EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Cahn's Impact in Materials, Physics and Applied Mathematics; and Poster Talks

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Chemistry & Physics of Materials Committee, Phase Transformations Committee, Thermodynamics & Phase Equilibria Committee
Program Organizers: William C. Johnson, University of Virginia, Materials Science & Engineering Dept.; William J. Boettinger, National Institute of Standards & Technology, Room A153 Bldg 223; Carol Handwerker, National Institute of Standards & Technology, Room A153 Bldg 223, Gaithersburg, MD 20899 USA; Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Dept.; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Monday AM Room: International Ballroom
October 12, 1998 Location: O'Hare Hilton Hotel

Session Chair: David Laughlin, Carnegie-Mellon University

8:30 AM
THE IMPACT OF JOHN W. CAHN ON CURRENT AND FUTURE RESEARCH IN PHYSICAL METALLURGY: *H. I. Aaronson*[1]; [1]Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

The swift ascent of John Cahn from an extraordinarily bright and productive young man who recently joined the General Electric Research Laboratory (in its days of scientific glory) to the dominating force in physical metallurgy, and more recently to the principal developer of the rapidly thickening connections between this field and those of 'materials physics','materials chemistry' and 'materials mathematics', has been centered throughout upon phase transformations. This interest soon evoked a parallel emphasis upon phase equilibria, particularly in the presence of coherency and of sharp gradients in composition. Dr. Cahn's seminal contributions to spinodal decomposition were themselves evolved in parallel with those made to nucleated phase transformations. In the latter processes, interfaces are the central theme, from nucleation through growth and thence into coarsening. A repeatedly deepening concern with the physics, chemistry and mathematics of interfaces per se as well as of configurations of interfaces has been perhaps the most important and influential 'tieline' of his research from its earliest days. The corpus of work John Cahn has produced has been uniquely influential in molding the current form of physical metallurgy—and will doubtless become still more important in the future as the increasing sophistication of physical metallurgical researchers permits them to 'mine' his work for theoretical applications as well asto apply it to experiment with growing effectiveness.

9:30 AM
THE IMPACT OF JOHN W. CAHN ON CURRENT AND FUTURE RESEARCH IN PHYSICS AND APPLIED MATHEMATICS: *J. S. Langer*[1]; [1]University of California at Santa Barbara, Physics Department, Santa Barbara, CA 93106 USA

For over forty years, John Cahn has been pointing out to the rest of us the most important problems in materials research. To a remarkable degree, he has defined the field by identifying the physical principles underlying an enormously wide range of materials phenomena. In most of these cases, his intuition has provided the basis for deep new investigations in physics and mathematics. I shall present some examples in this lecture.

10:30 AM BREAK

10:50 AM
SCALING BEHAVIOR OF THREE-DIMENSIONAL DENDRITES: *Christoph Beckermann*[1]; Qiao Li[1]; [1]University of Iowa, Dept. Mechanical Engineering, 2212 Engineering Building, Iowa City, IA 52242 USA

The scaling behavior of geometry parameters in three-dimensional dendritic growth is investigated through a detailed measurement of the microstructure of pure succinonitrile dendrites grown on the first microgravity flight of the Isothermal Dendritic Growth Experiment (Glicksman et al., Phys.Rev.Lett., Vol.73, 573, 1993). Measurements are performed of the integral paramters of a sidebranching dendrite, such as the envelope shape, projection area, contour length, volume, surface area, and solid volume fraction. It is found that self-similar scaling relations exist between these geometry parameters and the primary tip radius or speed in steady growth. These relations are valid far from the tip, up to a normalized distance equal to the inverse of the tip Peclet number. The experimental results are compared to analytical predictions and generally found to be in good agreement.

10:55 AM
MACROSEGREGATION CAUSED BY THERMOSOLUTAL CONVECTION DURING DIRECTIONAL SOLIDIFICATION OF Pb-Sb ALLOYS: *Guolu Ding*[1]; S. N. Ojha[1]; L. Yu[1]; J. Reye[1]; S. N. Tewari[1]; S. N. Tewari[1]; [1]Cleveland State University, Chemical Engineering Department, E 24th Street at Euclid Avenue, Cleveland, OH 44115 USA

Pb-5.8 wt pct Sb alloy samples were directionally solidified with a positive thermal gradient of 140 K/cm at growth speeds ranging from 0.8 to 30 micrometer per second, and then quenched to retain the mushy zone morphology. Chemical analysis along the length of the directionally solidified portion, and in the quenched melt ahead of the dendritic array, showed extensive longitudinal macrosegregation. The extent of macrosegregation increases with decreasing growth speed. Increasing solutal build up at the dendrite tips is expected at lower growth speeds. The antimony rich melt is lower in density. The solutal instability resulting from this density inversion causes convection in the melt. The flow in the bulk melt, ahead of the dendritic array, is also entrained into the mushy zone. This produces longitudinal macrosegregation along the sample length. Similar behavior is observed in directionally solidified Pb-2.2 wt pct. Sb alloy. However, increasing antimony content of the alloy causes more severe macrosegregation.

11:00 AM
MICROSTRUCTURAL EVOLUTION IN Ni-Cr-Al DIFFUSION COUPLES: *Frederick Meisenkothen*[1]; John E. Morral[1]; [1]University of Connecticut, Institute of Materials Science, 97 North Eagleville Rd., U-136, Storrs, CT 06269-3136 USA

Interdiffusion between $\gamma+\gamma'$ and $\gamma+\beta$ Ni-Cr-Al alloys can result in the creation of a number of possible microstructures in the diffusion zone. One $\gamma+\gamma'$ alloy has been coupled with numerous $\gamma+\beta$ alloys to provide insight into the types of microstructures that can be produced. The resulting diffusion couples have been modeled using DICTRA computer simulations as well as error function solutions. Experimental alloys have been created that verify the results of these models. The results have aided in the construction of a map, that when super-imposed onto the Ni-Cr-Al phase diagram, will allow the prediction of the diffusion induced microstructure changes.

11:05 AM

DIFFUSION STRUCTURES AND DIFFUSION PATHS IN THE Fe-Ni-Al SYSTEM: Mysore A. Dayananda[1]; *Yong-Ho Sohn*[1]; [1]Purdue University, School of Materials Engineering, 1289 MSEE Building, West Lafayette, IN 47907 USA

Multiphase diffusion was investigated in the Fe-Ni-Al system at 1000ϒC with solid-solid diffusion couples assembled with [beta](B2), [gamma](fcc) single phase alloys and [beta+gamma] two-phase alloys for the development of diffusion structures and diffusion paths. The diffusion structures were examined metallographically and by scanning electron microscopy. Concentration profiles were determined by electron microprobe analysis and were employed for the direct determination of interdiffusion fluxes. Diffusion structures and diffusion paths are presented for a series of beta/gamma diffusion couples assembled with a common [beta] but different [gamma] alloys and discussed in terms of the various morphological features including planar and nonplanar interfaces and two-phase layers observed in the zone. Diffusion structures for multiphase couples assembled with [beta+gamma] two-phase terminal alloys are also presented to illustrate the phenomenon of demixing of phases and formation of adjacent [beta] and [gam ma] phase layers. Examples of uncommon diffusion paths that deviate from the conventional S-shaped paths will also be presented and discussed in the light of the ternary diffusional interactions among the components and the miscibility gap between [beta](ordered) and [beta]'(disordered) phases.

11:10 AM

MORPHOLOGY AND GROWTH KINETICS OF DISCONTINUOUS COARSENING IN FULLY LAMELLAR Ti-44 Al (AT %) ALLOY: Shinji Mitao[1]; *Leonid A. Bendersky*[2]; [1]NKK Corporation, Advanced Materials Engineering Lab., Fukuyama 721 Japan; [2]NIST, Metallurgy Div., Gaithersburg, MD 20899 USA

The morphology of discontinuous coarsening in the Ti3Al(alpha two)/TiAl(gamma) fully lamellar Ti-44 at. % Al alloy has been studied. Three types of morphology were observed in discontinuously coarsened lamellar structures. Type-(I) lamellae have the low energy habit plane as their lamellar interfaces, and have the same lamellar direction as the original primary lamellae. Type-(II) lamellae have the same crystallographic orientation of the alpha-two plates as that in the original primary lamellae, but have a different lamellar direction from the original primary lamellae, and have irregular faceted lamellar interfaces. Type-(III) lamellae have a different lamellar direction and a different crystallographic orientation of the alpha two plates from that in the original primary lamellae, but have the low energy habit plane as their lamellar interfaces. The growth kinetics of these three types of lamellae are discussed by modifying the Livingston and Cahn treatment in order to obtain the dependence of the secondary lamellar morphologies on misorientation between the penetrated primary lamellae, the advancing secondary lamellae and the lamellar colony boundaries.

11:15 AM

INFLUENCE OF SOLUTE-DRAG ON THE GROWTH OF PROEUTECTOID FERRITE IN Fe-C-Mn ALLOYS: *Masato Enomoto*[1]; [1]Ibaraki University, Department of Materials Science, Hitachi, 3168511 Japan

The growth of proeutectoid ferrite in Fe-C-Mn alloys is simulated by Green function method incorporating the drag on ferrite: austenite interfaces by segregated alloying element (Mn). The retardation of growth appear to be insignificantly small at low undercoolings where the bulk Mn partition between ferrite and austenite was observed to occur. The drag effect is appreciable at high undercoolings where the bulk Mn partition does not occur, but accounts for only a small fraction of the differences in growth rate between theory and experiment previously reported.

11:20 AM

A RE-EXAMINATION OF CAHN AND HAGEL'S DIVERGENT PEARLITE: *C. R. Hutchinson*[1]; R. E. Hackenberg[1]; G. J. Shiflet[1]; [1]University of Virginia, Department of Materials Science and Engineering, Thornton Hall, Charlottesville, VA 22903 USA

An Fe-0.6C-5Mn alloy was investigated with the intention of reexamining the divergent pearlite reported by Cahn and Hagel (Acta Metall., 1963, vol. 11, pp. 561-574). Interlamellar spacing and growth rate measurements have been made using a 3-dimensional sectioning technique in order to circumvent the stereological problems traditionally encountered in pearlite studies. Analytical electron microscopy was utilized to determine the manganese concentration profile in the vicinity of the pearlite growth front. This, coupled with thermodynamic calculations, helps to illuminate the roles of manganese and carbon in accounting for the non-steady state growth rate and lamellar divergence peculiar to this phase transformation. The analysis of Cahn and Hagel will be re-evaluated in light of these findings. This research was supported by the National Science Foundation.

11:25 AM

THE DEVELOPMENT OF SPATIAL CORRELATIONS DURING OSTWALD RIPENING: A TEST OF THEORY: Vicki Snyder[1]; Jens Alkemper[1]; Peter W. Voorhees[1]; [1]Northwestern University, Materials Science and Engineering, 2225 N. Campus Drive, Evanston, IL 60208 USA

Coarsening experiments were performed in a model system consisting of solid Sn particles in a Pb-Sn eutectic liquid. This system satisfies all the assumptions of theory, thus allowing a comparison between the theoretical predictions and the experimental results. Spatial correlation functions on plane sections for coarsened samples with volume fractions of 0.1, 0.2, and 0.7 solid particles were measured. The correlations functions for 0.1 solid volume fraction samples are compared to the results of calculations of Akaiwa and Voorhees and to a random distribution of non-overlapping particles. The two-dimensional spatial correlations were determined at several different times for the 0.1 volume fraction samples. The correlations were found to evolve to a scaled time-independent distribution. The effect of volume fraction on spatial correlations is also shown using results from the 0.2 and 0.7 volume fraction samples.

11:30 AM POSTER VIEWING

GENERAL ABSTRACTS: Fatigue and Fracture

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; David Bourell, University of Texas, Austin, TX 78712-1063 USA; Rusty Gray, Los Alamos National Lab, Dynamic Properties, Los Alamos, NM 87545-0001; Howard Sizek, Inco Alloys International, Mechanical Testing, Huntington, WV 25705

Monday AM Room: Athens/Berlin
October 12, 1998 Location: O'Hare Hilton Hotel

Session Chair: Michael J. Vinarcik, Ford Motor Company, Product Development Center, Dearborn, MI 48121-2053

8:30 AM
IMPORTANCE OF CHEMICALLY SHORT-CRACK GROWTH ON FATIGUE LIFE: *Evan Jarrett Dolley*[1]; Robert P Wei[1]; [1]Lehigh University, Mechanical Engineering and Mechanics, 19 Memorial Drive West, Bethlehem, PA 18015-3085 USA

Environmentally enhanced fatigue crack growth (CFCG) rates in aircraft structural components must be explicitly taken into account in the development of life prediction and management methodology. CFCG behavior may be separated into a long-crack and chemically short-crack regime. Experimental data on 2024-T3 and 7075-T6 aluminum alloys used in aircraft construction show that the CFCG rates are about 10X that in a dry environment in the long-crack regime. In the chemically short-crack regime, the growth rates are further enhanced by more than a factor of two and the effect extends to crack lengths up to 6 mm. Crack growth results on 2024-T3 and 7075-T6 aluminum alloys are presented and compared. The role of dissolved oxygen and the micromechanism on chemically short-crack growth are discussed. The implication of this regime of CFCG on fatigue life is also considered. This research is supported by the AFOSR under Grant F49620-1-96-0245 and by the FAA under Grant 92-G-0006.

8:55 AM
EFFECT OF MIXED MODE (I-II) LOADING ON NEAR THRESHOLD FATIGUE CRACK GROWTH BEHAVIOR OF 2014-T651 ALUMINUM ALLOY: *P. S. Shankar*[1]; P. R. Arora[2]; E. S. Dwarakadasa[3]; [1]University of Utah, Metallurgical Engineering, 135 South 1460 East Rm 412, Salt Lake City, UT 84112 USA; [2]Indian Institute of Science, Department of Mechanical Engineering, Bangalore, Karnataka, 560 012 India; [3]Indian Institute of Science, Department of Metallurgy, Bangalore, Karnataka, 560 012 India

Near threshold fatigue crack growth response of 2014-T651 aluminum alloy was evaluated under pure mode I (β = 90ϒ) and two mixed mode I-II (β = 60ϒ & β = 45ϒ) loading conditions at load ratio R=0.1. Compact tension shear specimens were employed in conjunction with a loading device capable of providing seven different loading angles from β = 90ϒ (pure mode I) to β = 0ϒ (pure mode II). Equivalent threshold stress intensity factor range (ΔK_{Vth}) under mode I was determined by the method of load shedding whereas that under mixed mode was obtained using a load increment technique. ΔK_{Vth} under both mixed mode conditions is higher than under pure mode I. Under mixed mode, ΔK_{Vth} at β = 60ϒ is higher than that under β = 45ϒ. Roughness induced crack closure alters the kinetics of near threshold crack propagation significantly in both β = 60ϒ & β = 45ϒ. Oxide induced crack closure is also observed under β = 60ϒ.

9:20 AM
INFLUENCE OF PRE-EXISTING CORROSION PITS ON FATIGUE LIFE IN A 2024-T3 ALUMINUM ALLOY: Evan Jarrett Dolley[1]; *Baekho Lee*[1]; Robert P. Wei[1]; [1]Lehigh University, Mechanical Engineering and Mechanics, 19 Memorial Drive West, Bethlehem, PA 18015-3085 USA

The fatigue lives of aircraft structures may be reduced as a result of exposure to corrosive environments. One form of corrosion is pitting which can act as crack nuclei. Fatigue testing of a 2024-T3 aluminum alloy pre-corroded in a 0.5M NaCl solution from 2 to 16 days, was conducted and showed that fatigue lives can be reduced by more than one order of magnitude after 16 days of corrosion compared to uncorroded specimens. Crack evolution is monitored by the electrical potential drop (EPD) technique. The crack initiating pit sizes were determined by post-fracture SEM examination and found to be in the range of 20 to 100µm. The reduction in fatigue life is interpreted in terms of the influence of pit size or the time of exposure to the corrosive environment. The relative roles of crack nucleation and growth are discussed. Research was supported by FAA, Grant 92-G-0006, and AFOSR, Grants F49620-96-0245 and F49620-98-1-0198.

9:45 AM
COMPLIANCE CHANGE RESULTING FROM STRESS GRADIENT DRIVEN FLUX OF HYDROGEN: *Guiru Liu Nash*[1]; [1]IIT, MMAE, 10 W 32nd St., Chicago, IL 60616 USA

A compliance change was observed during fatigue testing of ASTM A710 HSLA steel when the environment was changed from hydrogen to air. This compliance change can be attributed to a change in Young's modulus locally at the crack tip. A finite element model was used to determine that the observed compliance change would result from a 25% change in modulus in a 0.0027 mm3 volume element at the crack tip. It is proposed that such a change can result from the dilation of the lattice resulting from a stress gradient driven flux of hydrogen to the crack tip. This results in a non-uniform distribution of hydrogen in the sample. Calculations of the magnitude of the diffusion flux to the crack tip under the stress gradients imposed during fatigue support this idea.

10:10 AM
INFLUENCE OF INTERFACIAL FRACTURE RESISTANCE ON THE MECHANICAL BEHAVIOR OF LAYERED CERAMICS: *Desiderio Kovar*[1]; M. D. Thouless[2]; John W. Halloran[3]; [1]University of Texas at Austin, Department of Mechanical Engineering, Building ETC, Rm, 9.158, MC C2201, Austin, TX 78712 USA; [2]University of Michigan, Mechanical Engineering and Applied Mechanics Department, 2282 G.G. Brown Building, Ann Arbor, MI 48109-2125 USA; [3]University of Michigan, Materials Science and Engineering Department, 2168 H.H Dow Building, Ann Arbor, MI 48109-2136 USA

Recently it has been shown that high strength, high energy absorption, and graceful failure can be achieved using low-cost layered ceramics with weak interfaces. In this study, multilayered ceramic composites consisting of layers of Si3N4 separated by a weak interphase containing BN+Si3N4 were prepared and tested. The composition of the interphase was varied to alter the interfacial fracture resistance. Measurements revealed that the interfacial fracture resistance could be varied from 30 J/m2 to greater than 90 J/m2 by the addition of Si3N4 to the interphase. The influence of interfacial fracture resistance on mechanical behavior was determined using flexural tests. It was found that energy absorption was maximized at low values of interfacial fracture resistance where widespread delamination cracking was observed within the weak interphase.

10:35 AM BREAK

10:50 AM
EFFECT OF LOW TEMPERATURE AGING ON TENSILE AND FRACTURE PROPERTIES OF CF8 STAINLESS STEEL: *M.D. Mathew*[1]; K.L. Murty[1]; V.N. Shah[2]; [1]North Carolina State University,

Department of Nuclear Engineering, Raleigh, NC 27695 USA; [2]Idaho National Engineering and Environmental Laboratory, Advanced Nuclear Energy Products, Idaho Falls, ID 83415-3870 USA

CF8 stainless steel is used for the main coolant loop piping, and pump and valve bodies in nuclear power plants. These components are subject to thermal aging at the reactor operating temperatures. Since the aged structures are prone to brittle failures, the aging characteristics of these materials are important to be well characterized. Cast CF8 SS has a duplex microstructure consisting of austenite and, about 17% delta-ferrite. Thermal aging leads to changes in the ferrite content and in general, results in increased strength and decreased toughness. We report here the effect of low temperature aging (673 K for 1.58x107 sec or 4388 hours) on the tensile and fracture behaviors of cast CF8 austenitic stainless. The aging resulted in a drop in the d-ferrite content by about 8%. Charpy impact tests were performed at temperatures ranging from 77 K to 423 K, and the aged material resulted in a decrease in the Charpy energy of about 45%. Correspondingly, the tensile strength increased. These materials are also known to exhibit strain-rate sensitive fracture toughness. Work is underway to investigate the influence of the strain-rate on the mechanical strength using conventional tensile tests. In addition, the nondestructive ball-indentation tests are in progress to characterize the mechanical and fracture characteristics, and the results to-date will be presented. This work is supported by the project funded by the INEEL University Research Consortium. The INEEL is managed by Lockheed Martin Idaho Technologies Company for the U.S. Department of Energy, Idaho Operations Office, under Contract No. DE-AC07-94ID13223. Thanks are due to Ms. Lori Lietzen for assistance in the experimental work.

11:15 AM

EFFECTS OF ANNEALING TREATMENT ON THE MECHANICAL BEHAVIOR OF BULK Zr-Ti-Ni-Cu-Be: *Peravudh Lowhaphandu*[1]; Lorie A. Ludrosky[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Dept. Materials Science & Eng., 10900 Euclid Ave., Cleveland, OH 44106 USA

The effects of annealing time and temperature on the mechanical behavior of bulk (i.e. 4 mm or 7 mm thick) Zr-Ti-Ni-Cu-Be metallic glass were determined. The annealing was performed at two temperatures at various time periods in an argon atmosphere. The evolution of the structure was characterized by X-ray diffraction, while the subsequent mechanical behavior was determined via a variety of test techniques; including compression testing, notched bend testing, fracture toughness testing, and microhardness. Both optical metallography and SEM fractography were utilized to characterize the evolution of deformation and fracture mechanisms in the as-received amorphous materials as well as the annealed materials. The properties of both 4 mm thick plate and 7 mm thick plate will be compared. The project is supported by AFOSR-AASERT-F49620-96-1-0228.

11:40 PM

THE INTERACTION BETWEEN SCREW DISLOCATIONS AND A STAR CRACK WITH A CENTRAL HOLE ALONG AN INTERFACE: *Sanboh Lee*[1]; Bii-Trong Chen[1]; [1]National Tsing Hua University, Dept of Materials Science, 101 Kuang Fu Rd., Sec.2, Hsinchu, Taiwan 30043 ROC

The elastic interaction between screw dislocations and a star crack with a central hole along an interface has been investigated. Consider M cracks uniformly distributed along the circumference of a circular hole. The shear moduli of upper and lower media are different. Based on the solution of an interfacial crack, this problem is analyzed using the conformal mapping technique. The complex stress field, stress intensity factor, strain energy and image force of dislocation are derived. The effect of the shear modulus ratio on the above physical parameters is studied. Two dislocation sources are included. When the disloction is located at the interface, the stress field, stress intensity facotr at the crack tip and image force on dislocation are obtained from the those in the single phase media if the shear modulus of single phase media is replaced by the effective shear modulus. The effective shear modulus is equal to the harmonic mean of both shear moduli of upper and lower media. This work was supported by the National Science Council, Taiwan, Republic of China.

GENERAL ABSTRACTS: Deformation

Sponsored by: TMS

Program Organizers: TMS, Warrendale, PA 15086 USA; David Bourell, University of Texas, Austin, TX 78712-1063 USA; Rusty Gray, Los Alamos National Lab, Dynamic Properties, Los Alamos, NM 87545-0001; Howard Sizek, Inco Alloys International, Mechanical Testing, Huntington, WV 25705

Monday AM Room: Sydney
October 12, 1998 Location: O'Hare Hilton Hotel

Session Chair: Viola L. Acoff, The University of Alabama, Department of Metallurgical and Materials Engineering, Tuscaloosa, AL 35487-0202 USA

8:30 AM

EFFECT OF THERMOMECHANICAL PROCESSING VARIABLES ON THE MECHANICAL PROPERTIES AND MICROSTRUCTURES OF HIGH STRENGTH LOW ALLOY (HSLA) STEELS: *M. Hadji*[1]; Y. Sikaddour[1]; M. Temar[1]; S. Zidelmel[1]; [1]University of Blida, Institute of Mechanical Engineering, P.O. Box 270, Blida Algeria

The type of High Strength Low Alloy (HSLA) steel has changed from conventionally heat treated steel to TMCP steel, because the latter steel provides improved strength-toughness balance and weldability. Enhanced properties of TMCP steel derive from control of the hot deformation and accelerated cooling process during plate production. In the present paper, the effects of thermomechanical processing and direct quenching on the strength of three HSLA steels, 0.15%C-1.5%Mn-0.18%Si-0.24%Ti- 0.15%C-1.5%Mn-0.18%Si-0.16%Ti-0.06%V-0.03%Nb, and 0.15%C- 1.5%Mn-0.18%Si-0.24%Ti- 0.06%V-0.03%Nb were investigated over a range of tempering temperature. Compared to standard reaustenitise and quench processing, plates produced with direct quenching showed consistently superior strength upon tempering. The processing and microstructural reasons for these differences will be evaluated and discussed.

