards synthesis of new ternary Si-Sn-C and Ge-Sn highly metastable phases which might have direct gaps will be highlighted. The presentation emphasizes an approach that combines novel precursor chemistries and modern deposition techniques (ultra-high vacuum chemical vapor deposition) to develop thin films of new inorganic materials.

11:10 AM
Explosively Bonded Refractory Metal Gun Tube Liners: *Jonathan S. Montgomery*[1]; Robert Lowey[2]; Roger L. Ellis[3]; [1]US Army Research Laboratory, 405 Arsenal St. Research Lab., Attn. AMSRL-MA-MA, Watertown, MA 02472 USA; [2]TPL, Inc., 3921 Academy Pkwy. N. N.E., Albuquerque, NM, USA 87109-4416; [3]Naval Surface Warfare Center

This paper reviews the requirements of a gun tube coating. Not only are the materials requirements quite demanding, but the integration of the coating technique into gun tube production is also just as demanding. Cost is always an issue with gun tubes as well. There are many, many coating processes. Nearly all of them have been tried in the effort to ameliorate gun tube erosion. These have included carburization and nitridization, electroplating, ion implantation, laser treatments (with and without alloying), various thermal spray techniques, and sputtering. We will discuss coating materials and processes that have been tried, and the coating failure modes for each of these. Explosive bonding is very promising coating technique that appears to meet all the materials and systems integration requirements. 25 mm M242 Bushmaster barrels have been clad with a tantalum alloy, machined, and test-fired. This paper reports the results of this test program.

11:35 AM
Oxidation Protection of Carbon Materials by Acid Phosphate Impregnation: *Deborah D.L. Chung*[1]; Weiming Lu[1]; [1]University at Buffalo, Dept. of Mech. & Aeros. Eng., The State University of New York, Buffalo, NY 14260-4400 USA

The tendency for carbons to oxidize and become a vapor is a problem that limits the use of carbons at high temperatures. Structural carbons include carbon fibers, carbon-carbon composites and graphite. They are used for structures and electrodes in aerospace, metals and other industries. Acid phosphate (aluminum phosphate containing excessive phosphoric acid) impregnation, with ozone pretreatment, improves the oxidation resistance of carbon materials (polycrystalline graphite and pitch-based carbon fiber), as shown by weight measurement in air up to 1500°C. The impregnation involves using phosphoric acid and dissolved aluminum hydroxide in the molar ratio 12:1 and results in a rough, white and hard aluminum metaphosphate coating of weight about 20% of that of the carbon before the treatment. Without the ozone treatment, the impregnation is not effective. Without aluminum hydroxide, the impregnation even degrades the oxidation resistance of the carbon.

12:00 PM
Electrolytic Co-Deposition of Copper and Alumina in Magnetic Fields: *John Dash*[1]; [1]Portland State University, Phys. Dept., Portland, OR 97207 USA

A method is described to electroplate a composite of copper and alumina, using a magnetic field to suspend the alumina particles in an electrolyte containing copper ions. Both alpha and gamma alumina have been co-deposited, whereas previous investigators had little success in co-depositing gamma alumina in an electrolytic process, probably because of chloride ion impurity on the surfaces of the gamma particles. It was found that hardness of the coatings increased with increasing current density during electroplating. Microscopy and microchemical analyses were used to characterize the deposits.

Index

A

B

C

D

E

L

M

N

U

V

W

X

Y

Z