8:55 AM

EFFECT OF MICROSTRUCTURE ON THE MECHANICAL PROPERTIES OF Ti-6Al-2Sn-2Zr-2Mo-2Cr: *Xiao-Dong Zhang*[1]; Jorg Wiezorek[1]; Paul Bonniwell[2]; William Baeslack[3]; Daniel Evans[4]; Daniel Evans[4]; Hamish Fraser[1]; [1]The Ohio State University, Dept. Mater. Sci. Eng., 2041 College Rd, Columbus, OH 43210 USA; [2]The Ohio State University, Dept. Ind. Sys. Welding Eng, 2041 College Rd, Columbus, OH 43210 USA; [3]The Ohio State University, College of Engineering, 2070 Neil Ave, Columbus, OH 43210 USA; [4]Wright Patterson AFB, Materials Directorate, WL/MLLM, Dayton, OH 45433 USA

The microstructural development of Ti-6Al-2Mo-2Cr-2Sn-2Zr (Ti-6-22-22) alloys with or without Si addition has been studied by optical microscopy, scanning electron microscopy (SEM), conventional transmission electron microscopy (CTEM) and high resolution electron microscopy (HREM) after various heat treatments, namely cooling rates from b and a+b phase field, a+b heat treatment temperature and aging temperature. It is found that microstructural morphologies change from colony structure to Widmanstätten lamellae with increase b cooling rate, while the volume percent of a and b phase is determined by a+b cooling rates. It has been observed that aging temperature has a significant influence on the precipitation and stability of the precipitates. These various microstructures have strong effect on the fracture toughness and tensile properties of the alloys.

Results on the macro and micro deformation modes associated with crack initiation and dislocation characteristics from different heat treatment will also be presented. The importance of these observations will be discussed in view of the fundamental understanding and further control of the structural stability of these type of a-b Ti alloys.

9:20 AM

TIME DEPENDENT TWINNING DURING AMBIENT TEMPERATURE COMPRESSION CREEP OF ALPHA Ti-Mn ALLOY: Carl A. Hultgren[1]; Charles A. Greene[1]; *Sreeramamurthy Ankem*[1]; [1]University of Maryland, Dept. of Materials and Nuclear Engineering, College Park, MD 20742-2115 USA

Recently it has been shown that time dependent twinning is the primary mechanism during ambient temperature tensile creep deformation of coarse grained alpha Ti-Mn alloy. In the present study, the same alloy was subjected to a compressive stress and ambient temperature creep deformation behavior studied. A similar type of time dependent twinning is also found to be the primary reason for the compressive creep. Some of the twins formed instantaneously and grew with time and other twins nucleated after many hours. Details of the investigation will be presented. *This work is being funded by the Office of Naval Research under Grant No. N000149610819.

9:45 AM

THE EFFECT OF GRAIN SIZE ON THE CREEP DEFORMATION OF GAMMA-BASED TITANIUM ALUMINIDES: *Eric A. Ott*[1]; Tresa M. Pollock[1]; [1]Carnegie Mellon University, Materials Science and Engineering, 5000 Forbes Ave., Pittsburgh, PA 15232 USA

A number of studies have shown that grain size influences creep deformation in gamma-based titanium aluminide alloys. This is often attributed to grain boundary sliding; however, there have been no detailed experimental studies of sliding in this system. This investigation explores grain size effects on high temperature creep deformation. The results of creep experiments performed at 760°C on equiaxed gamma microstructures for a Ti-48Al-2Cr-2Nb type alloy and a similar alloy with an addition of Mo will be presented. The dependence of minimum creep rates on grain size and the development of wedge cracks and intergranular damage in the later stages of creep for both alloys suggest that the relative motion of individual grains may contribute to the overall creep strain. Surface measurements of fiducial line displacements at grain boundaries will be reported for polycrystalline equiaxed structures with grain sizes of 10 to 100 microns which have been creep tested in vacuum.

10:10 AM

THE EFFECT OF Ni ON THE CREEP BEHAVIOR OF Ti-6Al-2Sn-4Zr-2Mo: *Robert Walter Hayes*[1]; P. Gouma[2]; G. B. Viswanathan[2]; Glenn S. Daehn[2]; Michael J. Mills[2]; [1]Metals Technology Inc., 19801 Nordhoff Street, Northridge, CA 91324 USA; [2]Ohio State University, Dept. of Materials Science, 2041 College Road, Columbus, OH 43210-1179 USA

Trace levels of Ni are found to have a significant impact on the high temperature creep behavior of the alloy Ti-6Al-2Sn-4Zr-2Mo over the temperature and stress regime of 510 to 560C and 172 to 414 MPa. The values of the stress exponents obtained for both low and higher Ni material suggest that creep deformation is controlled by a dislocation creep process over this temperature - stress regime. The apparent creep activation energy obtained for the low Ni material is in fair agreement with the recently reported value for the intrinsic self - diffusion in alpha titanium. The apparent creep activation energy obtained for the higher Ni material has decreased significantly relative to that for the lower Ni material and that for the intrinsic self - diffusion value. Preliminary results suggest that the influence of Ni on the creep behavior of this alloy occurs through the impurity enhanced self - diffusion in alpha titanium which has recently been discussed by Koppers et al.

10:35 AM BREAK

10:50 AM

STRUCTURE AND CREEP PROPERTIES OF Al-Sc ALLOYS: Christian B. Fuller[1]; David N. Seidman[1]; David C. Dunand[1]; [1]Northwestern University, Department of Materials Science and Engineering, 2225 North Campus Drive, Evanston, IL 60208-3108 USA

Al-Sc alloys with up to 0.35 wt.% scandium were solutionized and aged to form fine, coherent Al_3Sc precipitates within coarse aluminum grains, as observed by transmission electon microscopy and optical microscopy. Tensile creep properties of the dispersion-strengthened Al-Sc alloys were investigated in the temperature range of 200 - 350^o C. A threshold stress is observed and discussed in the light of dislocation-particle interaction.

11:15 AM

LOW TEMPERATURE DEFORMATION OF RUTHENIUM ALUMINIDE POLYCRYSTALS: *Ding-Chung Lu*[1]; Teresa M. Pollock[1]; [1]Carnegie Mellon University, Department of Materials Science and Engineering, 5000 Forbes Ave, Pittsburgh, PA 15213 USA

The flow behavior and dislocation substructure present in ruthenium aluminide polycrystals due to deformation at room temperature and 77 K have been studied. Compared with the other high temperature B2 compounds RuAl has a relatively high yield strength and work hardening rate. Dislocations with three different types of Burgers vectors have been identified after I~2% compression deformation at 77 K and room temperature: a<100>, a<110> and a<111>. The a<100> and a<110> dislocations are present with approximately equal densities, while the a<111> are only occasionally observed. Trace analyses show that the majority of the dislocations are mixed in character and lie on {110} type planes. The implications of these observations with regard to the number of independent slip systems and the intrinsic deformability of this material are discussed.

11:40 AM

PROPERTY MEASUREMENTS AND MICROSTRUCTURAL SIMULATIONS FOR POROUS METALS: *Peter Matic*[1]; Richard K. Everett[1]; Robert J. Bonenberger[2]; Anthony J. Kee[3]; [1]Naval Research Laboratory, Mechanics of Materials Branch, Code 6382, Washington, DC 20375 USA; [2]FM Technologies, Inc., 10529-B Braddock Road, Fairfax, VA 22032 USA; [3]Geo-Centers, Inc., P.O. Box 441340, Fort Washington, MD 20749 USA

Gasar porous metals feature a variety of complex pore morphologies over a wide range of porosities. The relationship of these morphologies to bulk properties has been studied using experimental and computational techniques. The purpose of this presentation is to combine recent results on bulk property measurements, microscopy and image based modeling of pure copper and aluminum alloy gasar pore microstructures. Measurements of elastic modulus, yield strength and nonlinear elastic-plastic responses of the bulk material are presented. Finite element simulations, aimed at translating microstructural images to these properties, have been used to gain insight into the role that pore morphology and near neighbor interactions have on the bulk material response and how these complex pore morphologies can be quantified for purposes of description and modeling of these microstructures. Finally, the role of microplastic connectivity in bulk yield is discussed as it relates to the comparatively large gap between loss of proportionality and the onset of bulk plastic flow seen in these materials.

12:05 PM

DISLOCATION TRANSMISSION THROUGH A BIMATERIAL INTERFACE BASED ON PEIERLS DISLOCATION MODEL: *X. J. Xin*[1]; *P. M. Anderson*[1]; *P. M. Anderson*[1]; *Y. Cheng*[1]; [1]The Ohio State University, Dept of Materials Science and Engineering, 2041 College Road, Columbus, OH 43210 USA

The interaction of a dislocation with a bimaterial interface is studied using a distributed core, Peierls-type, representation of a dislocation. The relation between relative slip and resolved shear stress across the operative slip plane is similar to that proposed by Rice and Beltz. The formalism is more general than the original Peierls approach, in that the strength of the interface in shear and the maximum stacking fault energy may be varied independently. The critical applied shear stress required to push a dislocation through a bimaterial interface is found to depend on the shear moduli of both materials and the unstable stacking energy. The effect of the shear-traction relation on the core structure of the dislocation and the strengthening mechanism for layered materials are discussed.

HOT DEFORMATION OF ALUMINUM ALLOYS: Microstructure Characterization, Scale

Sponsored by: Materials Design and Manufacturing Division, Structural Materials Division, Metal Processing & Fabrication of Mat. Soc., Non-Ferrous Metals, Shaping and Forming

Program Organizers: Thomas R. Bieler, Michigan State University, Dept. of Matls Sci & Mechanics, East Lansing, MI 48824-1226; Lawrence Lalli, Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA; Stuart MacEwen, Alcan International, Kingston, Ontario K7L 5L9 Canada

Monday AM — Room: Orchard
October 12, 1998 — Location: O'Hare Hilton Hotel

Session Chairs: R. D. Doherty, Drexel University, Dept. of Materials, Philadelphia, PA 19104 USA; L. P. Kubin, LEM, 29 Av. de la Division Leclerc, Cedex 92322 France

8:30 AM INVITED PAPER

LARGE-STRAIN DEFORMATION OF ALUMINUM SINGLE CRYSTALS AT ELEVATED TEMPERATURE: *M. E. Kassner*[1]; [1]Oregon State University, Dept. of Mechanical Engineering, Corvallis, OR 97331 USA

High-purity aluminum single crystals were deformed to large strains at a temperature of about 0.7 Tm, at moderate strain-rates. The purpose of the experiments was two-fold: First, there was an interest in establishing the dynamic restoration mechanisms, particularly in view of recent careful work by other investigators at elevated temperatures, also on single crystals. Candidate mechanisms are geometric dynamic recrystallization (GDX) and discontinuous dynamic recrystallization (DRX). Second, there was an interest in determining the presence of internal stresses, particularly in the vicinity of subgrain walls using convergent beam electron diffraction (CBED). This latter investigation provides insight into the controlling mechanisms of creep.

9:00 AM INVITED PAPER

HOT AND COLD DEFORMED AA1050 AA2104 DEFORMATION MICROSTRUCTURE AND RECRYSTALLIZATION BEHAVIOR: *D. Juul Jenson*[1]; M. T. Lyttle[1]; N. Hansen[1]; [1]Riso National Laboratory, Materials Research Department, Roskilde DK-4000 Denmark

Two aluminum alloys, AA1050 and AA3104 were deformed by rolling or plane strain compression (PSC) at room temperature and at 400°C to various strains in the range of 0.05- 2.5. The hot deformations were done by PSC as this process allows immediate quenching after the deformation. The deformation-microstructures were characterized by transmission electron microscopy (TEM) and the local distributions of crystallographic orientations were analyzed by Kikuchi patterns in the TEM and by electron back scattering patterns (EBSPs) in the scanning electron microscope (SEM). The development in deformation microstructure and in crystallographic subdivision of the original grains with increasing strain are described with most emphasis on the crystallographic subdivision. It is found that the original grains typically subdivide into a number of smaller volume elements bounded by low angle dislocation boundaries and by high angle boundaries having misorientations larger than 15% both during cold and hot deformation. The deformation temperature, however, has significant effects on the size and shape of these volume elements. In the paper, the results are presented and discussed according to a general framework for the microstructural development with increasing strain. As the recrystallization behaviour depends on the deformation microstructure, the deformation temperature also affects the recrystallization. In the present work this is investigated by a crystallographic Cahn-Hagel analysis of the growth rate kinetics. It is found that both the time and orientation dependencies of the growth rates are affected by the deformation temperature. These results are discussed and related to the observed deformation microstructures.

9:30 AM INVITED PAPER

DISLOCATION STRUCTURES FORMED DURING HOT AND COLD WORKING: *D. A. Hughes*[1]; A. Godfrey[1]; [1]Sandia National Laboratories, Center for Materials and Eng. Sciences, P. O. Box 969, Livermore, CA 94550 USA

During many types of deformation, grains are subdivided into smaller regions by dislocation boundaries. The classification of structures formed by grain subdivision has differed between structures observed following cold deformation and hot deformation. Cold deformed microstructures are characterized by grain subdivision at two size scales. Grain subdivision occurs at the larger scale by geometrically necessary boundaries that delineate differently deforming blocks that contain nearly equiaxed cells. In contrast, hot deformed structures have been described with a single level of subdivision by equiaxed subgrains. Infrequently an occasional long boundary provides subdivision at a larger level. However, new findings in hot deformed alloys show that there are closer ties between the cold and hot deformed structures. Specifically, two levels of grain subdivision are observed following some types of high temperature deformation when large areas are surveyed in the TEM. Furthermore, the probability densities of boundary misorientation angles at high temperature show the same universal scaling as those at low temperature. This work supported by the Office of Basic Energy Sciences, U.S. DOE, under contract no. DE-AC04-94AL85000.

10:00 AM INVITED PAPER

NEAR-NET SHAPE FORMING OF Al-Li 2195 ALLOY: MICROSTRUCTURE AND TEXTURE EVOLUTION: *Peter N. Kalu*[1]; [1]FAMU-FSU College of Engineering, National High Magnetic Field Laboratory, Tallahassee, FL 32310 USA

The microstructural and texture evolution in Al-Li 2195 alloy resulting from Near Net Shape forming has been evaluated. Although the processing method is complex, a detailed description of the microstructure and texture was accomplished via the use of optical microscopy and x-ray texture analysis. The texture developed at the early stages of processing was similar to that produced by conventional extrusion: two fibers, <111> and <100> aligned with the axial direction. At the intermediate processing stage, a through thickness microstructural gradient consisting of three distinct regions was developed. While the surface regions were dominated by {001}<110> shear, the mid-section region was characterized mainly by {112}<110> shear. The texture of the final product was dominated by {112}<110> shear. This accounted for over 40% of the texture across the thickness of the cylinder. Results of this investigation are discussed in terms of the mechanisms responsible for the development of texture and microstructure.

10:30 AM INVITED PAPER

THE EFFECT OF COMPOSITION ON THE MICROSTRUCTURE AND TEXTURE FORMATION DURING HOT DEFORMATION AA5XXX ALUMINUM ALLOYS : *M. A. Wells*[1]; D. J. Lloyd[2]; [1]University of British Columbia, Centre for Metallurgical Process Engineering, Department of Metals and Materials Engineering, 309-6350 Stores Road, Vancouver BC V6T 1Z4 Canada; [2]Alcan International Limited, Kingston Research and Development Centre, P.O. Box 8400, 945 Princess Street, Kingston, Ontario KZ7 5L9 Canada

During industrial hot deformation of AA5XXX aluminum alloys a vast difference in both the microstructure and texture evolution is observed. Plane strain compression and Gleeble compression tests were done on as-cast and hot breakdown rolled aluminum AA5182 and AA5052 samples at various strain, strain rates and temperatures, to examine the difference in flow stress and microstructural and texture evolution after hot deformation. This paper will examine the effect of %Mg on the recrystallization kinetics, flow stress and texture evolution for the AA5XXX aluminum alloys during hot deformation and compare these results to what has been found in the literature for similar alloys.

11:00 AM INVITED PAPER

A COMPARISON OF MICROSTRUCTURAL CHARACTERISTICS OF HOT DEFORMED STRIP CAST AND DC CAST AA 5182 ALLOYS: *Y. L. Liu*[1]; J. G. Morris[1]; [1]University of Kentucky, Light Metal Research Labs, Lexington, KY 40506 USA

As a package material, AA5182 aluminum alloy is mainly produced by the traditional DC casting method. However the strip cast route provides certain significant advantages. The different processing routes give rise to different microstructures, which have great influence on the mechanical properties and formability of the alloy. It is of significant industrial interest to initiate an investigation of the microstructural characteristics of the alloys produced by different processing routes. In the present study, AA5182 aluminum alloy was produced by both the DC cast and the strip cast processes, respectively. The microstructural characteristics of the hot rolled materials were investigated by the optical microscope and TEM. The effect of processing route on cold deformation, recrystallization and texture will be discussed in detail.

11:30 AM INVITED PAPER

HEREDITY OF HOT ROLLING TEXTURE AND ITS INFLUENCE ON PROPERTIES OF ALUMINUM ALLOYS: *Y. Liu*[1]; J. G. Morris[1]; [1]University of Kentucky, Department of Chemical and Materials Engineering, 177 Anderson Hall, Lexington, KY 40506 USA

Heterogeneous texture distribution is often found in hot bands of aluminum alloys. This condition can be inherited in the following cold rolling operation or it can be changed to other forms of heterogeneity by annealing. The texture and its heterogeneity in initial hot bands may extend its effect to final gauge products. It is important to investigate the impact of this texture heterogeneity on the properties of aluminum alloys. In the present work, direct chill (DC) cast AA6010 hot band was used. Various textures were produced by multipass cold rolling. The influence of the initial hot rolling texture on the preferred orientation evolution during cold rolling was examined throughly. The r-value and yield strength of the cold rolled samples were determined and their relationship to textures are discussed. Similar work was also performed on hot bands on DC and strip cast (SC) AA5182 alloys. The results are compared with those for AA6010 alloy with the regard to texture. Different hot rolling textures are identified in DC and SC hot bands. It was found that the trace of hot rolling texture and its heterogeneity can be detected in samples with reductions up to 70%. The properties of those alloys are strongly affected by the initial hot rolling texture before cold rolling and also influenced by the method of casting (DC or SC). The properties of samples with heterogeneous texture distribution from surface to the center layer are not simply determined by the average effect of textures.

INTERSTITIAL AND SUBSTITUTIONAL SOLUTE EFFECTS IN INTERMETALLICS II: Iron Aluminides

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee

Program Organizers: Ian Baker, Dartmouth College, Thayer School of Engineering, Hanover, NH 03755 USA; Easo P. George, Oak Ridge National Lab., Metals & Ceramics Division, Oak Ridge, TN 37831-6093 USA; Ronald D. Noebe, NASA Lewis Research Center, Cleveland, OH 44135 USA

Monday AM Room: Madrid
October 12, 1998 Location: O'Hare Hilton Hotel

Session Chairs: R. Zee, Auburn University, Materials Engineering Program, Auburn, AL 36849 USA; P. R. Munroe, University of New South Wales, Materials Science and Engineering NSW 2052 Australia

8:30 AM

EFFECT OF DEVIATION FROM STOICHIOMETRY AND ALLOYING ON DISLOCATION PROCESSES AND DEFORMATION BEHAVIOR IN B2 INTERMETALLIC COMPOUNDS: *Michael J. Mills*[1]; M. F. Savage[1]; R. Srinivasan[1]; R. D. Noebe[2]; T. Lograsso[3]; M. S. Daw[4]; [1]The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; [2]NASA, Lewis Research Center, Cleveland, OH 44135 USA; [3]Ames Laboratory, 111A Metals Development, Ames, IA 50011 USA; [4]Clemson University, Department of Physics and Astronomy, Clemson, SC 29634 USA

The relatively simple crystal structure of B2 intermetallics compounds such as FeAl and NiAl belies the fact that they exhibit a wide range of mechanical behaviors. These diverse characteristics may arise upon deviation from stoichiometry, particularly in both NiAl and FeAl systems which have wide B2 phase fields, as well as the presence of low-level impurities or the addition of ternary alloying additions. These compositional effects are often far more profound and complex than those anticipated from classical solute-dislocation interaction theories. In this presentation, we will present several examples of the dramatic effects of both stoichiometry and alloying on dislocation structure and deformation behavior in B2 compounds. As one example, we will contrast the behavior of Ni-50Al with Ni-44Al as a function of temperature ≤ particularly for [001] oriented single crystals. The effect of alloying additions, such as Hf, on the dislocation structures and properties of NiAl-based alloys will also be discussed. As a third example, the (Fe,Ni)-Al pseudobinary system offers an opportunity to explore the effect of Fe/Ni ratio while remaining within the ordered B2 phase field. In this way, the properties of substitutional alloys bridging the behavior from FeAl to NiAl may be investigated. Single crystals with a wide range of compositions have been grown, and the results of experiments and microstructure analysis following compression testing of <001> oriented single crystals with a wide range of Fe/Ni ratio will be presented. This research supported by the U.S. Department of Energy under contract no. DE-FG02-96ER45550 (for MFS, RS and MJM) and by the National Science Foundation under grant no. 95-10259 (for MSD).

9:00 AM

VACANCY-SOLUTE INTERACTIONS IN FeAl-BASED ALLOYS: *P. R. Munroe*[1]; [1]University of New South Wales, Materials Science and Engineering, Sydney, NSW 2052 Australia

It is now well established that high concentrations of thermal vacancies are retained in FeAl following high temperature heat treatments, and these act as powerful hardening agents. These point defects must be removed from the lattice, through prolonged low temperature heat treatment, if the intrinsic behaviour of this compound is to be understood. However, it has been shown that ternary additions to FeAl, both interstitial and substitutional, significantly affect the vacancy hardening behaviour of this material. For example, nickel additions, not only significantly slow the kinetics of vacancy removal, but also act as powerful hardening agents. The mechanisms by which nickel affects thermal vacancy behaviour are not understood. The aim of the this paper will be to describe the effects of vacancy-solute interactions on thermal vacancy hardening and discuss the possible mechanisms through which ternary additions can interact with thermal vacancies.

9:30 AM

SOLID SOLUTION STRENGTHENING OF B2 IRON ALUMINIDES BY THERMAL VACANCIES AND NICKEL: *J. H. Schneibel*[1]; L. M. Pike[1]; [1]Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

The room temperature strength of B2 iron aluminides containing 45 at. % Al and up to 10 at. % Ni was investigated after anneals at temperatures ranging from 673 to 1273 K. Care was taken to ensure that thermal vacancy equilibrium was reached. At 673 K, this required long anneals approaching one year. Vacancy concentrations were determined by density and lattice parameter measurements with reproducibilites of up to 1 part in 104. For a given Ni concentration, the strength was found to be proportional to the square root of the vacancy concentration. However, the hardening rate due to vacancies decreased as the Ni content increased, i.e., in the presence of Ni the vacancies became less effective hardeners. This gave rise to surprising effects such as solid solution softening by Ni. This research was sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract number DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

10:00 AM

BORON STRENGTHENING IN FeAl: *Ian Baker*[1]; X. Li[1]; H. Xiao[1]; O. Klein[1]; C. Nelson[1]; R. L. Carleton[2]; E. P. George[2]; [1]Thayer School of Engineering, Dartmouth College, Hanover, NH 03755 USA; [2]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831 USA

The effect of boron on the strength of B2-structured FeAl is considered as a function of composition, grain size and temperature. Boron does not affect the concentrations of anti-site atoms or vacancies present, with the former increasing and the latter decreasing with increasing deviation from the stoichiometric composition. When vacancies are absent, the strength increase per at. % B per unit lattice strain, Ds/(Dc x e) increases with increasing aluminum concentration, but when vacancies are present (> 45 at. % Al), Ds/(Dc x e) decreases again. Boron increases grain size strengthening in FeAl. B strengthening is roughly independent of temperature up to the yield strength peak but above the point, when diffusion-assisted deformation occurs, boron strengthening increases dramatically.

10:30 AM

PLASTIC FLOW INSTABILITIES IN Fe-RICH B2 FeAl: *K. Yoshimi*[1]; M. H. Yoo[1]; S. Hanada[2]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA; [2]Tohoku University, Institute for Materials Research, Sendai, 980-77 Japan

In Fe-rich B2 FeAl, plastic flow instabilities have been observed for the first time at room temperature. Plastic deformation with serrations occurs at room temperature in B2 FeAl single crystals fast-cooled from 1173 K. This serrated flow behavior is attributed to a supersaturation of vacancies retained by fast cooling. On the other hand, when the excess vacancies are eliminated, the B2 FeAl shows yield stress anomaly and also serrated flow up to the peak temperature. This is attributable to the increase in equilibrium concentration of thermal vacancies as temperature is raised. TEM deformation microstructure of the specimens strained at elevated temperatures is distinctly different from that of fast-cooled specimens strained at room temperature. Experimental results of both types of the flow instabilities in B2 FeAl will be discussed in terms of the change of dislocation-core structure with the absorption of vacancies and the dislocation-vacancies by-pass mechanism.

11:00 AM

HIGH TEMPERATURE INTERNAL FRICTION MEASUREMENTS ON Fe- AND Ni-ALUMINIDES: *W Herman*[1]; H.-G. Sockel[2]; [1]Siemens AG, Power Generation (KWU), 45466 Mülheim a.d. Ruhr, F.R. Germany; [2]Universität Erlangen-Nürnberg, Institut für Werkstoffwissenschaften, LS I, Martensstr. 5, 91058, Erlangen, F.R. Germany

In this work the elastic and anelastic behavior of two iron-aluminides ($Fe_{70}Al_{30}$, $Fe_{75}Al_{20}Si_5$) and two nickel-aluminides (Ni_3Al, NiAl) were investigated at elevated temperatures by a free-free beam vibration technique. The results of the internal friction investigations of the iron-aluminides lead to very interesting spectra exhibiting several clear maxima. These peaks are related to discontinuous changes in the temperature course of the elastic properties and can be assigned to ($D0_3$ <—> B2; B2 <—> A2), the movement and breakaway of dislocations and stress-induced changes in order. Internal friction measurements on the L12-ordered intermetallic Ni3Al reveal two different maxima between 1000ΥC and 1200ΥC, related to disordering processes due to the diffusion of Ni- and Al-atoms. These results are in good agreement with investigations on high γ'-[Ni_3(Al,Ti)]-content Ni-base superalloys. In B2 -ordered NiAl a strong exponential increase of the internal friction was observed at high temperatures, due to grain boundary effects.

11:30 AM

EFFECTS OF INTERSTITIAL BORON AND ALLOY STOICHIOMETRY ON ENVIRONMENTAL EFFECTS IN FeAl: *J. W. Cohron*[1]; E. P. George[1]; R. H. Zee[2]; [1]Oak Ridge National Laboratory, Metals and Ceramic Division, Oak Ridge, TN 37831-6093 USA; [2]Auburn University, Materials Engineering Program, Auburn, AL 36849 USA

Tensile tests were conducted on B-doped (300 wppm) and B-free polycrystalline FeAl alloys containing 37, 40, 45 and 48 at. % aluminum at room temperature (UHV to ~10^3 Pa). Intrinsic ductility, obtained in UHV, was found to decrease in both B-free and B-doped FeAl with increasing Al content. Fracture mode was independent of environment and dependent mainly on stoichiometry. Ductility was found to be very sensitive to environment, particularly in the lower Al alloys, independent of the dominant fracture mode. Alloys that exhibited >10 % ductility in UHV showed a decrease in elongation to fracture with increasing hydrogen pressure. Tests conducted in dry hydrogen gas result in greater ductilities than those conducted in equivalent moist environments, indicating that H_2O is more detrimental than H_2 to the ductility of FeAl alloys. Research sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

MECHANICAL PERFORMANCE OF LAMINATED COMPOSITES: Fracture and Fatigue Behavior

Sponsored by: Structural Materials Division, Structural Materials Committee

Program Organizers: Donald Lesuer, Lawrence Livermore Natl. Lab., Livermore, CA 94551 USA; Eric M. Taleff, The University of Texas, ASE/EM CO600, Austin, TX 78712

Monday AM Room: Paris C
October 12, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Donald R. Lesuer, Lawerence Livermore National Laboratory, Livermore, CA 94551 USA; Eric Taleff, Dept. of Aerospace Engineering and Engineering Mechanics, Austin, TX 78712 USA

8:30 AM INVITED PAPER

FRACTURE AND FATIGUE-CRACK GROWTH IN Nb-REINFORCED Nb3Al LAMINATED COMPOSITES: D. R. Bloyer[1]; K. T. Venkateswara Rao[1]; *R. O. Ritchie*[1]; [1]Lawrence Berkeley National Laboratory, Materials Science Division, 1 Cyclotron Road, Berkeley, CA 94720 USA

A study has been made of the resistance-curve (R-curve) and fatigue-crack propagation properties of a series of laminated Nb reinforced Nb3Al intermetallic-matrix composites with varying microstructural scale, but nominally identical reinforcement volume fraction (20% Nb). Crack-growth resistance under both quasistatic and cyclic loads, was improved with increasing metallic layer thickness (in the range of 50 to 250 mm) and was superior, for a given layer thickness, in the crack-arrester rather than the crack-divider orientations. Such enhanced crack-growth resistance was found to result from both intrinsic and extrinsic mechanisms, in the form of, respectively, crack renucleation across the metal layers (arrester orientations only) and crack bridging via intact metal-layer ligaments in the crack wake. Unlike particulate-reinforced composites, the bridging was quite resilient under cyclic loads. R-curve modeling using weight-function methods permitted the determination of simple approximation, for the bridging tractions, which were, then used to make small-scale bridging predictions for the steady-state toughness and fatigue-crack growth resistance of each laminate.

9:00 AM INVITED PAPER

MECHANICAL BEHAVIOR OF Nb-Si IN-SITU COMPOSITES: *J. J. Lewandowski*[1]; S. Soly'vev[1]; W. A. Zinsser[1]; [1]Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA

The fatigue and fracture behavior of in-situ composites based on the Nb-Si system and possessing a layered structure will be reviewed. The effects of loading rate on the fracture toughness of a cast/extruded and heat treated binary Nb-10Si alloy will be covered first, followed by the fatigue crack growth behavior of the same materials tested over a range of R-ratios and cyclic stress intensities. Extensive fractographic analyses have been conducted and it has been demonstrated that there are significant effects of changes in the above variables on the fracture and fatigue behavior of the Nb constituents in such materials. The presentation will conclude with very recent work where the fatigue behavior of highly alloyed variants of such materials have been conducted.

9:30 AM INVITED PAPER

CRACK PROPAGATION IN A LAYERED FUNCTIONALLY GRADED Ti/TiB COMPOSITE: R. D. Carpenter[1]; W. W. Liang[2]; G. H. Paulino[2]; Z. A. Munir[1]; *Jeffery C. Gibeling*[1]; [1]University of California, Department of Chemical Engineering and Materials Science, One Shields Avenue, Davis, CA 95616 USA; [2]University of California, Department of Civil and Environmental Engineering, One Shields Avenue, Davis, CA 95616 USA

The mechanisms and mechanics of crack propagation are examined in a layered functionally graded material composed of Ti and TiB phases. This material consists of seven layers ranging in composition from commercially pure Ti to 15Ti/85TiB by volume. Single edge notch bend (SENB) fracture specimens are oriented such that cracks propagate in the direction of the composition gradient (or crack arrestor orientation) from the Ti-rich layer to the TiB rich layer. For this orientation, the crack initiation resistance (KC) and the "average" R-curve response are investigated under 4-point loading. Crack propagation mechanisms are examined using scanning electron microscopy, with an emphasis of the interaction of the growing crack with the interfaces. In addition to the measured fracture results, the functionally graded beam is analyzed using a two-dimensional finite element model under plane stress loading. The materials are assumed to exhibit elastoplastic behavior, and the properties for each composition layer are estimated using a rule of mixtures approach. Characteristic response quantities are investigated at several stages of crack growth represented by crack length and crack opening displacement (COD). These analytical results are compared to the measured fracture parameters.

9:50 AM BREAK

10:10 AM INVITED PAPER

EFFECTS OF 6013 LAYER VOLUME FRACTION AND THICKNESS ON FRACTURE OF 6013//6090/SIC/25P LAMINATES: *J. D. Rigney*[1]; R. W. Bush[1]; J. Teply[1]; J. J. Lewandowski[2]; C. K. Syn[3]; D. R. Lesuer[3]; [1]Alcoa Technical Center, (Now at General Electric Aircraft Engines, Cincinnati, OH 45215-6301), Alcoa Center, PA 15069 USA; [2]Case Western University, Cleveland, OH 44106 USA; [3]Lawerence Livermore National Laboratory, Livermore, CA 94551 USA

Discontinuously reinforced aluminum (DRA) materials provide attractive combinations of specific stiffness and specific compressive strength, and fatigue resistance compared to conventional aluminum alloys. Unfortunately, additions of SiC reinforcement reduce the fracture toughness of these materials limiting their use in widespread structural applications. Recent work has investigated laminated macrostructures that provide both the beneficial properties of DRA composites while enhancing the damage tolerance through extrinsic mechanisms. Laminates of AA6090/SiC/25p toughened with monolithic AA6013 were produced varying in either the component vol.%, laminate thickness and/or total laminate thickness. Tensile and compressive experiments for strength and elastic moduli, and compact tension tests for fracture toughness and R-curve behavior were used to characterize properties of the laminates after T6 heat treatment. Fracture surface examinations and quantification of the fracture micromechanisms (plastic strain, delamination, and crack opening displacement) within the bridged-zone of the toughness specimens were used to understand the role of the laminate design on the extrinsic toughening mechanisms. This presentation will highlight the effects of laminate architecture on the fracture behavior and models under development to predict the fracture properties.

10:30 AM

MODE MIXITY EFFECT ON INTERFACIAL TOUGHNESS IN CERAMIC/METAL LAYERED MATERIALS: *Scott X. Mao*[1]; [1]The University of Calgary, Mechanical Engineering Department Canada

To understand ductile and brittle cracking behavior and mechanism along the metal/ceramic interface in layered material is important in the design of electronic package, multilayered coatings and nano- or micro-structured composites. Experiments on interfacial cracking in micro-layered Alumina/Au/Alumina composite has been carried out. And mode mixity effect on interfacial toughness has been found. It has been found that micro-cracks or "voids" exist along the interface and act as debond sources for interfacial cracking. From the observation on the debond surface by atomic force microscope (AFM), it has been found that the micro-cracks propagate along the interface with cleavage and then are arrested by dislocation slip steps. The width of each slip step is 1 mm and height is ~10 nm. Based on the AFM observation, a super-dislocation model in metal/ceramic layered material has been proposed to study the mode mixity effect on interfacial fracture toughness.

10:50 AM

LAYER TOUGHENING OF MOLYBDENUM DISILICIDE COMPOSITES: F. Ye[1]; *W. O. Soboyejo*[1]; [1]The Ohio State University, Department of Materials Science and Engineering, 2041 College Street, Columbus, OH USA

This paper examines the mechanisms and mechanics of ductile layer toughening in molybdenum disilicide micro-laminates reinforced with niobium layers. Toughening mechanisms are elucidated for model composites deformed under monotonic or cyclic loading. Ductile layer toughening is attributed to the combined effects of large scale bridging and crack-tip/layer interactions. These are shown to promote resistance-curve behavior and "stable" fatigue crack growth. The superposition of crack bridging and transformation toughening is also explored in layered molybdenum disilicide composites reinforced with transforming yttria-stabilized zirconia particles and niobium layers. Finally, the paper examines the possible engineering of synergistic toughening by the promotion of interactions between crack bridging and transformation toughening.

11:10 AM

FATIGUE BEHAVIOR OF A NEXTEL| 312(BN)/BLACKGLAS| LOW COST LAMINATED CERAMIC COMPOSITE AT ROOM AND ELEVATED TEMPERATURES: *Wei Zhao*[1]; Peter K. Liaw[1]; Ronald Belardinelli[2]; David C. Joy[3]; Charlie R. Brooks[1]; N. Allen Yu[4]; Carl J. McHargue[1]; [1]University of Tennessee at Knoxville, Dept. of Materials Science and Engineering, Dougherty Engr. Bldg., 1512 Middle Dr., Knoxville, TN 37996-2200 USA; [2]Northrop Grumman, 9314 W.Jefferson Blvd., Dallas, TX 75211 USA; [3]University of Tennessee, Dept. of Biochemistry, Cellular and Molecullar Biology, 232 Sci. & Engr. Facilities, Knoxville, TN 37996 USA; [4]University of Tennessee, Dept. of Mechanical and Aerospace Engineering and Engineering Sciences, 323 Perkins Hall, Knoxville, TN 37996 USA

In this paper, fatigue behavior for a Nextel312(BN)/Blackglas, a newly developed advanced low cost laminated ceramic-matrix composite, is investigated. The composite preform is composed of layers of a crowfoot satin Nextel 312 ceramic fiber fabrics. The Blackglas ceramic matrix is then pyrolyzed in an argon inert atmosphere. The interface between the Nextel fiber and the Blackglas is a boron-nitride (BN) coating. A four-point bending fatigue test is performed using a Material Testing System (MTS) at room and elevated temperatures. An acoustic emission technique is applied to monitor the crack initiation and propagation behavior. Failure mechanisms are analyzed through examining microstructures, fiber/matrix interfaces, and fracture surfaces by a scanning electron microscopy (SEM) for as-received and oxidized samples. Variations of chemical compositions after oxidation are also detected by a SEM line-scan technique. The present work is supported by National Science Foundation under a contract number, EEC-9527527, with Mary Poats as the program manager.

MECHANISMS AND MECHANICS OF COMPOSITE FRACTURE: Fiber Reinforced Composites

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

Program Organizers: Bhaskar S. Majumdar, UES, Inc., Dayton, OH 45432-1894 USA; S. Krishnamurthy, Allied Signal, Inc., Phoenix, AZ 85034 USA; Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

Monday AM — Room: Paris A
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Session Chair: Frank Zok, University of California, Materials Department, Santa Barbara, CA 93106 USA

8:30 AM INVITED PAPER

INTERFACIAL FRACTURE OF CERAMIC FILMS-THE ROLE OF SUBSTRATE PROPERTIES: *N. R. Moody*[1]; [1]Sandia National Laboratories, Livermore, CA 94551 USA

Thin ceramic films are used in many applications where special properties are required for performance. Of particular interest is the fracture resistance of thin tantalum nitride films on aluminum oxide and aluminum nitride substrates. We have therefore combined nanoindentation and nanoscratch test results with mechanics-based models to determine the fracture properties of these hard films on both substrates. The tests showed that all films failed along the film-substrate interfaces at mode I fracture energies ranging from 1.3 to 3.8 J/m2. However, the fracture energies are nearly a factor of two higher on aluminum oxide than on aluminum nitride substrates even though the nitride substrates were covered with a surface oxide. Comparison of the results and TEM of substrate deformation showed that the difference was most likely due to the higher ductility of the nitride substrates. These results will be used to show how mechanics and materials approaches can be used to determine resistance to fracture of ceramic films and the importance of substrate properties on film durability. This work supported by U.S. DOE Contract DE-AC04-94AL85000

8:55 AM INVITED PAPER

ANALYTICAL FRACTURE MECHANICS OF THE MICROBOND TEST INCLUDING THE EFFECTS OF FRICTION AND THERMAL STRESSES: *John A. Nairn*[1]; [1]University of Utah, Material Science & Engineering Department, Salt Lake City, UT 497907-1282 USA

An energy release rate model based on a generalized fracture mechanics of composites was developed for analyzing the microbond test. This model, which extended a previous model, includes both friction at the fiber/matrix interface and residual thermal stresses. A series of microbond tests on macroscopic specimens were carried out for evaluating the model. In some specimens we could observe debond crack growth. These results could be interpreted with a fracture mechanics R-curve which lead to a measured interfacial fracture toughness. In many specimens, debond crack growth could not be observed. We developed an approximate method for determining interfacial fracture toughness even without knowledge of debond crack size. The macroscopic specimens were designed for studying the optimal approach to analysis of microbond specimens. The geometry of the macroscopic specimens, however, could also be used to measure the mode II toughness of adhesive bonds.

9:20 AM INVITED PAPER
INTERFACIAL CRACKS IN THE PRESENCE OF FRICTION: *C. T. Sun*[1]; [1]Purdue University, School of Aeronautics & Astronautics, West Lafayette, IN 47907-1282 USA

Under shear loading or combined compression and shear loading, the surfaces of an interfacial crack may contact and cause frictional stresses. Examples include fiber pullout in composite and delamination in layered media under transverse impact. The asymptotic analysis shows that the near tip stress singularity is no longer an inverse square root as in the frictionless case. The presence of friction will either strengthen or weaken the singularity. In this paper, a numerical study based on finite element simulations is carried out to investigate energy release rates in the presence of friction. To resolve the vanishing strain energy release rate problem, we propose an alternative fracture criterion using the strain energy release rate associated with a nonvanishing crack extension as a fracture parameter. This finite extension strain energy release rate quantifies the magnitude of the singular stress field and can be regarded as a representation of the fracture driving force.

9:45 AM INVITED PAPER
THE ROLE OF INTERFACES IN COMPOSITES: MICRO-MECHANICS ANALYSIS OF CRACKS: *Ming Y. He*[1]; [1]University of California, Department of Materials, Santa Barbara, CA USA

The dominant influence of interfaces on the mechanical performance of brittle matrix composites is addressed. Two related basic problems are reviewed: 1. The tendency of a matrix crack to either cause interface debonding or to propagate through the interface into the fiber. 2. The tendency of interface debonding to either remain in the interface or kink out. These solutions were utilized to design interface properties to ensure the composite will survive matrix-cracking. Furthermore, the criterion has also been used to measure the interface strength in fiber reinforced composites by an indentation-induced crack-deflection method. The modifications have been made to consider effects of the fiber width. Finally, the criterion has been extended to consider the tendency of a grain-boundary crack to either deflect along another grain boundary or kink out upon reaching a triple junction.

10:10 AM INVITED PAPER
THE ROLE OF PLASTICITY IN BIMATERIAL FRACTURE WITH DUCTILE INTERLAYERS: *W. W. Gerberich*[1]; W. Schildgen[1]; J. Robach[1]; A. Volinsky[1]; M. Kriese[1]; [1]University of Minnesota, Materials Science Department, 421 Washington Avenue, Minneapolis, MN 55455 USA

Key to understanding the role of plasticity in interfacial fracture is the measurement of interlayer constitutive properties. Thus, for a first order solution, one at a minimum needs the properties appropriate to the small volumes involved. With nanoindentation, one can obtain both a large strain flow stress as well as the far field yield stress representing the small strain elastic-plastic boundary. Using these to estimate an appropriate elastic-plastic strain energy density, the work per unit fracture area can be estimated for copper as an interlayer. These are compared to external measures as obtained from axisymmetric buckling theory. While the latter is an elasticity solution, values in the range of 2 to 100 J/m2 are considered to realistic due to the small volume of plasticity involved. Experimental comparisons on evaporated and/or annealed Cu thin film interlayers between sputtered tungsten and silicon are made. Here, residual stresses of the interlayer or overlayer range from 0.1 to 1 Gpa. It is not clear at this time how to incorporate residual stress into the dissipation side of the energy balance.

10:35 AM BREAK

11:00 AM INVITED PAPER
RECENT ADVANCES IN INTERFACE MEASUREMENTS IN TITANIUM MATRIX COMPOSITES: *Daniel B. Miracle*[1]; S. G. Warrier[2]; B. S. Majumdar[2]; [1]Air Force Research Laboratories, Materials and Manufacturing Directorate, WPAFB, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Zenia Rd., Dayton, OH 45432 USA

The magnitude of the properties of the interfacial region are critical in determining the response of fiber-reinforced titanium matrix composites (TMC's). Over the past decade, a number of test techniques have been employed to quantify the interfacial properties in titanium matrix composites. Results from different test techniques will be compared and discussed. The relevance of specific interface properties will be discussed.

11:25 AM INVITED PAPER
FUNDAMENTAL ASPECTS ON INTERFACIAL CRACKING MECHANICS AND MECHANISM IN CERAMIC/METAL LAYERED MATERIALS: *Scott X. Mao*[1]; [1]The University of Calgary, Mechanical Engineering Department, 2500 University Drive, Calgary T2N 1N4 Canada

To understand ductile and brittle cracking behaviour is important in the design of electronic packages, and nano- or micro-structured composites. Experiments on interfacial cracking in micro-layered Alumina/Au/Alumina composite has been carried out. It has been found that micro-cracks or "voids" exist along the interface and act as debond sources for interfacial cracking. When a main crack front approaches these interfacial micro-cracks, high peak stress is generated by the constrained metal layer. Using atomic force microscope AFM, it has been found that the micro-cracks propagate along the interface with cleavage manner and then are arrested by dislocation slip steps. The width of each slip step is 1 micrometer and height is ~10 nm. A super-dislocation model has been proposed to describe the crack tip stress and the interfacial fracture energy. Crack tip blunting due to dislocation emission has been analyzed. The effect of length scale (thickness) of the metal layer on interfacial cracking is discussed.

11:50 AM
EFFECTS OF CARBON ON THE ADHESION OF ALUMINUM FILMS TO SAPPHIRE SUBSTRATES: *J. A. Schneider*[1]; S. E. Guthrie[1]; M. D. Kriese[2]; W. M. Clift[1]; N. R. Moody[1]; [1]Sandia National Laboratories, Livermore, CA 94551 USA; [2]University of Minnesota, Dept. of Chemical Engineering and Materials Science, Minneapolis, MN 55455 USA

The adhesion of aluminum (Al) films onto sapphire substrates in the presence of controlled amounts of carbon contaminants is being investigated. In this study, adhesion strength is evaluated by continuous nanoindentation tests to induce delamination of the Al film from the sapphire substrate. To promote blistering, highly stressed overlayers of 1000 nm sputtered tantalum (Ta) were deposited on 500 nm thick Al films. With the Ta overlayers, continuous nanoindentation techniques induced larger diameter delamination blisters in the specimens with carbon, than in the specimens without carbon. Resistance to blistering, or smaller induced blisters, is indicative of a higher interfacial strength. Auger and XPS were used to analyze the interface fracture surfaces. *This work was supported by the U.S. Department of Energy under Contract #DE- AC04-94AL85000.

12:15 PM INVITED PAPER
INTERFACE FRACTURE AND SLIDING IN METALLIC SYSTEMS: *C. L. Briant*[1]; R. Phillips[1]; A. Wagoner[1]; [1]Brown University, Division of Engineering, Providence, RI 02912 USA

This paper will address fundamental issues in grain boundary and interface fracture and sliding. One of the primary purposes of this paper will be to relate these mechanical processes to the structure of grain boundaries and interfaces. We will begin with low temperature grain boundary fracture and discuss the importance of grain boundary segregation on this process. Data will be drawn from both polycrystalline studies and bi-crystal studies to demonstrate that there is a relationship between segregation and structure and thus fracture and structure. Theoretical results will also be presented in which the quasi-continuum method is applied to this problem. The calculations demonstrate the response of the boundary to stress and show how dislocations are generated from the boundary as the crack approaches. We will then make comparisons with this type of fracture and separation of interfaces in composites and functionally graded materials. Another topic discussed in this presentation will be grain boundary sliding at high temperatures. Results will be presented for grain boundary sliding in aluminum and again compared with calculations performed by the quasi-continuum method for the same boundaries. Comparisons will be made between this type of failure and the behavior of other types of interfaces at elevated temperatures.

NEW & EMERGING APPLICATIONS FOR REFRACTORY METALS & MATERIALS: Refractory Metals and Alloys

Sponsored by: Structural Materials Division, Refractory Metals Committee

Program Organizers: Joseph W. Newkirk, Univ of Missouri-Rolla, Dept of Metallurgical Engineering, Rolla, MO 65409-0340; Edward N.C. Dalder, Lawrence Livermore National Lab, PO Box #808, Livermore, CA 94551-0808

Monday AM — Room: Dublin
October 12, 1998 — Location: O'Hare Hilton Hotel

Session Chair: Joseph W. Newkirk, U. of Missouri-Rolla, Dept. of Metallurgical Eng., Rolla, Missouri 65409 USA

8:30 AM
VANADIUM-BASE ALLOYS FOR MAGNETIC FUSION APPLICATIONS: *Dale L. Smith*[1]; [1]Argonne National Laboratory, Fusion Power Program, 9700 S. Cass Ave., Argonne, IL 60439 USA

Vanadium-base alloys have been identified as a leading candidate low-activation structural material for fusion first-wall blanket applications. Candidate vanadium alloys exhibit favorable safety and environmental characteristics, good fabricability, high temperature and heat load capability, good compatibility with liquid metals and resistance to irradiation damage. The focus of the vanadium alloy development program has been on the vanadium-chromium-titanium (0-15% Cr, 1-20% Ti) alloy system. A V-4Cr-4Ti alloy is currently identified as the reference alloy. Significant progress has been made in the development of vanadium alloys for fusion applications. This paper will present an update on results of fabrication, mechanical properties, corrosion/compatibility, and effects of irradiation on the properties of vanadium alloys for fusion power applications.

9:00 AM
TUNGSTEN: A LEADING PLASMA FACING COMPONENT MATERIAL FOR FUSION ENERGY SYSTEMS APPLICATIONS: *Suri Sastri*[1]; [1]Surmet Corporation, 33 B St., Burlington, MA 01803 USA

Plasma Facing Components in a fusion reactor must survive high heat fluxes, plasma induced sputter erosion and heavy doses of neutron flux. Because of its high melting point, relatively good thermal conductivity and excellent resistance to sputter erosion, tungsten metal has emerged as one of the best choices for Plasma Facing Component (PFC) material in fusion energy systems applications. Extremely dependable bonding of the plasma facing armor to the copper cooling structure must be developed before its use in a fusion energy system. The structure must also be designed to accommodate major plasma disruptions characterized by possible vertical displacement and quenching of the entire plasma energy on the chamber walls in less than 1 ms. The problems associated with fabricating such a reliable structure will be discussed in detail. Due to neutron transmutation effects conventional joining processes which involve the use of noble metals such as silver and gold cannot be used to fabricate such a structure. Novel alternate processing routes for fabricating tungsten copper structures using powder metallurgy approach along with an innovative method of fabricating a classical 'brush like' structure will be presented.

9:20 AM
REFRACTORY METALS IN AMTEC SYSTEMS: *Robert C. Svedberg*[1]; [1]AMPS, Materials Engineering, 4667 Freedom Drive, Ann Arbor, MI 48108 USA

AMTEC (Alkali Metal Thermal to Electric Conversion) systems are being developed to provide power for space craft with missions to Pluto and Jupiter. These AMTEC cells are constructed with refractory metal and alloy components. Nb-1Zr is used for the external structure, Mo for the wick and current collector structures, and Ta alloys for high creep resistant fastener applications. Na is the working fluid in the AMTEC system. Beta"-alumina solid electrolytes (BASE) and TiN electrodes are the electrochemical components in the cell. Unique fabrication, processing and handling procedures required to produce AMTEC cells are described. Methods used to test and evaluate cell components in the Na and vacuum exposure environments are reviewed. Models and techniques that will be used to verify system life will also be discussed.

9:40 AM
TEXTURE DEVELOPMENT DURING ROLLING AND DRAWING OF TANTALUM: *Clyde L. Briant*[1]; [1]Brown University, Division of Engineering, Box D, Providence, RI 02912 USA

This paper will report a study of the development of texture in both rolled and drawn tantalum products. The measurements were made using electron back-scattering diffraction pattern analysis (EBSP) in the scanning electron microscope. The ingot was initially prepared by vacuum arc remelting, which produced large columnar grains in the material. After this structure was deformed by a combination of forging and rolling, it was found that a strong <110>{001} texture formed in the bands in the material. Between these bands of strong texture, a much more random distribution of orientations was observed. Further rolling and annealing produced a texture in the rolled product that was a combination of <110>{001}, <110>{111}, and <110>{112}. Annealing of rolled samples produced a texture in which the <111> direction was parallel to the normal to the rolling plane. Drawing of tantalum produced a texture in which the <110> direction was parallel to the drawing direction.

10:00 AM
THE MECHANICAL PROPERTIES OF HIGH PURITY CHROMIUM: *Clyde L. Briant*[1]; [1]Brown University, Division of Engineering, Box D, Providence, RI 02912 USA

This paper will report results on the mechanical properties of high purity chromium. In the initial study, powders of high purity chromium were compacted to form a polycrystalline sample. We found that during the hot pressing step interstitials were absorbed into the chromium and the result was that all samples behaved similarly in both compression tests and bend ductility tests. The ductile to brittle transition temperature was above room temperature. A single crystal of high purity chromium showed excellent ductility in bending at room temperature, even when the sample was notched. We will discuss the relative importance of interstitials versus the presence of grain boundaries in causing this improvement. Preliminary investigations of the corrosion resistance of these materials will also be presented.

10:20 AM
EFFECTS OF TI ADDITION ON CLEAVAGE FRACTURE IN Nb-Cr-Ti SOLID SOLUTION ALLOYS: *Kwai S. Chan*[1]; David L. Davidson[1]; [1]Southwest Research Institute, Materials Engineering De-

partment, 6220 Culebra Road, P.O. Drawer 28510, San Antonio, TX 78228-0510 USA

The fracture toughness of Nb-Cr-Ti solid solution alloys have been shown to be greatly improved by Ti addition, but the mechanism of toughness enhancement has not been established. In this study, critical experiments were performed on the tough Nb-Cr-Ti alloy to investigate the origin of fracture toughness. In addition, theoretical calculations of the unstable stacking energy (USE) and the Peierls-Nabarro (P-N) energy and stress were performed. The experimental results indicate that the fracture toughness in the tough Nb-Cr-Ti alloy originates from extensive crack-tip slip emission that suppresses cleavage crack propagation from the crack tip. The theoretical calculation indicates that Ti addition lowers the P-N energy and stress, but has little effect on the unstable stacking energy. These results are used to elucidate the effects of Ti addition on cleavage fracture in Nb-Cr-Ti alloys by considering the influence of the P-N energy and stress values on (1) crack-tip slip emission, (2) dislocation mobility, (3) fracture toughness, and (4) brittle to ductile fracture transition.

10:40 AM

AN EVALUATION OF Ti-44%NB AEROSPACE ALLOYS: *D. Scott MacKenzie*[1]; [1]University of Missouri-Rolla, Dept. of Metallurgical Eng., B-19 McNutt Hall, Rolla, MI 65409 USA

Ti-44%Nb rivets are extensively used in aerospace applications because of their ductility. There has been very little published in the literature regarding the microstructure of this alloy for this application. Since this alloy is used in rivets used near jet engines, there is a concern regarding the possible loss of strength during high temperature excursions. A study of the recrystallization of this alloy was conducted at elevated temperatures. An evaluation by optical metallography and x-ray diffraction of the microstructural development of Ti-44%Nb will also be presented.

11:00 AM

SYNTHESIS AND FABRICATION OF Mo-W COMPONENTS FOR NEUTRON RESONANCE TEMPERATURE MEASUREMENT: *Sherri R. Bingert*[1]; [1]Los Alamos National Laboratory, Materials Science & Technology Div., P.O. Box 1663, Mail Stop G770, Los Alamos, NM 87545 USA

A $Mo-W^{182}$ alloy was specified for an application that would ultimately result in the measurement of temperature during the steady state time following the shock loading of various materials. The W^{182} isotope provides a tag for the analysis of neutron resonance line shape from which the temperature may be calculated. The material was specified to have 1.8 atom percent W, with W rich regions no larger than 1 micron in size. Both the composition and W distribution are critical to the experiment. Three synthesis and processing routes have been explored to fabricate the required alloy components. First, mechanical alloying (MA) followed by hot isostatic pressing (HIP) and warm forging was attempted. More recently, parallel paths using arc melting techniques and solution co-precipitation of the powders have been pursued. Following arc melting, the button must be warm worked (forged and rolled) into a form from which discs may be machined. The co-precipitated powders must be hydrogen reduced prior to consolidation and warm forging. The results of the processing routes and complete characterization of the materials produced will be presented.

11:20 AM

REFRACTORY METAL-CONTAINING COMPOSITES FOR REPLACING LEAD IN SMALL CALIBER BULLETS: *Richard A. Lowden*[1]; [1]Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831 USA

The firing of small arms ammunition for training, sporting, law enforcement, and military purposes is a major source of environmental pollution and poses a health hazard. Large quantities of lead have been and continue to be deposited at the numerous public, private, and government-operated indoor and outdoor shooting ranges. Significant reductions in environmental pollution and human exposure can be achieved through the development of ammunition projectiles, which are composed, of materials that are not environmental or health hazards. "Composite simulants" for lead are being explored to replace the lead in small caliber bullets. High-density metals such as tungsten are mixed with lighter, softer metals or polymers to produce components with controllable physical and mechanical properties. Bullets can be formed directly to shape, or cores can be produced that can be swaged into projectiles, with or without jacketing. Bullets that are a one-to-one replacement for their lead analogs have been developed and tested. Internal and external ballistics are identical to current bullets for the density of the core materials can be tailored to exactly match that of lead or any lead alloy. The powder metallurgy composites are top candidates in an interagency program dedicated to finding a functional replacement for lead in military ammunition. Results to date have shown that "green" bullets fabricated employing powder metal composites are viable, and could possibly be technologically superior to lead.

11:40 AM

WHY TANTULUM IS REPLACING GRAPHITE IN PHARMACEUTICAL CONDENSER APPLICATIONS: *Kurt Mosher*[1]; [1]H. C. Starck, 45 Industrial Place, Newton, MA 02164 USA

The pharmaceutical industry has traditionally used resin-impregnated graphite block heat exchangers for handling corrosive fluids. During recent years, tantalum shell and tube condensers have emerged as a versatile alternative offering superior corrosion resistance. The advantages of tantalum over graphite in pharmaceutical processing include: reduced downtime for repairs and replacements; insuring the purity of the pharmaceutical end product; and the versatility to process any pharmaceutical end product using the same equipment, while meeting the strictest regulatory standards. Corrosion rates and life cycle comparisons demonstrate why this refractory metal is often a preferred construction material in highly corrosive environments.

PLASMA CASE HARDENING & RELATED PROCESSES FOR SURFACE MODIFICATION: Session I

Sponsored by: Materials Design and Manufacturing Division, Surface Modification & Coatings Technology Committee
Program Organizers: Krassimir Marchev, Barnett Institute, Northeastern University, Boston, MA 02115 USA; Clark V. Cooper, United Technologies Research Ctr, East Hartford, CT 02618 USA; Bill Giessen, Barnett Institute, Northeastern University, Boston, MA 02115 USA; Osman T. Inal, New Mexico Inst of Mining & Tech, Matl & Met Dept., Socorro, NM 87801 USA; Tali Spalvins, NASA, Lewis Research Center, Cleveland, OH 44135-3191 USA; Kumar Sridharan, University of Wisconsin, Madison, WI 53706 USA

Monday AM — Room: London
October 12, 1998 — Location: O'Hare Hilton Hotel

Session Chairs: Krassimir Marchev, Northeastern University, Barnett Institute, Boston, MA 02115 USA; Clark V. Cooper, United Technologies Research Ctr., East Hartford, CT 02618 USA

8:30 AM

A REVIEW OF ADVANCES IN ION NITRIDING OVER THE PAST 20 YEARS: *William L. Kovacs*[1]; [1]Elatec Technology Corporation, Wilmington, MA 01887 USA

Ion Nitriding is perceived to be a new process, yet it actually was discovered in the late "20's". The process has advanced in sporadic bursts, with vastly different levels of introduction and acceptance in different countries, and in different industries within these countries. Equipment has also improved. We now have Pulsed Inverter D.C. power supplies. Auxiliary Heating, Automatic Gas Mixing, computerized H.R.C. Detection and Arc Suppression Conforming Anodes, Center Anodes, and multiple purpose process furnaces are among the hardware innovations. Multiple processes with Ion Nitriding as diffusion based process, covered by a surface coating such as titanium nitride or diamond-like amorphous coatings are providing remarkable wear properties. The science of the plasma process itself is also becoming understood and will be reviewed. An updated list of companies providing treatment services for a fee will also be supplied.

8:50 AM

EPITAXIAL TEXTURES ON PLASMA NITRIDED 316L STAINLESS STEELS: *K Marchev*[1]; M. Landis[2]; R. Vallerio[2]; C. V. Cooper[3]; B. C. Giessen[2]; [1]Northeaestern University, Barnett Institute, Boston, MA 02115 USA; [2]Northeastern University, Chemistry Department, Boston, MA 02115 USA; [3]United Technologies Research Center, East Hartford, CT 06108 USA

In our earlier work on the formation of a nitrogen-rich single-phase compound layer (m phase) in 31 6L stainless steel we described its gradually changing nitrogen concentration, lattice parameter and microhardness characteristics, and most recently, the observation from pole besides the of strong preferred orientation in the (m phase layer) obtained figure studies that show in-surface orientational anisotropy more readily expected normal-to-surface anisotropy. These anisotropy's have now been identified as reflecting the underlying rolling textures of the steels and are due to epitaxial growth of m on and into the steel {111} fiber texture and {200} cube textures of the steel have propagated into related m phase textures despite the strong tetragonality of m. Further studies on steels with more pronounced and different rolling textures and the development of m phase textures on them are reported here. The existence of 3-D anisotropy significance and is therefore an parameter in ion nitriding.in nitrided layers may have practical important, previously not considered.

9:10 AM

INVESTIGATION OF GLOW DISCHARGE CONDITIONS IN PLASMA ASSISTED BORIDING PROCESS OF STEEL SUBSTRATES USING BCl_3-H_2-Ar ATMOSPHERE: *E. Rodriguez Cabo*[1]; K. T. Rie[2]; [1]Volkswagen AG, Zentrallabor, Wolfsburg Germany; [2]Institut fur Oberflächentechnik and Plasmatechnische Werkstoffentwicklung, TU-Braunschweig Germany

Boriding is a technique of thermo- chemical treatment that permits producing boride layers to prevent different metallic materials against abrasive and adhesive wear. The plasma bonding process using a gaseous atmosphere has several advantages compared to conventional techniques that utilize a solid medium like powder or paste. These advantages result in minimizing the manual work degree, a possibility of process control and automation and a reducing of boron containing residues that have to be disposed. The use of BCl3 as precursor in a plasma bonding process requires a parameter study to avoid corrosive reactions of the chlorine with the substrate material. This work examines the influence of the glow discharge in order to suppress the pore formation during bonding. The investigations are carried out by optical emission spectroscopy, analyzing different active species in the plasma, and show the possibility to produce compact boride layers without pores on steel substrates by plasma diffusion treatment.

9:30 AM

DIFFUSION OF BORON IN IRON: A COMPARISON OF TRADITIONAL BORIDING TECHNIQUE TO ION IMPLANTATION BORIDING: *J. A. Davies*[1]; P. J. Wilbur[2]; D. L. Williamson[3]; R. Wei[4]; J. J. Vajo[4]; [1]Implant Sciences Corporation, Wakefield, MA 01880 USA; [2]Colorado State University, Department of Mechanical Engineering, Fort Collins, CO 80523 USA; [3]Colorado School of Mines, Physics Department, Golden, CO 80401 USA; [4]Hughes Research Laboratories, Malibu, CA 90625 USA

The speed of boron diffusion during bonding is an essential element in controlling the growth rate of the boride layer and thus the speed of processing. A comparison of diffusion rates of boron through iron for traditional pack, bath and plasma bonding methods to the recently established ion implantation bonding method, which will be reviewed, is given. The diffusion during ion implantation bonding is found to be slower than during traditional bonding. The average diffusion activation energy and pre-exponential factor for traditional bonding techniques between 1000 and 500ΥC are 1.21 eV/atom and 2.5 x 10^{-3} cm^2s, while ion implantation boriding yields 0.76 eV/atom and 7.0 x 10^{-8} cm^2s for the same temperature range. The effect of boron arrival rate to the iron surface during boriding is discussed.

9:50 AM

PLASMA ASSISTED BORIDING OF COBALT-BASED ALLOYS: *S. Biemer*[1]; J. Olfe[1]; K. -T. Rie[1]; [1]Institut fur Oberflächentechnik und Plasmatechnische Werkstoffentwicklung, TU Braunschweig, Germany

Boriding is used as a means of increasing the wear resistance and hardness of many materials. In industrial application this process is mostly carried out using powders. In this work plasma-boroding has been carried out in a BCl3-H2 -Ar pulsed DC-glow discharge. Stellite 6B, a cobalt-base alloy has been treated at temperatures between 800 and 900ΥC for several hours. After the treatment the Co-base alloy has been analyzed by XRD, SEM and ESCA. Hardness profiles were measured. Cross sections of plasma-bonded cobalt-base alloy substrates shows a compound layer without the pores in the transition area to the base material which are often observed after plasma-bonding of steels. XRD-analysis of the surface indicates the formation of CoB and CrB. The maximum hardness of the compound layer was 2850 IIK 0.01 and remains constant in the whole compound layer. It is shown that plasma bonding is a promising tool for increasing the hardness of cobalt-base alloys.

10:10 AM BREAK

10:30 AM

PLASMA NITRIDING PROCESS CONTROLLING STRATEGY: COMPARISON OF PLASMA CURRENT VERSUS VOLTAGE: *T. Wu*[1]; [1]Diesel Technology Company, HT Business Unit, Kentwood, MI 49512 USA

In plasma nitriding process, plasma glow characteristics have been controlled by two strategies based on the Passion Curve: (1) current controlling voltage; (2) voltage controlling current. The advantages and disadvantages of each system are compared in this paper. Process stability and robustness are also evaluated.

10:50 AM

SURFACE PROPERTIES OF ION NITRIDED AUSTENITIC STEEL WITH SINGLE AND MULTIPHASE CASE STRUCTURES: K. Marchev[1]; *E. Rolinski*[2]; M. Landis[1]; R. Vallerio[1]; C. V. Cooper[3]; B. C. Giessen[1]; [1]Northeastern University, Barnett Insitute, Boston, MA 02115 USA; [2]Advanced Heat Treat Corp., Monroe, MI 48162 USA; [3]United Technologies Research Center, East Hartford, CT 06108 USA

The 300 series stainless steels can be nitrided in a broad range of temperature from approximately 750 to 1450ΥF. This creates opportunity of producing a very wide spectrum of the case structures and to study their effect on properties reported here. The m single phase structure and multiphase structures containing ε, γ, and CrN have been investigated. The potentiodynamic method has been used to determine the electrochemical behavior and the corrosion resistance of the layers. The corrosion resistance of the single-phase layer is superior to that of the multiphase layers; however, both types maintain very good properties in alkaline solution. Pin-on-disc testing has shown that tribological properties of single-phase layers are much better at small contact pressure but the multiphase layers have much greater load resistance. Nanoindentation tests performed on the surfaces and in cross section indicate that nitriding affects the Young's modulus of the steel primarily when the multilayer structure is formed.

11:10 AM
ION NITRIDING OF INCONEL: *Pramod Kotwal*[1]; Paul Nowil[1]; [1]Nitron Inc., Lowell, MA USA

Inconel is used in applications demanding high resistance to surface wear and toughness at elevated temperatures. It is known that its surface hardness and wear resistance is increased by nitriding. Gas nitriding is commonly used to nitride Inconel. Ion nitriding offers several technical and economic advantages which would be of interest to the designers and engineers interested in using Inconel. The thin and ductile nitride layers that ion nitriding can produce eliminates all post treatment grinding to remove the brittle and thick layers associated with other processes. In Inconel this can amount to very significant cost savings. This study investigates ion nitriding of Inconel. Samples of Inconel will be treated to a varied combination of time, temperature and gas composition. Resulting structures will be characterized and their properties will be investigated.

11:30 AM
INTENSIFIED PLASMA-ASSISTED PROCESSING: SCIENCE AND ENGINEERING: *E. I. Meletis*[1]; [1]Louisiana State University, Mechanical Engineering Dept., Baton Rouge, LA 70803 USA

Intensified Plasma-Assisted Processing (IPAP) is a surface modification technique developed in our laboratory. Plasma intensification and energetic flux bombardment of the work piece is accomplished by using a triode glow discharge. In our previous studies it was demonstrated that IPAP can achieve enhanced plasma nitriding kinetics at relatively low temperatures even in systems that are difficult or merely impossible to nitride with conventional means. IPAP employs high cathode currents at low pressures, allowing enhanced flow and controlled energies of the particles bombarding the work piece leading to formation of desired nitrides and deep diffusion zones. The paper will review our understanding of the fundamentals behind this technique and will present examples where significant improvements in the properties of engineering materials such as Ti-6Al-4V, 304 and 316 stainless steels, ductile cast iron and Al alloys have been achieved.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VII: Advanced Metallics and Intermetallics

Sponsored by: Structural Materials Division, Structural Materials Committee
Program Organizers: T. Srivatsan, Univ of Akron, Dept. of Mech. Engr., Akron, OH 44325-3903; K. A. Khor, Nanyang Technological University, Sch of Mech & Prod Engrg, Singapore 639798 Singapore

Monday AM	Room: Chicago
October 12, 1998	Location: O'Hare Hilton Hotel

Session Chairs: Dr. R. A. Varin, The University of Waterloo, Department of Mechanical Engineering, Waterloo, Ontario N2L3G1 Canada; Dr. S. K. Varma, The University of Texas at El Paso, Department of Metallurgical and Materials Engineering, El Paso, TX 79968-0520 USA

8:30 AM INVITED PAPER
INTERMETALLICS PROCESSED BY UNCONVENTIONAL METHODS OF COLD WORK: *R. A. Varin*[1]; J. Bystrzycki[1]; [1]The University of Waterloo, Department of Mechanical Engineering, Waterloo N2L 3G1 Canada

A grain refinement which is usually very beneficial for the improvement of mechanical properties of metal alloys is conventionally achieved by successive cold work and recrystallization. However, conventional cold work routes such as rolling, forging, extrusion etc. are difficult to be carried out for intermetallics which as a rule rather than an exception, are inherently brittle and develop sever microcracking even after application of a relatively light cold work. The present paper is an overview describing the results of our studies on the processing of cubic intermetallics by two unconventional methods resulting in a heavy cold deformation of intermetallics: (a) controlled mechanical (ball) milling and (b) shock-wave (explosive) deformation. Intermetallics cold deformed by these two methods underwent subsequent annealing in order to investigate recrystallization. Two intermetallics with cubic lattice structure were studied (a) $L1_2$ titanium trialuminides based on Al3Ti and (b) b2 FeAl with some alloying additions. The latter is known to be heavily strengthened by the mechanism of vacancy hardening. Microstructural evolution brought about by both methods of cold work and subsequent annealing as well as microhardness changes will be presented and discussed.

9:00 AM INVITED PAPER
EVOLUTION OF MICROSTRUCTURES DURING CYCLIC AND STATIC THERMAL STABILITY OF Ti-44Al-11Nb ALLOY: Alexander Oey[1]; R. Mahapatra[1]; *S. K. Varma*[1]; W. E. Frazier[2]; [1]The University of Texas at El Paso, Department of Metallurgical and Materials Engineering, El Paso, TX 79968-0520 USA; [2]Naval Air Warfare Center, Aircraft Division, Patuxent River, MD 20670 USA

A high temperature application of Taw-44Al-11Nb alloy requires that it should have microstructural stability. The aircraft materials go through thermal cyclic oxidation when used at height temperatures. A cyclic oxidation study has been conducted in air for this alloy when it undergoes heating for 55 minutes and then air cooled for 5 minutes which constitutes on thermal cycle. Static oxidation involves the continuous heating of the alloy in air. The microstructural features responsible for the changes in the oxidation characteristics evolved as a result of static and cyclic oxidation for up to 168 hours, or greater, at 900, 950 and 1000ΥC has been examined by SEM, TEM and optical microscopy. A correlation between the microstructures and oxidation behaviour has been made in order to understand this ternary system.

9:30 AM INVITED PAPER
PROCESSING OF INTERMETALLIC THIN FILMS: AN INDENTATION CRACKING STUDY: *M. Manoharan*[1]; G. Muralidharan[2]; [1]Nanyang Technological University, Division of Materials Engineering, School of Applied Science 639798 Singapore; [2]Institute of Microelectronics, Failure Analysis and Reliability, 11 Science Park Road, Science Park II 117685 Singapore

Thin metallic films are being used for a wide range of applications-from interconnects in microelectronics to coatings for wear resistance. Many studies have been conducted on the microstructural evolution and structure-property relationships in such thin films. When these films are exposed to temperatures where either bulk diffusion or short circuit diffusion occurs, interdiffusion as well as intermediate phase formations are to be expected. Ti and Al(Cu) have been cosputtered and sequentially sputtered on a Si substrate and the composite structure heat-treated at various temperatures. The variation in the mechanical properties of the composite film has been monitored using indentation fracture tests. These results along with its implications on processing of thin intermetallic films will be presented.

10:10 AM INVITED PAPER
PROCESSING AND CHARACTERIZATION OF Be-Al ALLOYS: *X. D. Zhang*[1]; V. Shah[1]; J. M. K. Wiezorek[1]; H. A. Lipsitt[1]; G. Meyrick[1]; H. L. Fraser[1]; F. C. Grensing[2]; [1]The Ohio State University, 2041

College Road, Columbus, OH 43210 USA; [2]Brush Wellman, Elmore, OH 43416 USA

Be-Al alloys, due to their high elastic modulus, low density and a relatively high melting point, are one of the most promising light weight materials for aerospace and high speed ground transport applications. It has bee reported recently that Be-Al alloys can be cast and extruded successfully, and exhibit reasonably promising mechanical properties. However there is lack of fundamental understanding of the physical metallurgy of these alloys, such as the formation of the cast microstructure which has previously been attributed to the existence of a submerged metastable monotectic reaction in high Be-Al compositions. In the present study we will present and discuss the results on the microstructural characterization on both as-cast and extruded alloys. The fracture and deformation mechanisms observed from tensile and compressive test samples will be reported and discussed in terms of the mobility and the nature of dislocations in Al and Be phase respectively. More recent work on the alloy development with improved processing process will also be presented. This work has been supported by a grant from the Office of Naval Research with Dr. Steven J. Fishman as program manager.

10:40 AM

EFFECTS OF OXYGEN ON SOLIDIFICATION BEHAVIOR IN THE Cu-Nb SYSTEM: *D. Li*[1]; M. B. Robinson[1]; T. J. Rathz[2]; G. Williams[2]; [1]NASA/Marshall Space Flight Center, Space Sciences Laboratory, Huntsville, AL 35899 USA; [2]University of Alabama, Huntsville, AL 35899 USA

At present there are two types of proposed Cu-Nb phase diagrams exhibiting an important discrepancy: a liquid immiscible characteristic and a flattened liquidus but not immiscibility. In this investigation, the melts of Cu-Nb alloys at compositions ranging from 5 to 86 wt pct Nb were processed in different environments and solidified at relatively low cooling rates of 50 to 75 “C/s to determine liquidus temperatures and to study solidification behavior. For all samples processed under very clean conditions, only Nb dendrites in a Cu matrix were observed; while in the presence of oxygen impurities the alloys containing 5 to 35 wt pct Nb exhibited microstructure of Nb-rich spheroids and Nb dendrites in the Cu matrix. The results obtained from clean conditions are in fair agreement with the Cu-Nb phase diagram having an S-shaped, near-horizontal appearance of the liquidus. The formation of Nb-rich droplets at slow cooling rates is discussed in terms of a stable liquid miscibility gap induced by oxygen.

11:00 AM

DYNAMIC FRACTURE OF ALUMINUM ALLOYS: INFLUENCE OF ALLOY CHEMISTRY AND AGING CONDITIONS: *T. S. Srivatsan*[1]; P. C. Lam[1]; M. Manoharan[2]; [1]The University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA; [2]Nanyang Technological University, Division of Materials Engineering, School of Applied Science 639798 Singapore

The dynamic fracture of aluminum alloys is of interest for a number of structural applications. The fracture behavior of these age hardenable alloys is influenced by the alloy chemistry, the aging behavior as well as the nature and types of notches involved. The microstructure influences not only the overall fracture toughness of the alloy but the local crack path as well. The effects of the microstructure on the stress field at the crack tip could manifest itself in the form of shear localization in some materials. The degree of shear localization has been shown to be related to the overall far field stress, the shear susceptibility of the materials and the nature of the notch tip. Traditional fracture tests have evaluated the behavior of these alloys under the influence of a single notch as per standard ASTM standards. In the present study a variety of dynamic fracture experiments with a range of notch types have been conducted. In this paper the results of these experiments will be rationalized and related to the microstructures of the materials.

11:20 AM

EFFECT OF VOLUME FRACTION ON MECHANICAL PROPERTIES OF SINTERED STAINLESS STEEL (SUS 3160) SPECIMEN PRODUCED BY METAL INJECTION MOLDING: *Rosdi Ibrahim*[1]; Sainsiah Sulaiman[1]; Mazli Mustapha[1]; Patthi Hussain[1]; Azmi Idris[1]; [1]Advanced Materials Research Centre (AMREC), Metal Technology Laboratory, SIRIM Berhad 1, Periaran Dato' Menteri, P.O. Box 7035, Shah Alam, Selangor 40911 Malaysia

This paper presents the investigation carried out to determine the effect of volume fraction of powder (58% and 65%) on mechanical properties of sintered stainless steel (SUS 316L) specimen produced by metal injection molding (MIM) technique. The stainless steel (SUS 316L) powder was mixed with the thermo-plastic binder system which comprises of polyethylene, paraffin wax and stearic acid at ratio of 25/60/15 by using z-blade mixer. The specimen was molded by using 70-ton Arburg injection molding machine and sintered under hydrogen atmosphere. The mechanical properties of the sintered specimen at each volume fraction were compared in term of density, ultimate tensile strength, hardness, modulus young and strain. The microstructure and carbon contents of sintered specimen will be discussed.

11:40 AM

SCANNING ELECTRON MICROSCOPY ANALYSIS OF AUSTEMPERED UNALLOYED DUCTILE ALUMINUM CAST IRON: *S. M. A. Boutorabi*[1]; [1]Iran University of Science and Technology, Department of Metallurgy and Materials, Narmak, Tehran Iran

The effect of austempering processing variables on the microstructure of the Fe-C-Al family of spheroidal graphite cast irons has been investigated. The Iron containing 3.2-3.4 %C , 2.1-2.3 Al% and Si, Ni, Mg, Sulphur, Phosphorus and tin in small quantities, was austenitived at 850, 900 and 956ϒC, and austempered in the temperature range 300ϒ-450ϒC, for times between 30 to 300 minutes. Scanning electron microscopy was used to analyze the microstructure. It has been shown that the microstructures obtained at higher austenitizing temperature revealed two type of retained austentle one with fine morphology and the other in a blocky form surrounded by ferrite growing in different directions. The length of bainitic ferrite appears to Increase with austenitizing temperature. Increasing the austempering temperature was shown to increase structural coarseness and the retained austenite content.

12:00 PM

MICROSTRUCTURAL STUDIES OF Ti-Ni SHAPE MEMORY ALLOY: *K. Sadrnezhaad*[1]; [1]Sharif University, Metallurgy Department, P.O. Box 11365-9466, Tehran Iran

Relationship between microscopic crystallographic data and electrical resistance mechanical testing results are studied by analyzing combustion synthesized polycrystalline Ti-Ni Alloys. Effect of chemical composition, thermomechanical treatment and ageing on R-phase transformation and hysteresis width was investigated. Microstructural studies showed that ageing affects on composition and relative volumes of metastable intermetallic compounds. Thermomechanical properties of Ti-50.03 atomic percent Ni, Ti-50.23 atomic percent Ni, Ti-50.33 atomic percent Ni shape memory alloys were influenced by cold working, solution treatment, ageing and amount of interstitial contaminations.

TEMPUS - SPACELAB PROCESSING OF METALLIC MELTS: Session I

Sponsored by: Materials Design and Manufacturing Division, Solidification Committee
Program Organizer: W. Hofmeister, Vanderbilt University, P.O. Box 1543 Station B, Nashville, TN 37235

Monday AM — Room: Rome
October 12, 1998 — Location: O'Hare Hilton Hotel

Session Chair: Jan Rogers, NASA Marshall Space Flight Center, Space Sciences Laboratory, Huntsville, AL 35812 USA

8:30 AM
TEMPUS FACILITY - MSL - SPACELAB OPERATIONS AND FUTURE DESIGN: *J. Piller*[1]; A. Seidel[1]; M. Stauber[1]; W. Dreier[2]; [1]Dornier Gambh, Daimler-Benz Aerospace, Friedrichshafen D-88039 Germany; [2]Deutsches Zentrum fur Luft, und Raumfahrt, (DLR), Bonn D-53227 Germany

The first part deals with the performance of the containerless processing facility TEMPUS during the two Spacelab flights of the MSL-1 mission in 1997. An overview of the observed positioning quality of the samples together with the achieved maximum and minimum temperatures is given. Furthermore, the processing environment, vacuum and gas atmosphere, the performance of sample diagnostics, pyrometers and cameras, and the quality of sample excitation for the determination of materials properties as surface tension, viscosity, specific heat etc. are described. The second part presents the results of a design study, how the well-developed technology of TEMPUS Spacelab can be transferred to a Space Station facility of TEMPUS. In particular, the modularity and flexibility with respect to the exchange of samples, cameras, pyrometers and other experiment specific devices for a long-term usage of the facility are emphasized.

8:50 AM
THERMOPHYSICAL PROPERTY OF LIQUID METALS IN MICROGRAVITY: *I. Egry*[1]; G. Lohoefer[1]; S. Schneider[1]; I. Seyhan[1]; B. Feuerbacher[1]; [1]German Aerospace Center, Institute for Space Simulation, Cologne, Germany DLR 51170 USA

Thermophysical properties of liquid metals and alloys are difficult to measure and therefore not well known. Containerless processing using electromagnetic levitation is an elegant way to obtain such data, in particular in the undercooled regime. During the MSL-1 Spacelab mission in 1997, such experiments have been performed in microgravity, using the TEMPUS facility. The oscillating drop technique was applied to determine surface tension and viscosity from the frequency and damping of surface oscillations. A non-contact inductive method was used for the measurement of the electrical conductivity. Results for a Co80Pd20 alloy and a eutectic Pd78Cu6Si16 will be presented.

9:10 AM
THERMAL EXPANSION OF GLASS FORMING METALLIC ALLOYS IN THE UNDERCOOLED STATE: *B. Damaschke*[1]; K. Samwer[1]; [1]University of Augsburg, Augsburg Germany

Measurements of thermal expansion of Zr and some Zr-based glass forming alloys were made under microgravity conditions in the TEMPUS facility, an electromagnetic levitation facility which enabled us to work under UHV and gas environment. The method is based on a CCD camera device imaging the sample from the side. The video signal was recorded on board of the orbiter and the tapes were analyzed after the mission by digital image processing. The shape and volume of the samples as a function of time and temperature was evaluated. The samples showed a rotational motion which lead to a deformation to an ellipsoid. The volume thermal expansion coefficients of Zr-based samples investigated in the melt are comparable to the value of 6∞10-5 K-1 measured on earth in our laboratory in the highly undercooled state of Zr65Al7.5Cu17.5Ni10. Financial aid from the DLR (former DARA) is acknowledged.

9:30 AM
INVESTIGATION OF THERMOPHYSICAL PROPERTIES OF GLASS FORMING Zr-BASED LIQUID ALLOYS BY NON-CONTACT AC-CALORIMETRY UNDER REDUCED GRAVITY CONDITIONS: *R. K. Wunderlich*[1]; A. Sagel[2]; C. Ettel[2]; H. -J. Fecht[2]; [1]Technical University of Berlin, Institute for Metallic Materials - Metal Physics, 36, PN 2-3, D-10623, Hardenbergstr, Berlin Germany; [2]University of Ulm, Faculty of Enginering, Materials Science Department, Albert-Einstein-Alle 47, Ulm D-89081 Germany

Zr-based alloys form the basis of many metallic glasses including binary eutectics with critical cooling rates in the range of 105 K/sec and the multicomponent bulk metallic glass formers with critical cooling rates as low as 10 K /sec. In order to evaluate the glass forming ability of these alloys we have measured the specific heat capacity of the stable and undercooled melt of some binary and multicomponent eutectic alloy compositions. Because of the high chemical reactivity and in order to achieve a large degree of liquid undercooling experiments were performed with electromagnetic containerless processing under reduced gravity during conditions in the IML-2 and MSL-1 spacelab missions. For these investigations the method of contactless ac-calorimetry was developed and successfully applied. In addition, based on calibration of electromagnetic power input by ac-calorimetry, the heat of fusion can be evaluated thus allowing a determination of the thermodynamic functions of the stable and undercooled melt. We will report results on binary eutectics and on the bulk metallic glass formers ZrAlCuNi and ZrAlCuNiCo. The latter showed good undercooling and exceptional stability in the undercooled melt. In the temperature range down to a relative undercooling of 0.82 a monotonous increase in the specific heat capacity is observed. However, conventional DSC measurements performed on glassy alloys just above the calorimetric glass transition seem to indicate a nonmonotonous behavior of the specific heat in the undercooled liquid. In addition, we will present results on the crystallization microstructure and phase analysis in comparison with the equilibrium phases.

9:50 AM
DENDRITE GROWTH VELOCITY MEASUREMENTS IN UNDERCOOLED Ni AND Ni-C MELTS: *Matthias Barth*[1]; Holland- Moritz[1]; D. M. Herlach[1]; D. M. Matson[2]; M. C. Flemings[2]; [1]German Aerospace Center (DLR), Institute of Space Simulation, DLR, Wb-RS, Koeln D-51170 Germany; [2]Massachusetts Institute of Technology, Cambridge, MA 02139 USA

Under terrestrial processing conditions, convection in the melt shows a major impact on metallic solidification, especially at small crystal growth velocities. Previous experiments e.g. on undercooled Ni melts showed a systematic theoretical underestimation of the growth velocities if convection is not considered. The aim of the present experimental studies was to test, under the conditions of reduced convection in microgravity, the predictions of dendrite growth models by comparative measurements of the growth velocities in pure Ni and dilute Ni-0.6 at.% C alloy melts. A comparison of these velocity data also allows to verify the prediction of marginal stability theory of growing dendrites that the addition of a strongly partitioning element may lead to an enhancement of the growth velocity compared to the case of pure metals. The experiments were performed during the MSL-1-R mission in July 1997 using the electromagnetic levitation facility.

10:10 AM BREAK

10:30 AM
NUCLEATION EXPERIMENTS ON TEMPUS IN LOW EARTH ORBIT: *W. H. Hofmeister*[1]; C. M. Morton[2]; M. B. Robinson[3]; R. J. Bayuzick[1]; [1]Vanderbilt University, Department of Chemical Engineering, Nashville, TN 37235 USA; [2]Teledyne Advanced Materials, Lavergne, TN USA; [3]Space Sciences Laboratory, Marshall Space Flight Center, AL USA

The TEMPUS facility on Spacelab mission MSL-1R was used to perform statistical nucleation experiments on pure zirconium. Two sets of experiments with approximately 50 undercooling and nucleation cycles each were performed at two distinct positioner power settings on one sample. The two positioner power settings cause different flow conditions during the undercooling experiments. The data sets were compared using standard statistical methods to determine if the flow conditions influence nucleation behavior. In addition, the nucleation kinetics for the experiment were evaluated using a statistical technique. Some evidence for fluid flow effects on nucleation was found at very high flow velocities.

10:50 AM
PHASE SELECTION AND RAPID SOLIDIFICATION OF UNDERCOLLED Fe-Cr-Ni STEEL ALLOYS IN MICROGRAVITY: *Douglas M. Matson*[1]; Merton C. Flemings[1]; [1]Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA USA

Molten samples of ternary steel alloys were successfully processed in a containerless fashion using the TEMPUS electromagnetic levitator during the Microgravity Sciences Laboratory (MSL-1) mission aboard the Shuttle Columbia. Transformation from the metastable ferritic phase to the stable austenitic phase was observed following either triggered or spontaneous nucleation for samples with a nominal composition of Fe-12wt%Cr-16wt%Ni and Fe-16wt%Cr-12wt%Ni. As in ground-based experiments, the delay time between recalescence events is a strong function of both composition and undercooling but the critical undercooling required to initiate this double recalescence is lower in microgravity than on the ground.

11:10 AM
THE MEASUREMENT OF THE SURFACE TENSION AND VISCOSITY OF UNDERCOOLED MELTS UNDER MICROGRAVITY CONDITIONS: RESULTS FROM MSL-1: *Robert W Hyers*[1]; Gerardo Trapaga[1]; [1]MIT, Materials Science and Engineering, 77 Massachusetts Ave., Room 4-043, Cambridge, MA 02139 USA

The accurate determination of the thermophysical properties of liquid metals is essential to the development of a number of scientific and engineering fields, including process modeling, nucleation and glass transition studies, and the fundamental properties of the liquid state. In this paper, we present and discuss some experimental results and calculations from the successful containerless surface tension and viscosity measurements performed using electromagnetic levitation (EML) in TEMPUS during the MSL-1 Space Shuttle missions (STS-83 and -94). Containerless processing allows measurements of undercooled and highly reactive melts. These measurements were performed using the oscillating drop technique for different sample materials over a wide range of temperature, including both superheated and undercooled regimes. Microgravity is essential in reducing the magnetically driven flow in the droplets, as well as allowing access to the lower temperature range of these measurements. We were able to obtain the temperature dependence of both surface tension and viscosity of these materials. The success of these measurements provides proof that the microgravity environment allows noncontact measurements of the molecular viscosity of liquid metals by this technique. Supporting calculations allow comparison of the flow conditions for different sample materials and operating conditions. These calculations involve a semi-coupled solution of Maxwell's equations and the Navier-Stokes equations, which describe the flow behavior of the system.

11:30 AM
ELECTRICAL CONDUCTIVITY OF METALLIC MELTS: *Georg Lohoefer*[1]; [1]German Aerospace Center, Institute of Space Simulation, POB 906058, Koeln 51170 Germany

During the MSL-1 Spacelab mission metallic samples in solid and liquid states have been processed containerlessly in the electromagnetic levitation facility TEMPUS. Under microgravity an electromagnetic levitation facility yields in priciple an optimal environment to measure by the way in an inductive manner the electrical conductivity σ of the processed samples. Although TEMPUS was not specifically designed for this purpose, the 'housekeeping data', i.e., generator voltage, current, and frequency, monitored by the facility, can be used to determine σ. In the talk we analyze these data and give values of σ for different samples.

TEXTURE IN FILMS AND COATINGS: Session I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Texture & Anisotropy Committee
Program Organizer: J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

Monday AM Room: Zurich/Tokyo
October 12, 1998 Location: O'Hare Hilton Hotel

Session Chair: David P. Field, 392 E. 12300 S. Suite H, Draper, UT 84020 USA

8:30 AM INVITED PAPER
EVOLUTION OF TEXTURE IN GROWTH OF THIN FILMS & MULTILAYERS: *John C. Bilello*[1]; Steven M. Yalosove[1]; [1]University of Michigan, Department of Materials Science & Engineering, Center for Nanomaterials Science, Ann Arbor, MI 48109-2136 USA

Polycrystalline thin films and multilayers can be grown via sputter deposition with distinct out-of-plane and in-plane preferred orientations. The format has sometimes been designated as "fiber" orientation and latter has been regarded as analogous to a "rolling" texture. The usage of both these terms is inappropriate and may actually obscure the interpretation of underlying physical phenomena. These in-plane and out-of- plane textures occur at low homologous temperatures and their crystallographic orientations can be varied by controlling the processing conditions. High resolution grazing angle incident x-ray diffraction (GIXS) juxtaposed to plane and cross section HREM have been used to observe and quantify the evolution of texture as function of processing conditions and film thickness. The degree of the texture at different growth depths has been nondestructively determined via GIXS Surprisingly, strong out-of-plane texture has observed to form in as little as 80nm of growth for Mo thin films. While in-plane texture becomes well organised after a film thickness of 1.0 um is reached. A simple phenomenological model will be presented to explain the evolution of out-plane and in-plane textures in low temperature sputtered deposited metalic films.

9:05 AM
EPITAXIAL POTASSIUM NIOBATE THIN FILMS: *Barbara M. Nichols*[1]; *Bruce W. Wessels*[1]; [1]Northwestern University, Materials Science and Engineering, 2225 N. Campus Drive, Evanston, IL 60208 USA

Epitaxial potassium niobate thin films were grown by metalorganic chemical vapor deposition (MOCVD). The influence of substrate epitaxial film orientation was investigated. The films were characterized by x-ray diffraction and atomic force microscopy. Epitaxial $KNbO_3$ with a (110) orientation was deposited on single crystal strontium titanate. In contrast, films grown on (100) spinel exhibited a multidomain structure, consisting of (110) and (001) oriented domains. Second harmonic generation measurements were utilized to determine the effective nonlinear optical susceptibility of the films and were related to the domain structure.

9:25 AM

TEXTURE CONTROL IN THIN FILMS USING ION BOMBARDMENT: *Gary S. Was*[1]; Hong Ji[1]; ZhenQiang Ma[1]; [1]University of Michigan, Nuclear Engineering and Radiological Sciences, 2355 Bonisteel Blvd., 1911 Cooley Building, Ann Arbor, MI 48109-2104 USA

Texture control is achieved during the deposition of thin films by a number of processes. Both fiber texture and in-plane texture can be accurately controlled via ion bombardment during deposition. In this paper, thin films of aluminum used in metallization of flat panel display circuits, and niobium films used in microlaminates are deposited in a high vacuum during simultaneous bombardment with an ion beam of inert gas in the energy range 200-1200 eV and at a normalized energy up to 400 eV/deposited atom. Aluminum films are synthesized in such a manner as to change the fiber texture from (111) to (110) in order to address the problem of hillocking. Results show that films with a predominant (110) texture are considerably more resistant to hillocking than are films with a (111) texture. Niobium films are deposited on single crystal sapphire substrates using ion bombardment at an angle of 50ϒ; to the normal to induce an in-plane texture and a specific orientation relationship with the sapphire substrate. A model is also developed which shows that the surface roughness is a direct result of the film texture.

9:45 AM

SIMULATION OF DIFFUSION PROCESSES IN POLYCRYSTALLINE SOLIDS: *Hualong Li*[1]; J. A. Szpunar[1]; [1]McGill University, Department of Metallurgical Engineering, 3610 University St., Montreal, PQ H3A 2B2 Canada

Understanding of diffusion processes is of great importance for understanding of processes like oxidation, precipitation, creep, annealing and other processes. In this paper, a two dimensional computer simulation model is proposed to simulate the diffusion process in polycrystallion solids which contains defects such as point defects, dislocations and grain boundaries. In this model, the microstructure is described by unit cell with specified orientations and the grain size and grain shape are introduced to the model using available experimental data. One million of unit cells is used in the model. Each unit cell has a property defined by diffusion coefficient which represents the lattice diffusion coefficient, or the dislocation diffusion coefficient or the grain boundary diffusion coefficient. Grain boundary diffusion coefficient is a function of grain boundary misorientation. The diffusion process is simulated with the Random Walk method. The output of simulation is presented as a normalized profile of oxygen concentration and a two dimensional contour map which display the distribution of diffusing species. Using this simulation model, the effects of grain shape, grain size and a role of various structure defects on the diffusion process can be studied. The model was tested using various experimental data and results obtained are compared with Fick/Es and Whipple/Es theoretical analysis.

10:05 AM

COMPUTER SIMULATION OF NiO TEXTURE FORMATION ON SINGLE CRYSTAL SUBSTRATE: *Hualong Li*[1]; F. Czerwinski[1]; J. A. Szpunar[1]; [1]McGill University, Department of Metallurgical Engineering, 3610 University, Montreal, QC H3A 2B2 Canada

One of the major factors that affect the diffusion process is the oxide texture, which determines the grain boundary character distribution (GBCD). Understanding of the oxide texture formation is therefore critical to the understanding of how and to what extent the oxide film can protect the alloy from oxidation. In this paper, a mechanism of oxide texture formation on Ni substrate was proposed. This mechanism is based on the assumptions that the lattice matching between the oxide and metal plays a main role in the stage of nucleation and the minimization of surface energies plays a main role in the stage of the oxide grain growth. A two-dimension computer model based on the proposed mechanism was developed and applied in the study of the texture formation process of NiO on (100) and (111) Ni single crystal substrate. The simulated oxide texture has good agreement with the experimental results.

10:25 AM BREAK

10:35 AM

TEXTURES AND MICROSTRUCTURES IN AS-DEPOSITED AND RECRYSTALLIZED Ni ELECTRODEPOSITS: *Insoo Kim*[1]; Sae Gwang Lee[1]; [1]Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 730-701 Korea

Textures and microstructures of Ni electrolytically deposited an the on the stainless steel substrate at room temperature, were studied as a function of current density and acid density. Annealing of deposits obtained from each acid bath were carried out at various time and temperature. Their textures and microstructures were measured by using pole figure goniometer, X-ray diffractometer and optical microscopy.The pole figure of each as-deposited Ni electrodeposits shows various types of fiber texture in various electrolysis conditions. The texture was influenced by a current density. Recrystalization texture is compared with texture of as-deposits.

10:55 AM

TEXTURE FORMATION IN ELECTRODEPOSITED COATINGS: *Oleg B. Girin*[1]; [1]State Metallurgical Academy of Ukraine, Duipropetrovsk 320635 Ukraine

New experimental and theoretical results are given that support and develop the views on texture formation in electrodeposited coatings presented earlier.The phenomena of accommodation grain/subgrain coarsening in the grain texture component due to coalescence growing grains by atomic plane flexure are addressed that give rise to texture development. Texture formation controlled by the surface, the grain boundary and/or the volume energy of the main component grains at the various stages of their development is discussed in relation to the initial structural condition of the random component grains. Formation bahavior of axial texture and texture-related substructure anisotropy is dealt with in relation to electrocoating crystal lattice type, supercooling in electrocrytallization, and depositon rate.

11:15 AM

TEXTURE OF ELECTRODEPOSITED COPPER COATINGS AS RELATED TO THEIR SUBSTRUCTURE, GRANULAR STRUCTURE AND SURFACE MORPHOLOGY: *Yullya O. Proshenko*[1]; Evgenii P. Kalinushkin[1]; Oleg B. Girin[1]; [1]State Metallurgical Academy of Ukraine, Dnipropetrovsk 320635 Ukraine

Recent data of comprehensive studies into relationships of the texture to the substructure, granular structure and surface morphology are presented for electrodeposited copper coatings. Quantitative evaluation of texture with consideration for extinction anisotropy, and determination substructure and granular structure characteristics in the axial and random components of the texture are addressed. The phenomena of texture-related anisotropy of substructure and granular structure inhomogeneity are discussed for copper plates. The relationships underlying the effects of deposition rate and supercooling in electrocrystallizaion on quantitative characteristics of texture and texture-relalated sub structure anisotropy in copper plates are considered.

11:35 AM

THE EVOLUTION OF TEXTURE DURING ANNEALING OF NANOCRYSTALLINE ELECTROLES: NICKEL ALLOY

DEPOSITS: *Dong Nyung Lee*[1]; [1]Seoul National University, Division of Materials Science and Engineering, Seoul 151-742 Korea

The electroless Ni-Cu-P films on 5086 aluminum alloy sheets obtained from nickel sulphate-copper sulphate solutions and the electroless Ni-Co-P films on 5086 aluminum sheets obtained from nickel sulphate-cobalt sulphate solutions showed similar microstructures, which are characterized by nickel nm dia, nickel solid solution grains with <111> orientation embedded in amorphous matrix. However, their annealing textures were very different, i.e., the former developed major <111> + minor <100> texture, and the latter <l00>. The result cannot be explained by a model basrecrystallization texture model based on dislocations, which was advanced by the present author, because the dislocation density might be very little in nanoscale grains. The result may be explained by interface energy between grains and amorphous matrix, and strain energy of films. In addition, a recent work on annealing textures of nanocrystalline Ni-45% Fe electrodeposits by Czerwinski et al. has been discussed.

11:55 AM

MICROTEXTURE EVOLUTION IN Al-Cu AND Cu THIN FILMS: David P. Field[1]; [1]TexSEM Labs, 392E 12300S, Draper, UT 84020, USA

The texture in integrated circuit interconnect lines is important in controlling electromigration and stress voiding behavior. As the minimum feature size of these lines continues to shrink with each new generation, the importance of controlling the film structure increases. A strong (111) fiber texture is necessary for optimum reliability of the interconnect lines. The microstructural feature which controls reliability, however, may be the structure of grain boundaries and the associated energetics and diffussive properties. This study investigates structure evolution of films and lines during annealing. Crystallographic texture and microtexture were monitored during grain growth and the statistics of the distributions were tracked. It is shown that the grain boundaries tend to certain preferred structures during the process. The results can be explained by a simple model using a combination of grain boundary and free surface energies as the controlling parameters.

DISCONTINUOUSLY REINFORCED ALUMINUM-COMPOSITES: PRESENT & FUTURE: Applications

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

Program Organizers: Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433; Warren H. Hunt, Aluminum Consultants Group, Inc., Murrysville, PA 15668 USA

Monday AM — Room: 13
October 12, 1998 — Location: Rosemont Convention Center

Session Chair: Dr. Benji Maruyama, Air Force Research Laboratory, Materials and Manufacturing Directorate, WPAFB, OH 45433 USA

8:30 AM

AUTOMOTIVE METAL MATRIX COMPOSITE APPLICATIONS: PAST AND PRESENT: *Dale A. Gerard*[1]; [1]General Motors Corporation, Light Alloy Materials, Advanced Materials Development Center, P.O. Box 5073, Saginaw, MI 48605-5073 USA

The potential for Discontinuous Metal Matrix Composites (DMMCs) to reduce mass, enhance emissions, and increase fuel economy of automobiles has been discussed thoroughly in the literature over the past 20 years. DMMC technology, although still in its infancy, has matured to a level which is allowing it to be selected more frequently for automotive applications. This presentation will review past and present MMC-automotive applications. In addition, a brief summary will be given which elaborates on several key challenges which must be overcome to allow DMMC-based products to become a truly competitive alternative for high volume automotive applications.

9:00 AM

AIRCRAFT APPLICATIONS OF DISCONTINUOUSLY REINFORCED ALUMINUM SYSTEMS: *L. Keith Austin*[1]; [1]Lockheed Martin Technical Aircraft, P.O. Box 748, MS 1720, Fort Worth, TX 76101 USA

The evolution of Discontinuously Reinforced Aluminum (DRA) Composites from laboratory curiosities to high performance aircraft materials solutions has been a success story based on its use in F-16 Ventral Fins, and F-16 Fuel Access Cover Doors, as well as fan exit guide vanes for the Pratt & Whitney 4000 Series aircraft engines. This paper captures the magnitude of the life-cycle-cost estimates and the benefits of the DRA sheet material for aging aircraft and new aircraft applications. The presentation will also address current efforts to increase scale and expand supportability of sheet-based DRA products.

9:30 AM

APPLICATION OF DISCONTINUOUSLY REINFORCED ALUMINUM METAL MATRIX COMPOSITES IN ELECTRONIC PACKAGING AND PRODUCTION MACHINERY: *Carl Zweben*[1]; [1]FASM Consultant, 62 Arlington Road, Devon, PA 19333 USA

In a relatively short time, metal matrix composites consisting of aluminum reinforced with ceramic particles (discontinuously reinforced aluminum, i.e. DRA) have become important electronic packaging materials. They are now being used in both commercial and aerospace products. Current annual production runs for some applications are reported to be as high as hundreds of thousands of components. In this paper, we review the history of DRAs in electronic packaging along with properties, processes and applications. We also examine needs, opportunities and the future of these important materials throughout the electronics industry, including not only packaging, but also production machinery.

10:00 AM

RESEARCH AND DEVELOPMENT OF DISCONTINUOUSLY-REINFORCED METALS: *Daniel B. Miracle*[1]; [1]Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLM, 2230 Tenth Street, WPAFB, OH 45433 USA

Although a large amount of information is available on the deformation and fracture of discontinuously reinforced metals (DRX), a coherent understanding of the issues which limit the application of these materials is still lacking. Specific material properties required to allow widespread structural usage will be described, and research and development efforts which can provide the guidance needed to develop the next generation of DRX will be presented and discussed.

10:30 AM

THE ALUMINUM MMC CONSORTIUM: *Warren H. Hunt*[1]; [1]Aluminum MMC Consortium, FASM, 4530 William Penn Highway, #3900, Murrysville, PA USA

A consortium of corporate, academic, and government participants has been formed to advocate expansion of opportunities for Discontinuously Reinforced Aluminum (DRA) materials, which have seen successful niche applications in a number of markets. The main activities of the Consortium are facilitating the development of manufacturing process technology and promoting the materials through a User Resource Center.

DYNAMIC BEHAVIOR OF MATERIALS: Dynamic Behavior I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Flow & Fracture Committee, Mechanical Metallurgy Committee

Program Organizers: Rusty Gray, Los Alamos National Lab, Dynamic Properties, Los Alamos, NM 87545-0001; Marc A. Meyers, IMM, University of California, San Diego, CA 92093 USA; Naresh Thadhani, Georgia Inst of Tech, Sch of Matls Sci & Engrg, Atlanta, GA 30332-0245; Ken S. Vecchio, University of California, Dept of Ames, San Diego, CA 92093 USA

Monday PM   Room: Sydney
October 12, 1998   Location: O'Hare Hilton Hotel

Session Chairs: George T. Gray, Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA; Kenneth S. Vecchio, University of CA, Dept. of AMES, La Jolla, CA 92093-0411 USA

2:00 PM OPENING COMMENTS

2:05 PM
THE INFLUENCE OF OXYGEN CONTENT ON THE SHOCK-LOADING RESPONSE OF TITANIUM: *George T. (Rusty) Gray*[1]; T. A. Mason[1]; *G. C. Kaschner*[1]; S. R. Chen[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545* USA

In this study, the influence of oxygen content on the shock-hardening response, propensity of ω-phase formation, propensity for deformation twin formation, and texture evolution in two grades of α-titanium is presented. The influence of shock-wave deformation on the phase stablility, shock hardening, and substructure and texture evolution of high-purity (low-interstitial) Ti and A-70 (3700 ppm)Ti was probed by utilizing "soft" shock recovery. Wave profile studies of shock-loaded high-purity Ti revealed an ω-phase pressure-induced transition at 10.4 GPa while A-70 Ti, shocked at pressures up to 35 GPa, exhibited no evidence of a three-wave structure. Suppression of the α-ω phase transition in A-70 Ti is seen to simultaneously correlate with the suppresion of deformation twinning which in turn alters the texture evolution in this Ti material. The influence of interstitial content on the kinetics of ω-phase formation/retention, substructure evolution, and shock hardening is discussed and contrasted in the context of previous literature studies. *Work performed under the auspices of the U.S. Department of Energy

2:25 PM
THE LIMITING SPEEDS OF DISLOCATIONS (LORENTZ CONTRACTION A MYTH?): *John J. Gilman*[1]; [1]University of California at Los Angeles, Materials Science and Engineering, 6532 Boelter Hall, UCLA, Los Angeles, CA 90095 USA

Sir Charles Frank showed in 1949 that the displacement field of a stationary screw dislocation in an infinite, uniform material can be converted into a traveling solution of the appropriate elastic wave equation by transforming the coordinate system by means of the Lorentz-Fitzgerald function. This is true. Then a cottage industry arose, making a variety of deductions based on this result. Most of these are either not true or have no connections with the operational (real) world. One significant problem is that the real world is finite, so solutions in elasticity theory do not exist unless the boundary conditions are specified and satisfied. In this particular case, the material spins unless countervailing tractions are applied which obviate the L-F function. An alternate, simple derivation of the limiting speed is given in this paper.

2:45 PM
AN ULTRASONIC STUDY OF SHOCK HARDENING IN Cu AND Cu-Al ALLOYS: Kenneth S. Vecchio[1]; *Aashish Rohatgi*[1]; George T. Gray[2]; [1]UC San Diego, Materials Science Group, Dept. of Applied Mechanics, La Jolla, CA 92093 USA; [2]Los Alamos National Laboratory, Group MST-8, Structure / Property Relations, MS G755, Los Alamos, NM USA

Cu and Cu-Al solid-solution alloys have been extensively studied in the past to correlate the quasi-statically deformed and shock-deformed microstructures to their respective stacking fault energies (SFE). In this alloy system, the SFE varies from 78 ergs/cm2 for pure copper to as low as 5 ergs/cm2 for Cu-6wt.% Al. Decreasing SFE results in a greater tendency to form stacking faults, with wider spacing between the bounding partial dislocations. Correspondingly, the extent of cross slip decreases, while the extent of deformation twinning increases. In these materials, shock deformation produces greater strengthening than obtainable by conventional deformation to an equivalent strain. This shock hardening has been ascribed to a greater dislocation and deformation twin density caused by the high strain rates (approx. 10^9 /s) associated with shock loading. However the extent of shock hardening as a function of SFE, remains an unresolved issue. Specifically, it is unclear how dislocation-line length changes as a function of SFE, and how this influences the shock hardening behaviour. In this work, Cu and Cu-Al alloys with 0.2, 2, 4 and 6 wt.% Al were tested in compression over a range of strain rates, as well as subjected to shock pressures of 10 GPa and 35 GPa. Owing to the extremely high dislocation densities in the shock prestrained microstructure, conventional transmission electron microscopy techniques are of limited value for providing measurements of dislocation-line length. This limitation was overcome by using ultrasonic measurements of velocity and attenuation. The Granato-Lucke theory was then used to estimate the dislocation density and dislocation-line length. While both the quasi static and shock-reload strength increases with decreasing SFE (i.e. increasing Al content in Cu), the strength increment is relatively insensitive to SFE. The role of dislocation-line length in the shock hardening behavior of Cu-Al alloys, in conjunction with the solid-solution hardening and deformation twinning, will be presented.

3:05 PM
THE MECHANICAL RESPONSE OF A DEPLETED URANIUM ALLOY AS A FUNCTION OF STRAIN RATE AND TEMPERATURE: *Carl M. Cady*[1]; G. T. Gray[1]; S. Hecker[1]; D. Thoma[2]; D. Korzekwa[2]; A. Patterson[2]; P. Dunn[2]; [1]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-6, MS-G770, Los Alamos, NM 87545 USA

The compressive stress-strain response of a U-6Nb alloy as a function of temperature and strain rate was investigated. The yield and flow stresses of the U-6Nb alloy was found to exhibit a pronounced rate sensitivity, while the hardening rates were found to be insensitive to strain rate and temperature with the intrinsic rate-controlling mechanism in U-6Nb being overcoming the Peierls stress. The overall stress-strain response of the U-6Nb exhibits a sinusoidal hardening behavior consistent with multiple deformation modes, including twinning and martinsite formation, and similar to shape-memory behavior. The stress-strain response of U-6Nb under quasi-static uniaxialtension will be compared to the compression data to show the sensitivity of stress-state. The material response of U-6Nb will be compared to pure uranium and other low symmetry materials.

3:25 PM
DYNAMIC EFFECTS IN HIGH-SPEED FRICTIONAL SLIDING: *A. Molinari*[1]; Y. Estrin[2]; S. Mercier[1]; [1]LPMM, Universite de Metz, F-57045, Metz, Cedex France; [2]The University of Western Australia, Department of Mechanical and Materials Engineering, MME, Nedlands, WA 6907 Australia

Dry friction of metals at high sliding velocities is of interest in many engineering applications, particularly, in high-speed machining. While the effect of heat evolving due to dynamic sliding on the coefficient of friction has been considered previously, no convincing model has been offered so far. In this paper, a new approach to modeling high-speed unlubricated friction of metals is proposed. The sliding resistance is considered to be governed by shearing of adhesive asperities. Due to high rate of shearing, asperities are considered to fail by adiabatic shear banding. Analytical solutions for a coupled thermomechanical problem are given for different choices of the constitutive equation for the material undergoing frictional sliding. They are investigated numerically for the case of steel-on-steel friction. The predicted decrease of the coefficient of friction with the sliding velocity, which is accentuated by increased normal pressure, is supported by experimental data. The range of validity of the model is considered to be 1 - 10m/s. Above 10 m/s, such phenomena as enhanced oxidation and surface melting may interfere with the asperity shearing mechanism considered. The talk will provide an outline of the basic features of the model and will give illustrations of its descriptive capabilities.

3:45 PM BREAK

3:55 PM
DEFORMATION BEHAVIOR OF TANTALUM AT HIGH AND LOW STRAIN RATES: *Christopher Bull*[1]; Clyde L. Briant[1]; David Lassila[2]; [1]Brown University, Division of Engineering, P.O. Box D, Providence, RI 02912 USA; [2]Lawrence Livermore National Labs, 7000 East Avenue, Livermore, CA 94550 USA

This paper will present a detailed study of deformation mechanisms in polycrystalline tantalum. The results will show that the initial microstructure prior to the test is very sensitive to the annealing temperature. Once deformation begins, the dislocation structures that form depend on the strain rate and the temperature of testing. At low strain rates, a cell structure forms with increasing deformation. However, at strain rates typical of those produced in a Kolsky bar, the cell structure is replaced by one of dislocation tangles. Comparisons will be made between samples tested in compression on an Instron machine and a Kolsky bar and between samples tested on a Kolsky bar in compression and in torsion. The effects of test temperatures and tungsten additions to the material will be discussed.

4:15 PM
HIGH-STRAIN-RATE RESPONSE OF LOW-IMPEDANCE MATERIALS: *Weinong Chen*[1]; Bin Zhang[1]; Binling Zhou[1]; [1]The University of Arizona, Aerospace & Mechanical Engineering, 1130 N. Mountain, Tucson, AZ 85721 USA

Two techniques based on the split Hopkinson pressure bar have been developed for determining the mechanical responses of soft materials such as polymers and foams at high strain rates. Modifications were made on the transmission bar. An aluminum hollow transmission bar increases the transmitted strain signal by an order of magnitude as compared to a conventional steel solid bar. The hollow bar performed satisfactorily in testing low mechanical impedance specimens such as plastics and polymeric composites. For specimen materials of very low impedance such as soft rubbers and foams, a piezoelectric transducer embedded in an aluminum transmission bar directly measures the small transmitted force with a resolution of within one Newton. The incident pulse must be shaped in order to ensure the stress equilibrium in the soft specimen and to achieve a constant strain rate during an experiment. Experimental results on a rubber and a silicone are presented.

4:35 PM
COMPARISON BETWEEN HIGH STRAIN RATE AND LOW STRAIN RATE DEFORMATION IN TANTALUM: *Rajeev Kapoor*[1]; Sia Nemat-Nasser[1]; [1]UCSD, Materials Science Department-0418, 9500 Gilman Drive, La Jolla, CA 92093 USA

In order to understand the constitutive behavior of tantalum, compression tests were carried out for a range of strain rates (0.0001/s to 3000/s) and a range of temperatures (296K to 1000K). The flow stress was assumed to be a sum of a thermal component and an athermal component. At high strain rates (3000/s), the thermal component was observed to be independent of strain, whereas the athermal component was observed to be independent of the temperature and strain rate history. At lower strain rates, however, such a separation between strain, and strain rate and temperature was not observed. At lower strain rates and higher temperatures, the effects of dynamic strain aging were observed. Dynamic strain aging did not affect the thermal component of stress, and only affected the evolution of the structure. The evolution of the structure in turn affected the athermal stress component. Another interesting observation was that the athermal components of flow stress were different for high strain rate tests as compared to low strain rate tests. By performing change of strain rate tests at these higher temperatures, it was concluded that such a difference could not arise because of difference in structure evolution at different strain rates. There probably exists another strain rate dependent mechanism at these higher temperature.

4:55 PM
TEMPERATURE AND STRAIN RATE DEPENDENCE OF MECHANICAL BEHAVIOR OF BODY-CENTERED CUBIC STRUCTURE MATERIALS: *Pietro Paolo Milella*[1]; [1]ANPA, Via V. Brancati, 48, Rome 00144 Italy

It is well known that metals with a body-centered cubic (BCC) lattice, like carbon steels, show a mechanical behavior strongly dependent on temperature T and strain rate ε. This, in general, is not observed on the other family of metals having a face-centered cubic (FCC) structure, in particular as far as the yield strength is concerned, which is almost independent of temperature. In between these two opposite behaviors are close-packed hexagonal structures. Many attempts have been made to correlate the yield strength σ_y to temperature T and strain rate ε deriving what is known as the mechanical equation of solid state $\sigma_y=f(\varepsilon,T)$. The paper presents a new treatment to derive the mechanical equation of the solid state in BCC metals based on the Cottrell atmosphere and solubility of interstitial atoms, supported by the experimental evidence obtained on a large variety of steels having very different mechanical properties, from low to very high strength. The starting point is the basic difference between BCC and FCC structure behavior that can be attributed to lattice distortion caused by dislocations, which is small in BCC materials and large in FCC ones. The consequence is that in BCC crystals the Cottrell atmosphere is more concentrated with respect to surrounding atoms in solid solution, mainly carbon and nitrogen, while in FCC structure is not, exerting a relevant drag force when the body is forced to deform plastically. By increasing the temperature, more interstitial atoms enter the solid solution balancing the two concentrations, reducing the drag force and, therefore, the yield strength. The opposite happens when temperature is decreased: foreign atoms leave the solid solution decreasing the relative concentration and unbalancing the system. If the instant variation $d\sigma_y$ of the yield strength σ_y, resulting from a variation of temperature T, is related to that dn of interstitial atoms in solid solution: $d\sigma_y/\sigma_y=dn/n$ (1) a first equation is derived which takes the form: $\sigma_y=A(\exp B/T)$ (2) where A is a constant characteristic of the particular alloy and B is related to the solubility of foreign atoms, carbon and nitrogen in particular. In a $\ln\sigma_y$ versus 1/T plot, eq. 2 is a line whose intersection O with the y axis, i.e. the $\ln\sigma_y$ axis, provides the value of the constant A. Eleven different steels were analysed using eq. 2 in a wide range of temperatures. They did show the linear trend predicted by eq. 2 in a $\ln\sigma_y$ versus 1/T plot and could be grouped into three different families, namely high, medium and low strength, each having a characteristic and different slope: higher for the low strength steels and lower for the high strength ones, all converging towards almost the same yield strength at extremely low temperature, about -250 °C. Moreover, by increasing the strain rate from quasi-static to dynamic conditions, up to 540 s^{-1}, the experimental data continued to show the linear trend, changing slope, but without changing the intersection point O with the $\ln\sigma_y$ axis, keeping the same A value, as shown in fig. 1 for a low carbon steel type A 533B. The slope m of the lines so obtained at different strain rate ε, all lay on a straight line on an m versus lnε plot, as shown in fig. 2 for the same steel tested at four different strain rates, from 10_{-3} to 540 s_{-1}. This findings have led to the final solid state equation: σ_y=A(exp B/T $\varepsilon^{C/T}$) (3) where A, B and C are constants characteristic of the particular material that can be derived experimentally. At variance with any other existing solid state equation, eq. 3 shows that temperature has a direct effect on strain rate since it enters the ε exponent: an increase of temperature will decrease the strain rate effect, till washing it out completely. On the contrary, by decreasing the temperature the strain rate effect will be augmented. Eq. 3 and the theory, also provide a very simple way to derive the values of the three constants and, in particular, that of the exponent C and, therefore, to know the effect on the yield strength of materials of any strain rate at any temperature. It is sufficient, in fact, to run a minimum of two tests at two convenient temperatures under quasi-static conditions and only one at any other temperature and strain rate. For convenience, ε can be chosen equal to 1 or 10 s^{-1} , available on any testing machine avoiding sophisticated test rigs such as a Hopkinson bar or wave propagation methods etc. The two tests at different temperatures under quasi-static conditions will provide the base line in a $\ln\sigma_y$ versus 1/T plot whose intersection O on the y axis will yield, as already said, the value of the constant A and whose slope m1 a data point in the m versus lnε dyagram. The other test at a given strain rate will yield a single point in the $\ln\sigma_y$ versus 1/T plot. By joining this point to the already found intersection O with the y axis, which is the same for all strain rates and temperatures, a new line will be obtained whose slope m2 together with the previous one m1 will build another line in an m versus lnε plot. The coefficients of the equation of this line will provide the B and C values.

5:15 PM

THE DYNAMIC COMPRESSIBILITY OF SOFT SOILS IN THE PRESSURE RANGE OF 0.05-1.5 GPA: *Anatoly M. Bragov*[1]; *Andrey K. Lomunov*[1]; Sergey A. Lomunov[1]; [1]Research Institute of Mechanics, State University of Nizhny Novgorod, 23, Gagarin Ave, Nizhny Novgorod, Nizhegorodska'ya 603600 Russia

The present paper describes the laboratory methods for studying the dynamic compressibility of soft soils and gives the experimental data on the dynamic properties of some soft soils in the pressure range from several tens of MPa to 1500 MPa and strain rates over 10^3 s^{-1} obtained using two methods. For the moderate pressure range (up to 200 MPa), a jacket-confined modification of a well-known Split Hopkinson Pressure Bar method is used, where radial strain of a specimen is confined by a rigid jacket. In addition to obtaining the deformation diagrams, the modification presented allows one to determine shear resistance of soils as a function of pressure, based on the results of jacket-confined tests and measurements of the circumferential strain of the jacket. For the pressures of over 300 MPa and strain rates in excess of 10^4 s^{-1}, the response of soils is investigated using a plane-wave impact experiment. A soil specimen is placed between two plates and loaded by a 57 mm-dia striker-plate accelerated in the bore of a gas gun. Measuring the kinematic parameters of the compression waves and using a popular method of impedance matching allows one to obtain the data on the dynamic compressibility of soils. The conducted tests resulted in the dynamic diagrams and shock adiabatic lines plotted for sand, clay and loam. Based on the data obtained using the two complementary methods, the present authors pioneer generalized dynamic compressibility curves for soft soils in the previously unstudied range of loading parameters.

EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Crystallographic Effects in Microstructure Formation

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Chemistry & Physics of Materials Committee, Phase Transformations Committee, Thermodynamics & Phase Equilibria Committee

Program Organizers: William C. Johnson, University of Virginia, Materials Science & Engineering Dept.; William J. Boettinger, National Institute of Standards & Technology, Room A153 Bldg 223; Carol Handwerker, National Institute of Standards & Technology, Room A153 Bldg 223, Gaithersburg, MD 20899 USA; Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Dept.; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Monday PM — Room: International Ballroom
October 12, 1998 — Location: O'Hare Hilton Hotel

Session Chair: Leonid Bendersky, NIST, Metallurgy Division, Gaithersburg, MD 20899 USA

2:00 PM

N-DIMENSIONAL CRYSTALLOGRAPHY: SYMMETRY AND PHYSICS: *D. Gratias*[1]; [1]LEM-CNRS/ONERA, Chatillon 92322 France

We will show that standard crystallographic symmetries, where objects superimpose exactly on top of each other, can be extended to "weak" symmetries, where objects superimpose only partially but following certain geometric rules. These rules will be discussed in both the N-dimensional picture and the "Harmonious Ensemble" concept defined by Y. Meyer. We will show that these weak symmetries are pertinent with respect to invariance in physics. In other words, we will see that the icosahedral symmetry of quasicrystals can be considered as a true symmetry and not a pseudo- or approached symmetry although quasicrystals do not have exact five-fold rotation axes in the geometric sense.

2:45 PM INVITED PAPER

EQUILIBRIUM SHAPES OF Pb INCLUSIONS AT GRAIN BOUNDARIES IN Al: *Uli Dahmen*[1]; [1]Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Building 72, 1, Cyclotron Road, Berkeley, CA 94720 USA

The equilibrium shapes of solid and liquid Pb inclusions between 5 and 500nm in size embedded in a solid Al matrix have been investigated by transmission electron microscopy. For solid inclusions, the equilibrium shape is cuboctahedral with an aspect ratio that depends on size. Solid inclusions at grain boundaries adopt a composite shape made of two partial equilibrium shapes, one from each grain, joined across the boundary. The composite shape depends on the misorientation and inclination of the grain boundary and is affected by the requirement of shape compatibility at the interface junction. The melting process of these inclusions has been observed directly by in-situ electron microscopy. It is found that the melting behavior depends strongly on the inclusion shape. For liquid inclusions with approximately isotropic interface energy, the equilibrium shape at grain boundaries is lens-shaped and at triple junctions is acorn shaped. Both, solid and liquid equilibrium shapes are analyzed using the Hoffman/

Cahn ξ-vector construction. This work is supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract No. DE-ACO3-76SFOOO98.

3:30 PM BREAK

3:50 PM INVITED PAPER

THE THEORY OF GENERALISED CAHN-HOFFMAN XI-VECTORS: *A. A. Wheeler*[1]; [1]University of Southampton, Department of Mathematics, Southampton, S017 1BJ U.K.

In this paper we briefly review both the diffuse interface theory of interfaces pioneered by Cahn, e.g. the Cahn-Allen equation for interphase boundaries and the Cahn-Hilliard equation for spinodal decomposition, as well as the Hoffman-Cahn theory of sharp interfaces with anisotropic surface energy based on the notion of a xi vector. The central thesis of this paper is that these two intrinsically different models of an interface may be unified in the notion of generalised xi vector which may be associated with anisotropic diffuse interface models. We will demonstrate that the generalised xi vector provides a natural theoretical setting for the diffuse interface models and provides a connection to the sharp interface xi vector theory. We build on this idea to show that the generalised xi vector is associated with the type of the underlying partial differential equations of diffuse interface models. It also plays an important role in the identification of a stress tensor which represents the reversible stress distribution within in an interface due to its surface energy and provides a connection to the notion of surface tension in a sharp interface model. We illustrate these ideas mainly in the setting of a phase-field models but also discuss how they are implemented in more realistic and sophisticated diffuse interface models of interfaces in, for example, an ordered fcc material, eutectic and peritectic alloys, or the isotropic-nematic phase transition in a liquid crystal.

FATIGUE BEHAVIOR OF TITANIUM ALLOYS: Mechanisms of Fatigue Crack Initiation and Propagation of Conventional Alloys

Sponsored by: Structural Materials Division, Titanium Committee
Program Organizers: Rod Boyer, Boeing Commercial Airplane Group, Seattle, WA 98124-2207 USA; Daniel Eylon, Univ. of Dayton, Graduate Materials Engineering, Dayton, OH 45469-0240; J. P. Gallagher, University of Dayton, UDRI-Structural Integrity, Dayton, OH 45469-0120 USA; G. Lutjering, TU Hamburg-Harburg, Hamburg 21093 Germany

Monday PM — Room: Athens/Berlin
October 12, 1998 — Location: O'Hare Hilton Hotel

Session Chairs: R. Boyer; M. Niinomi

2:00 PM INVITED PAPER

FATIGUE-CRACK INITIATION AND GROWTH IN Ti-6Al-4V UNDER HIGH-CYCLE FATIGUE CONDITIONS: B. L. Boyce[1]; J. P. P.[1]; O. Roder[1]; A. W. Thompson[1]; *R. O. Ritchie*[1]; [1]University of California, Department of Materials Science and Engineering, 463 Evans Hall, Berkeley, CA 94720-1760 USA

Fatigue-crack initiation and growth under high-cycle fatigue (HCF) loading is currently a primary concern for the aircraft engine industry. In-flight loading spectra characterized by high cyclic frequencies ($\upsilon \sim$ 1-2 kHz) necessitate a damage-tolerant design approach based on the fatigue-crack growth threshold, ΔK_{TH}. Furthermore, regions of surface damage, e.g., fretting and foreign object damage (FOD), are suspected crack initiation sites. Given these service conditions, definition of limiting states of surface damage which lead to HCF failure is critical. To this end, both small crack (a<500 µm) and long-crack (a>5 mm) techniques are employed on a Ti-6Al-4V blade alloy (~50% primary α + transformed β) to estimate lower-bound values of ΔK_{TH} for υ = 50-1000 Hz in the presence of notches and various states of microstructural damage, e.g., FOD and fretting damage. Effects of load ratio, mixed mode loading and HCF/LCF interactions on ΔK_{TH} are considered. *Funded by the Air Force Office of Scientific Research through the Multidisciplinary University Research Initiative on High-Cycle Fatigue to the University of California at Berkeley.

2:30 PM

MICROCRACK PROPAGATION IN Ti-6Al-4V: *J. A. Hines*[1]; J. O. Peters[1]; G. Lutjering[1]; [1]Technische Universitat Hamburg-Harburg, Eissendorfer Str. 42, Hamburg, FRG 21073 Germany

With the aging of components in service under HCF conditions (NF = 106 -107), it is important to understand microcrack propagation and fatigue life in this region of deformation. In this study, microcrack propagation in Ti-6Al-4V was examined in regions just above and below the fatigue limit. Two different duplex or bi-modal materials were examined: an alloy containing approximately 40% primary alpha phase in a lamellar structure and an alloy containing 60% primary alpha. Microcracks (approximately 20-40µm) were nucleated in smooth specimens in the LCF regime during tests conducted at R = -1 so that internal stresses could be minimized. These samples were then tested in the HCF regime to determine crack propagation rates for R values of -1, 0.1, and 0.5. In addition, conventional fatigue crack propagation curves for macrocracks using CT-specimens were measured. Both materials exhibited a stress amplitude dependence in the microcrack propagation curves for given R values which is explained by the increasing roughness of the crack front profile with increasing size of the microcracks.

2:50 PM

RELATIONS BETWEEN MICROSTRUCTURE AND FATIGUE PROPERTIES IN TITANIUM ALLOYS: *Anthony W. Thompson*[1]; [1]Lawrence Berkley National Lab, Mat'ls. Science Division, MS 62-203, Berkeley, CA 94720 USA

The role of microstructural variables in controlling mechanical properties of alpha-beta titanium alloys has been well explored. Particularly for fatigue properties for both low-cycle and high-cycle fatigue, there are a number of generalizations which appear appropriate, involving the separate roles of primary and secondary alpha phase, and the size of both individual alpha plates, and of alpha packets or colonies. Variations in these dependencies due to composition and texture are known to occur. The current status of this understanding is the basis for recommendations of additional research.

3:10 PM INVITED PAPER

FATIGUE OF BI-LAMELLAR MICROSTRUCTURES: *G. Schroeder*[1]; J. Albrecht[1]; G. Lutjering[1]; K. -D. Folkers[1]; Ch. Liesner[1]; [1]Technische Universitat Hamburg-Harburg, Eissendorfer Str. 42, Hamburg, FRG 21073 Germany

It has been shown that so-called bi-lamellar microstructures have a potential to improve the properties of titanium castings. The treatment to produce this microstructure can be easily incorporated into the standard processing scheme of an investment cast part. It consists of a simple heat-treatment in the $\alpha+\beta$-phase field and can be performed even under air. By the heat treatment, fine plates are precipitated in the β-phase of the lamellar microstructure, reducing the slip length and thereby improving the mechanical properties. The paper focusses on the fatigue properties of bi-lamellar microstructures, particulary in Ti-6Al-4V, in comparsion with conventional lamellar microstructures. The fatigue properties will be discussed on the basis of crack nucleation and crack propagation of micro- and macrocracks. The influence of the processing parameters of the annealing treatment as well as the influence of the starting lamellar structure on the fatigue properties will be addressed. Conditions which received a mill anneal treatment will be compared with conditions which were aged to precipitate fine Ti_3Al particles.

3:30 PM BREAK

3:50 PM

ROLE OF MICROSTRUCTURE AND TEXTURE IN THE MEAN STRESS DEPENDENCE OF Ti-6Al-4V: *Svetlana G. Ivanova*[1]; Frederic Cohen[2]; Richard D. Sisson[1]; Ronald R. Biederman[1]; [1]Worcester Polytechnic Institute, 100 Institute Road, Worcester, MA 01609-2280 USA; [2]Prat & Whitney, Mail Stop 114-40, 400 Main Street, East Hartford, CT 06108 USA

The high cycle fatigue properties of Ti-6Al-4V with six different microstructure/texture combinations were experimentally investigated. Only materials with lamellar and fine bimodal microstructures exhibited linear Goodman relationship on the constant fatigue life diagram. Materials with coarse bimodal and equiaxed microstructures had anomalous mean stress dependency, with HCF strength at intermediate mean stresses being lower than predicted by Goodman relationship, regardless of whether material was forged or cross-rolled. The roles of microstructure and texture in mean stress sensitivity behavior of Ti-6Al -4V are presented and discussed with the focus on crack initiation mechanisms.

4:10 PM

MEAN STRESS EFFECTS ON FATIGUE STRENGTH IN TITANIUM ALLOYS: *J. Lindermann*[1]; L. Wagner[1]; [1]Technical University of Brandenburg at Cottbus, Department of Materials Technology, 101344, Postfach, Cottbus 03013 Germany

The stress ratio or mean stress dependence of the 107 cycles fatigue strength of a material is usually evaluated from a so-called Smith diagram, where the maximum allowable stress is plotted versus the mean stress. If only the HCF strength at R = -1 and UTS are known, the allowable stress amplitude for a given tensile mean stress can be roughly estimated by the equation: $\sigma_{a107\ (m>0)/=}\ \sigma_{a(m=0)}(1-\sigma_m/UTS)$ For must structural materials such as steels and aluminum alloys, this simple relationship is conservative. However, titanium alloys can show a so-called anomalous mean stress sensitivity (AMSS) in that their HCF strength at low tensile mean stresses can be significantly lower than would be predicted from the above equation. In the present work, AMSS in titanium alloys was investigated by studying the fatigue response of the various alloy classes, i.e., α, $(\alpha+\beta)$ and β alloys. AMSS was observed only in $(\alpha+\beta)$ alloys. The effects of microstructure (phase morphology, degree of age-hardening) and crystallographic texture including microtexture on AMSS will be discussed.

4:30 PM

MEAN STRESS EFFECTS ON FATIGUE CRACK GROWTH BEHAVIOR OF STRUCTURAL TITANIUM ALLOYS: AN OVERVIEW OF MICROSTRUCTURAL AND MECHANICS ISSUES: *K. S. Ravichandran*[1]; S. K. Jha[1]; [1]The University of Utah, Department of Metallurgical Engineering, 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

The mean stress or stress ratio of the fatigue cycle is known to have a significant effect on fatigue crack growth responses of titanium alloys. An understanding of this effect is important from the perspective of life-prediction of structures. A survey of the literature as well as the authors' research indicates that the degree of mean stress effect is varied, depending on the alloy type, microstructure and environment. In this presentation, the most significant material and microstructural issues have been identified and discussed. Additionally, outcome of the attempts to correlate the mean stress effect through simple mechanistic approaches has been illustrated. Directions for future work are identified.

4:50 PM

AN INVESTIGATION OF THE EFFECTS OF MICROSTRUCTURE AND STRESS RATIO ON THE FATIGUE CRACK GROWTH BEHAVIOR OF SHORT AND LONG CRACKS IN Ti-6Al-4V: *V. Sinha*[1]; C. Mercer[1]; W. O. Soboyejo[1]; [1]The Ohio State University, Department of Materials Science and Engineering, 177 Watts Hall, 2041 College Road, Columbus, OH 43210 USA

The effects of microstructure and stress ratio on the micromechanisms of short and long fatigue crack growth in Ti-6Al-4V are discussed in this paper. Short crack anomalies in equiaxed and Widmanstätten microstructures are related to underlying crack/microstructure interactions. The combinations of stress range and crack length associated with short crack behavior are also discussed within the context of a Kitagawa diagram. The micromechanisms of long fatigue crack growth in equiaxed and Widmanstätten microstructures are examined at different stress intensity factor ranges and stress ratios. Crack-tip deformation mechanisms are also elucidated by careful examination of transmission electron microscopy foils extracted from the tips of cracks in the near-threshold, Paris and high ΔK regimes.

5:10 PM

FATIGUE CRACK GROWTH AT HIGH MEAN STRESS IN Ti-6Al4V AT 1.5 KHZ: *David L. Davidson*[1]; [1]Southwest Research Institute, P.O. Box 28510, 6220 Culebra Road, San Antonio, TX 78228-0510 USA

Fatigue cracks have been grown at $0.6 < R < 0.8$ in vacuum and air environments and observed under high resolution conditions in the SEM. Crack opening displacements and deformation near the crack tip has been observed and measured for cracks growing in the 10-11 to 10-12 meters/cycle range. Crack growth is intermittent and plastic zones are very small. Crack opening loads were measured indicating that even at these high R values, crack closure still exists. Air environment increases crack growth rates by over 1000 times. Interaction with microstructural features occurs, but no generic behavior has been catalogued to date. Reduction of mean stress to zero results in reduced crack opening loads, increased cyclic plasticity at the crack tip and larger crack opening displacements.

GENERAL ABSTRACTS: Materials Processing

Sponsored by: TMS

Program Organizers: TMS, Warrendale, PA 15086 USA; David Bourell, University of Texas, Austin, TX 78712-1063 USA; Rusty Gray, Los Alamos National Lab, Dynamic Properties, Los Alamos, NM 87545-0001; Howard Sizek, Inco Alloys International, Mechanical Testing, Huntington, WV 25705

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October 12, 1998 — Location: O'Hare Hilton Hotel

Session Chair: Eric Taleff, The University of Texas at Austin, Aerospace Engineering and Engineering Mechanics, Austin, TX 78712-1085 USA

2:00 PM

MATERIALS-RELATED ADVANCES IN RE-BRAZEABLE SNM PACKAGES: *John J. Stephens*[1]; James D. Pierce[1]; Jeffrey G. Bobbe[1]; Charles A. Walker[1]; F. Michael Hosking[1]; Charles V. Robino[1]; [1]Sandia National Laboratories, Dept. 1833, PO Box 5800, MS0367, Albuquerque, NM 87185-0367 USA

Concepts for re-brazeable special nuclear materials (SNM) packages have been developed in detail at SNL/NM. The initial package

concept is based on the re-brazeable qualities of the Au-18Ni (AWS BAu-4 alloy) with flange surfaces of Ni-200. Whereas the overall container includes a 304L stainless steel body, the initial package concept also included the use of a higher melting point braze (AWS BNi-5 alloy) to braze the Ni-200 flange to a 304L stainless body. Recent advances in the re-brazeable package concept include: (a) evaluation of filler metals to permit Ni-200/304L welds, thereby eliminating the first-step BNi-5 braze; and (b) the use of braze alloys with process temperatures lower than the BAu-4 alloy. Mechanical properties for both welds and braze joints will be discussed. These data include the effect of multiple de-braze/re-braze cycles on joint strength. * 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.

2:25 PM

THE BEHAVIOR OF A319 DURING HEAT TREATMENT: *Matt L. Newman*[1]; Jonathan A. Dantzig[1]; Huseyin Sehitoglu[1]; [1]University of Illinois, Mechanical Engineering, 140 Mechanical Engineering Building, 1206 West Green Street, Urbana, IL 61801 USA

Desired weight savings and increased performance has led to the replacement of steel by aluminum alloys in many applications, particularly in the automotive field. Most aluminum alloys must undergo a heat treatment procedure after casting to attain the properties required. Often, this heat treatment involves a quench from high temperature in the solid solution phase domain. The large, rapid thermal deformations experienced during this quench can induce large-scale plastic deformation and distortion in the cast part, as well as significant residual stresses. We wish to reduce these effects using computational modeling. To accurately model the material behavior and resulting deformation during this quench, a material model must be used which correctly portrays the material properties at different stages of the process, including microstructural changes associated with precipitation. We describe experiments to develop and test material models of cast aluminum alloy A319. The material models are implemented in both analytical and finite-element solutions.

2:50 PM

DENSITY FIELD VISUALIZATION IN CONVECTING METALLIC ALLOYS: *Jean N. Koster*[1]; [1]University of Colorado, Dept. Aerospace Engineering Sciences, Boulder, CO 80309-0429 USA

Experimental verification of theoretical investigations into the behavior of liquid metal convective flows and solidification is required to validate analytical models and numerical simulation codes. A real time radioscopic density visualization system has been developed. The X-ray facility for opaque low Prandtl number fluid research will be described. Density changes as low as 10^{-3} g/cm^3 can be visualized by careful control of scatter radiation. The new capability is demonstrated with natural convection benchmark experiments in a narrow vertical layer of pure gallium melt. Good agreement between calculations and natural convection as well as Rayleigh-Bénard convection experiments is obtained. Upon applying a horizontal temperature gradient to a gallium melt alloyed with indium, the binary melt developed a vertical concentrational stratification and at small temperature gradients, heat transfer was by conduction only. Convective flow developed at a higher temperature difference, which may be termed “critical”. After reducing the temperature difference the thermosolutal convection breaks down and a conductive state reappears at ΔT. This threshold for onset of natural convection in binary Ga-In melts is in need of a theoretical explanation. An estimate of the observed chemical segregation has been calculated and compared to existing theory. The experimental findings have never been predicted in modeling efforts of similar situations including convection; possible reasons are discussed. Similar experiments were done with AlSb, showing unusual gravitational segregation. None of the visualized phenomena, flow pattern or interface shape, can be predicted with current numerical models.

3:15 PM

UTILIZING VIBRATION TO PROMOTE UNIFORM COMPOSITE GROWTH IN HYPERMONOTECTIC ALLOYS: *Richard N. Grugel*[1]; Shinwoo Kim[2]; Shinwoo Kim[2]; [1]Universities Space Research Association, c/o MSFC, MS ES-75, Huntsville, AL 35812 USA; [2]Hoseo University, Dept. of Materials Science and Engineering, Chung-Nam, Korea Korea; [2]Hoseo University, Dept. of Materials Science and Engineering, Chung, Nam Korea

Gravity driven separation, among other factors, precludes uniform microstructural development during processing of hypermonotectic alloys. It is well established that liquid/liquid suspensions, in which the respective components are immiscible and have significant density differences, can be established and maintained by utilizing ultrasound. A historical introduction to this work is presented with the intent of establishing the basis for applying the phenomena to promote microstructural uniformity during solidification processing of immiscible alloys. Experimental work based on transparent organics, as well as salt systems, will be presented in view of the processing parameters and related to a preliminary model. Support from NASA grants NAG8-1231 and NCC8-66 is gratefully acknowledged.

3:40 PM BREAK

3:55 PM

GLASS FORMATION AND CHARACTERIZATION OF AlNiFeRE ALLOYS BY MECHANICAL ALLOYING: *Weigang Meng*[1]; Davin O’Neill[1]; Gary Shiflet[1]; [1]University of Virginia, Dept. of Materials Science & Engineering, Thornton Hall, Charlottesville, VA 22903 USA

The glass formation of AlNiFeRE (RE = Ce, Nd, Sm, Gd, Dy, Ho, Er, and Y) alloys by mechanical alloying (MA) of elemental powders is investigated by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). MA technique is systematically used to identify a superior quaternary system (Al-Ni-Fe-RE). Gd, Sm and Y- containing systems are thoroughly mapped, yielding optimum compositions for amorphization. Measurements of half peak width vs. milling time from XRD patterns in selected systems are reported. The microstructural evolution of the milled powders is examined in detail by SEM and TEM. Initial milling produces layered microstructure of Al and RE (Gd and Sm) while further milling leads to amorphous powders with finely dispersed particles, in support of an inter-diffusion based mechanism of amorphization. Research supported by UVA-AEP and AFOSR.

4:20 PM

PULSED LASER WELDING OF TRICLAD METAL: A COMPARISON OF EXPERIMENTAL AND COMPUTATIONAL RESULTS: *Marcelino Essien*[1]; Joseph A Romero[1]; David Noble[1]; Adonis C. Kassinos[1]; Gerald A. Knorovsky[1]; [1]Sandia National Laboratories, Organization 1833, Mail Stop 1411, Albuquerque, NM 87185 USA

An investigation of laser spot welds made on Triclad, a roll-bonded composite of 0.004 inch copper between two 0.008 inch sheets of Kovar provides interesting insights into the penetration mechanisms that occur in pulsed laser welding. Bead-on-plate spot welds were produced using a 400 Watt pulse Nd:YAG laser, operating with pulse energies from 6 to 14 joules. The resulting weld cross sections were metallographically examined to study the degree of penetration, and the extent and characteristics of mixing that occurred during welding. Comparison of computational prediction and experimental results shows that the weld penetration is stable, predictable, and consistent up to the second copper/Kovar boundary, but becomes increasingly inconsistent and unpredictable beyond that point. The experimental results also show that considerable mixing occurs for laser pulse widths of 5-8 milliseconds. In contrast, a computational model that assumes conduction mode welding predicts that very little mixing occ urs on the time scale of the experimental laser pulse widths. The model incorporates surface tension driven fluid flow, but fails to predict the degree of mixing found experimentally. These results suggest that other mechanisms contribute strongly to or dominate the welding regime investigated in this study. The presentation will discuss the investigation and specific comparisons of experiment and computation, and will propose an alternate material transport mechanism. *This work was supported by the United States Department of Energy under Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy.

4:45 PM

MACRO-PARTICLE DISTRIBUTION AND DEPOSITION RATE VARIATION OF TiAlN COATING BY MAGNETIC FILTERED CATHODIC ARC DEPOSITION: *Y. W. Lee*[1]; M. S. Leu[1]; I. C. Hou[1]; W. C. Lih[1]; [1]Industrial Technology Research Institute, Materials Research Laboratories, Bldg. 52, 195 Chung Hsing Road Section 4, Chutung, Hsinchu, Taiwan 310 ROC

The TiAiN coatings deposited by magnetic solenoid filtered arc is studied by measuring the surface roughness, particle density and film thickness. With specimens located at different position in front of the coatings induced by the arc discharge is measured. The distribution of the particles is found to be a bell-shape with high particle density and surface roughness along the center axis of the target. Similar result is obtained for the film deposition rate by measuring the film thickness. The deposition rate and particle density are found to be a function of the strength of magnetic field. The deposition rate is increased by increasing the current of the magnetic coil. While the particle density of the film is apparently decreased by the current.

5:10 PM

STRIP-EDGE PHENOMENA IN THE GAS WIPING OF A CONTINUOUS HOT-DIP GALVANIZING LINE: *Jeong-Real Park*[1]; Sun-Ho Jeon[1]; Ro-Bum Park[1]; [1]POSCO, POSCO Technical Research Labs., Rolling Products Research Team, 699 Kumho-dong, Kwangyang, Chonnam 545-090 Republic of Korea

Turbulance of wiping gas at the edge of steel strip during gas wiping in a continuous hot-dip galvanizing line has generated noise and edge-overcoating problems. They were examined as a function of pressure and angle of purging gas to the strip and knife-to-strip distance. Level of noise decreased by avoiding collision of purged gas at the edge of strip and with decreasing pressure and increasing knife-to-strip distance. An employment of edge baffles resulted in the decrease of noise level by up to 10dBA. Overcoating of zinc on the edge area from edge to 50mm inside the strip was measured and analyzed. The overcoating was compared with edge drop of the cold-rolled steel substrate, resulting in edge build-up at the edge area from the edge to 10 to 30mm inside the strip if the edge drop could not accomodate the overcoating. The overcoating could decrease by up to 80% with edge baffles when the baffle-to-strip distance was adjusted to be 18mm or less.

HOT DEFORMATION OF ALUMINUM ALLOYS: Superplasticity, Creep, and Friction Welding

Sponsored by: Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Thomas R. Bieler, Michigan State University, Dept. of Matls Sci & Mechanics, East Lansing, MI 48824-1226; Lawrence Lalli, Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA; Stuart MacEwen, Alcan International, Kingston, Ontario K7L 5L9 Canada

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Session Chairs: Dr. Thomas R. Bieler, Michigan State University, Dept. of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; T. R. McNelley, Naval Postgraduate School, Monterey, CA 93943-5146 USA

2:00 PM INVITED PAPER

PROCESSING FOR HIGH-STRAIN-RATE SUPERPLASTIC ALUMINUM ALLOYS: *Mamoru Mabuchi*[1]; Kenji Higashi[2]; [1]National Industrial Research Institute of Nagoya, Hirate-cho, Nagoya, Kita-ku 462 Japan; [2]Osaka Prefecture University, College of Engineering, Department of Mechanical Systems Engineering, Gakuen-cho, Sakai, Osaka 599-8531 Japan

Effects of reduction, large second phase particles (including reinforcements) and dynamic precipitation on grain refinement by hot extrusion have been investigated in high-strain-rate superplastic aluminum alloys and their composites by tensile tests and microstructural investigation. The grain refinement was attributed to an interaction of recrystallization and dynamic precipitation, which was enhanced by large second phase particles (reinforcements). Not only fine-grained microstructure but also stress relaxation of stress concentrations which are caused at matrix/large second phase particle (reinforcement) interfaces during superplastic flow are required to attain large elongations. Partial melting resulting from segregation plays an important role in relaxing the stress concentrations. DSC investigations revealed that the degree of segregation was increased by hot extrusion.

2:30 PM

SUPERPLASTICITY OF 1N90 PURE ALUMINUM AND THE THEMOMECHANICAL PROCESSING: *Tsunemichi Imai*[1]; Sumito Kojima[2]; Daming Jiang[3]; Jianfu Mao[3]; Gilles L'Esperance[4]; Bande Hong[4]; [1]National Industrial Research Institute of Nagoya, 1 Hirate-cho, Kita-ku, Nagoya 462 Japan; [2]Nagoya Municipal Industrial Research Institute, 3-4-41 Rokuban-cho, Atsuta-ku, Nagoya Japan; [3]Harbin Institute of Technology, Harbin 150001 P.R. China; [4]Ecole Polytechnique de Montreal, P.O. Box 6079, Station A, Montreal, Quebec H3C 3A7 Canada

High Strain Rate Superplasticity (HSRS) is expected to establish an efficiently near net shape forming for metal matrix composites and mechanically alloyed (MA) aluminum alloys, but the deformation mechanism has not yet been understood entirely. Superplastic characteristics and the microstructure for 1N90 pure aluminum were investigated in order to reveal the deformation mechanism of the HSRS. 1N90 pure Al alone hot-rolled after being extruded with R=44 exhibits m values of 0.30~0.47 and a total elongation of 300~500% at strain rate of 0.01s-1 and at 893~913K. 1N90 pure Al consists of grains of about 10~20um and subgrain of 0.6~1.0um. It is, therefore, thought that dynamic recrystallization might contribute to promote

HSRS in addition with fine grain boundary sliding and interfacial sliding at semi-solid phase.

2:50 PM INVITED PAPER

EQUAL CHANNEL ANGULAR EXTRUSION-A TURN IN THE RIGHT DIRECTION: *R. E. Goforth*[1]; K. Theodore Hartwig[1]; Roy Cornwell[1]; [1]Texas A&M University, Mechanical Engineering Department, College Station, TX 77843-3123 USA

Equal Channel Angular Extrusion (ECAE) is an innovative deformation process capable of extremely large total deformation after multiple passes without a change in original billet cross-section. Although ECAE has many technological advantages, one of its primary benefits is the capability of creating a variety of microstructures and substructures including sub-micron grains. It is applicable to a wide variety of materials, e.g., pure metals, alloys, intermetallics, and polymers by cold, warm or hot working. Many materials have been extruded (ECAE) and evaluated at TAMU over the past several years. In most cases the primary objective was to attain a fine uniform grain structure throughout the billet material in order to improve the structural properties or to facilitate the manufacturing process, e.g., superplastic forming. In this paper, the ECAE research conducted on specific aluminum alloys (6061, 2095, 2195) with respect to microstructural evolution, recovery and recrystallization, grain refinement and superplasticity will be reported.

3:20 PM INVITED PAPER

PROCESSING OF ALUMINUM ALLOYS FOR HIGH STRAIN RATE SUPERPLASTICITY: Patrick B. Berbon[1]; Minoru Furukawa[2]; Zenji Horita[3]; Minoru Nemoto[3]; Nikolai K. Tsenev[4]; Ruslan Z. Valiev[5]; *Terence G. Langdon*[1]; [1]University of Southern California, Department of Materials Science and Mechanical Engineering, Los Angeles, CA 90089-1453 USA; [2]Fukora University of Education, Department of Technology, Munakata, Fukuoka 811-41 Japan; [3]Kyushu University, Department of Materials Science and Engineering, Faculty of Engineering, Fukuoka 812-81 Japan; [4]Ufa State Petroleum Technical University, Institute of Chemical Technology, Ufa 450062 Russia; [5]Ufa State Aviation Technical University, Institute of Physics of Advanced Materials, UFO 450000 Russia

Equal-channel angular (ECA) pressing is a processing procedure in which substantial microstructural refinement may be introduced into a material through intense plastic shearing. This paper describes an investigation of the effects of ECA pressing on an Al-Mg solid solution alloy and on a commercial aluminum alloy fabricated by casting. The results demonstrate the potential both for producing a very small grain size and for achieving superplastic tensile ductilities of up to > 1000% at high strain rates (above 10^{-2} s^{-1}).

3:50 PM INVITED PAPER

SUPERPLASTICITY OF ULTRAFINE-GRAINED Al-Mg-Sc ALLOYS PRODUCED BY EQUAL-CHANNEL ANGULAR PRESSING: Shogo Komura[1]; Patrick B. Berbon[2]; Atsushi Utsunomiya[1]; *Minoru Furukawa*[3]; Zenji Horita[1]; Minoru Nemoto[1]; Terence G. Langdon[2]; [1]Kyushu University, Fukuoka 812-81 Japan; [2]University of Southern California, Los Angeles, CA 90089-0241 USA; [3]Fukuoka University of Education, Munakata 811-41 Japan

An Al-3%Mg-0.2%Sc alloy with a grain size of ~0.2 mm was produced using equal-channel angular (ECA) pressing. Microstructural observations using transmission electron microscopy revealed that fine-grained structure with a grain size of a few micrometers was retained after annealing at 673 K for 1 h. The ECA pressed alloy exhibited large ductilities: >600 % and >1000% at testing temperatures of 573 K and 673 K, respectively, at an initial strain rate of 3.3x10-2 s-1. These results demonstrate the potential for using ECA pressing to produce superplastic structure.

4:20 PM

CREEP BEHAVIOR OF POWDER METALLURGY A1-6092 AND A1-6061 ALLOYS: *Yong Li*[1]; Terence G. Langdon[1]; [1]University of Southern California, Department of Materials Science and Mechanical Engineering, Los Angeles, CA 90089-1453 USA

Aluminum-based alloys fabricated by powder metallurgy (PM) represent a class of material which is different from Al alloys manufactured by ingot metallurgy (IM) procedures. The creep behavior of PM Al alloys has received considerable attention in recent years, primarily because these alloys are often used as the matrix materials in the production of metal matrix composites. This paper describes the results obtained in an investigation of the creep properties of a PM Al-6092 alloy. A comparison is made with published data obtained using a PM Al-6061 alloy.

4:50 PM

A HEAT FLOW MODEL FOR FRICTION STIR WELDING: *Zhili Feng*[1]; Jerry E. Gould[2]; *Thomas J. Lienert*[2]; [1]Edison Welding Institute, Engineering and Materials Team, 1250 Arthur E. Adams Drive, Columbus, OH 43221-3585 USA; [2]Edison Welding Institute, Resistance and Solid-State Welding Team, 1250 Arthur E. Adams Drive, Columbus, OH 43221-3585 USA

The friction stir welding (FSW) process is a recently developed process, largely exploited for welding of aluminum alloys. The process relies on the friction between the tool and the workpiece, as well as the high strain-rate deformation, to generate sufficient heat to soften the materials underneath the tool where extensive deformation takes place to form a solid state joint. In this regard, suitable temperature fields critically control the deformation process and subsequently the quality and properties of the weld. In this work, a heat flow model has been developed for friction stir welding. The model considers the heat generation from the tool shoulder and the heat conduction into the weldment. The model is based on the Rosenthal equation for a moving heat source, and numerically integrates over the heat generation region of the tool. Results of the model are presented, and correlation made with actual friction stir welds and temperature measurements. Results show that the temperature range in the stir zone of a quality weld generally falls within the optimum forging temperature range for aluminum alloys.

5:20 PM

DEFORMATION MICROSTRUCTURES IN FRICTION STIR WELDS ON 6061-T6: *Thomas J. Lienert*[1]; *Richard J. Grylls*[2]; Jerry E. Gould[1]; *Hamish L. Fraser*[2]; [1]Edison Welding Institute, Resistance and Solid-State Welding Team, 1250 Arthur E. Adams Drive, Columbus, OH 43221-3585 USA; [2]The Ohio State University, Materials Science and Engineering Department, 2041 College Road, Columbus, Ohio 43210 USA

Friction stir welding (FSW) is a relatively new joining process that is proving especially effective with Al alloys. FS welds are produced by traversing a rotating, non-consumable tool along a butt joint formed by two rigidly held plates. The tool is maintained in compressive contact with the plates, and localized heating results from the deformation produced by passage of the tool. FS welds were produced on 1/4" thick plates of 6061-T651 using optimized processing conditions. The results revealed the presence of several different weld zones with distinct microstructures. Microstructures of the various weld zones were characterized using optical microscopy with quantitative image analysis, SEM and TEM with EDS. Mechanical properties of the joints were assessed by microhardness and tensile tesing. The grain size, dislocation density and precipitate distributions in each zone will be discussed and related to the local microhardness as well as the estimated temperature, strain and strain rate fields of each zone . Microstructural features will also be related to those resulting from forging and extrusion of 6061-T6.

INTERSTITIAL AND SUBSTITUTIONAL SOLUTE EFFECTS IN INTERMETALLICS II: Nickel Aluminides

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee
Program Organizers: Ian Baker, Dartmouth College, Thayer School of Engineering, Hanover, NH 03755 USA; Easo P. George, Oak Ridge National Lab., Metals & Ceramics Division, Oak Ridge, TN 37831-6093 USA; Ronald D. Noebe, NASA Lewis Research Center, Cleveland, OH 44135 USA

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Session Chairs: M. H. Yoo, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA; M. A. Crimp, Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226

2:00 PM
EFFECTS OF DEVIATIONS FROM STOICHIOMETRY ON THE MECHANICAL PROPERTIES OF SINGLE-CRYSTAL Ni3Al: *Toshiyuki Hirano*[1]; Dmitri V. Golberg[1]; Masahiko Demura[1]; [1]National Research Institute for Metals, Mechanical Properties Division, 1-2-1, Sengen, Tsukuba, Ibaraki 305 Japan

Ni_3Al exhibits a positive temperature dependence of the yield stress, which is known as the yield stress anomaly. So far a number of models for this curious behavior have been proposed with considerable success. However, these models are based on the experimental data of ternary or off-stoichiometric n alloys. This is a problem because the effects of ternary additions and deviation from the stoichiometry can be reasonably expected in intermetallic compound, Ni_3Al. It is not easy to predict it from the data of ternary or off-stoichiometric alloys. The lacking of these data is attributed to the difficulty of single crystal growth because binary stoichiometric Ni_3Al solidifies incongruently. Recently, we have successfully grown single crystals for binary stoichiometric composition and single crystal-like ones for Al-rich composition, using a floating zone method. This enabled us to study the effect of deviation from the stoichiometry on the mechanical properties without influence of ternary additions. One of the interesting results is that the CRSS for the primary octahedral slip holds Schmid's law in stoichiometric composition but not in Al-rich composition. The former is quite different from the previous data in ternary and off-stoichiometric alloys.

2:30 PM
SOLUTE EFFECTS ON THE CREEP OF NiAl: *W. D. Nix*[1]; P. H. Kitabjian[1]; R. D. Noebe[2]; A. Garg[2]; [1]Stanford University, Department of Materials Science and Engineering, Stanford, CA 94305-2205 USA; [2]NASA, Lewis Research Center, Cleveland, OH 44135 USA

The high temperature creep strengthening effects of the substitutional solutes Ti, Hf and Zr in NiAl are reported and discussed. We show that these strengthening effects are significantly greater than those typically found in ordinary metallic solid solutions. Atomic size factors for Ti, Hf and Zr in NiAl are estimated from lattice parameter measurements. These factors indicate large solute size effects and suggest that solute-drag may be an important creep strengthening mechanism in these solid solutions. Evidence for such solute-drag strengthening is sought in the creep data for these materials and in the dislocation substructures found in creep-deformed crystals. Much of our attention is focused on the creep properties of single crystals of NiAl(Ti) containing 2.5 and 3% Ti. The compression creep properties of these crystals have been studied extensively in both the "hard" <001> and "soft <111> orientations at temperatures ranging from 850ΥC to 1250ΥC. The steady-state flow data exhibit a near 3rd power stress dependence at the highest temperatures and lowest stresses, indicating possible solute-drag control. In addition, the near 3rd power stress dependence breaks down at stresses sufficient to cause dislocations to pull away from their solute atmospheres, again supporting the solute-drag hypothesis. Also, the dislocation structures formed under creep conditions are similar to those found for solute-drag controlled creep of solid solutions. However, the strain rate transients associated with stress changes during creep are not consistent with solute-drag control. A model of deformation based on both substructure strengthening and solute-drag control is briefly outlined and shown to be consistent with many of the creep characteristics of these solid solutions. It suggests that both ordinary dislocation hardening and solute-drag may be responsible for the high creep resistance of these solid solutions.

3:00 PM
EFFECTS OF PURITY ON THE PROPERTIES OF BETA-NiAl SINGLE CRYSTALS: *M. J. Kaufman*[1]; J. Hu[1]; V. I. Levit[2]; [1]University of Florida, Materials Science and Engineering, Gainesville, FL 32611 USA; [2]AS&M, NASA Langley Research Center, Hampton, VA 23681 USA

High purity (HP) single crystals of β-NiAl were grown by the Bridgman technique using pure alumina crucibles and pure nickel and aluminum feedstock. Further purification was attempted using the containerless zone melting apparatus at the University of Tennessee and the results indicated that the Bridgman-grown crystals were of comparable purity before and after zone refining. These crystals contained essentially no metallic impurities and much lower silicon and carbon levels than the "conventional-purity (CP)" crystals used in the more recent studies of single crystal NiAl. As one measure of crystal purity, the 4.2K resistivity of the HP crystals was about half that of the CP material after annealing to minimize the point defect concentration. Furthermore, the mechanical property studies revealed that NiAl behaves similarly to pure metals, i.e., the higher the purity, the lower the critical resolved shear stress (CRSS) and the higher the tensile elongations that can be achieved. It will be shown tha t, in spite of the fact there appears to be no problem with either dislocation nucleation or mobility, the fracture remains brittle even after elongations exceeding 300%. The effects of heat treatment, deviation from stoichiometry, purity, test temperature, specimen geometry (including orientation), surface finish and prestrain on the tensile properties of NiAl will be summarized in some detail and the implications of these results on the potential use of this class of materials will be provided. This work is supported by The Air Force Office of Scientific Research (URI Grant F49620-93-0309) under the direction of Dr. Charles H. Ward.

3:30 PM
STATIC AND DYNAMIC STRAIN AGING IN MICROALLOYED NiAl-BASED ALLOYS: *M. L. Weaver*[1]; R. D. Noebe[2]; [1]University of Alabama, Department of Metallurgical and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; [2]NASA, Lewis Research Center, Cleveland, OH 44135 USA

Several manifestations of strain aging have been identified as playing a role in the deformation behavior of the intermetallic compound NiAl. These include the occurrence of sharp upper yield points, strain rate sensitivity (SRS) minimums, yield stress plateaus as a function of temperature, flow stress transients upon a change in strain rate, and serrated stress-strain curves. Until recently, however, the significance of strain aging and it's potential impact on the mechanical behavior of NiAl has been all but ignored. In this study, static and dynamic strain aging have been investigated in a series of NiAl-based alloys microalloyed with Zr and Si. The 0.2% offset yield stress generally decreased with increasing temperature for all of the alloys investigated. However, local plateaus or maximums were observed at intermediate temperatures for all of the alloys investigated. Very distinct SRS minimums were also observed with coincident flow stress transients upon an upward change in strain rate. These phenomena have been attributed to dynamic strain aging (DSA). Static strain aging (SSA) studies using the classical yield point return and stress relaxation techniques revealed that the time dependence of the strain aging events followed a $t^{1/3}$ relationship in alloys containing Zr and a $t^{2/3}$ relationship in Zr free alloys. This suggests that substitutional species are responsible for strain aging in Zr containing alloys while interstitial species dominate in Zr free alloys. The results are discussed with respect to recent observations of strain aging in binary NiAl alloys.

4:00 PM

SOLUTE HARDENING AND SOFTENING EFFECTS IN B2 NICKEL ALUMINIDES: *L. M. Pike*[1]; C. T. Liu[1]; I. M. Anderson[1]; Y. A. Chang[2]; [1]Oak Ridege National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA; [2]University of Wisconsin, Department of Materials Science and Engineering, Madison, WI 53706 USA

The effects of substitutional solute additions on the hardness of B2 ordered NiAl compounds were investigated. It was found that through careful control of metallurgical factors such as stoichiometry, solute size, and heat treatment effects ranging from rapid hardening to softening could be observed. In the case of Fe additions to NiAl, vacancy concentrations were determined using density and lattice parameter measurements. ALCHEMI experiments were performed to establish anti-site defect concentrations. By establishing these defect concentrations it was then possible to rationalize and quantitatively describe the observed hardening and softening effects. The hardening effects of other alloy additions to NiAl including Mn, V, and Pd were also investigated. This research was sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract number DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

4:30 PM

OBSERVATIONS OF DISLOCATIONS IN THE CRACK TIP REGION OF BULK 4-POINT BEND SPECIMENS OF SINGLE CRYSTAL NiAl ALLOYS USING ELECTRON CHANNELING CONTRAST IMAGING: *B. -C. Ng*[1]; M. A. Crimp[1]; [1]Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA

The electron channeling contrast imaging (ECCI) technique, performed in a scanning electron microscope, has been used to observe dislocations in the crack tip region and along crack edges of loaded and unloaded bulk 4-point bend specimens of single crystal NiAl alloys. Significant numbers of dislocations have been observed ahead of loaded and unloaded cracks in both commercial purity and high purity materials, suggesting that a degree of toughening as a result of dislocation generation at crack tips can occur in these materials. However, crack tip plasticity has been found to be more extensive in high purity NiAl, with extensive slip band activation found at arrested crack tips. Dislocations are distributed heterogeneously along the crack edges in both materials. In general, fewer dislocations were observed along the crack edges in regions where the cracks propagated in a fairly straight manner, whereas larger numbers of dislocations were found along the cracks at points where crack deflections occurred. This suggests that dislocation generation and subsequent toughening are inconsistent in both commercial and high purity materials. This work has been supported by the Office of Naval Research (Grant No. N00014-94-1-0204).

5:00 PM

MECHANISMS OF DISCONTINUOUS YIELDING IN A Nb-Ti-Al ALLOY: *Richard J. Grylls*[1]; R. Wheeler[2]; S Banerjee[3]; S Perungulam[1]; D.-H. Hou[1]; H.L. Fraser[1]; [1]The Ohio State University, Department of Materials Science and Engineering, 2041 College Road, Columbus, OH 43210 USA; [2]UES Inc., 4401 Dayton-Xenia Road, Dayton, OH 45432 USA; [3]Bhabha Atomic Research Centre, Bombay 400 085 India

Alloys based on the composition Nb-40Ti-15Al (at%) are being investigated for possible use in high-temperature corrosive environments. These B2-matrix alloys show an interesting combination of mechanical properties, including high yield strength maintained through 800°C, and room temperature ductility exceeding 30%. On aging this alloy at intermediate temperatures, a yield-point is observed on subsequent room-temperature tensile deformation. Also, discontinuous yielding is observed upon testing at intermediate temperatures. These phenomena have been investigated from both a mechanical and microstructural standpoint, and both areas will be addressed here. On testing at intermediate temperatures, a critical strain for the appearance of serrated yielding and a negative strain-rate sensitivity are observed, consistent with a Portevin-Le-Chatelier-type effect. The microstructural changes during yielding have been studied using transmission electron microscopy (TEM). Evidence will be presented which suggests that it is the dynamic precipitation and subsequent dissolution of the w-phase which is responsible for the reported effects. This work has been supported by the US ONR, Dr. George Yoder as Program Manager.

MECHANICAL PERFORMANCE OF LAMINATED COMPOSITES: Deformation Behavior

Sponsored by: Structural Materials Division, Structural Materials Committee

Program Organizers: Donald Lesuer, Lawrence Livermore Natl. Lab., Livermore, CA 94551 USA; Eric M. Taleff, The University of Texas, ASE/EM CO600, Austin, TX 78712

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Session Chairs: Chol K. Syn, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; Eric Taleff, University of Texas at Austin, Department of Aerospace Engineering and Engineering Mechanics, Austin, TX 78712 USA

2:00 PM INVITED PAPER

IN SITU HIGH VOLTAGE ELECTRON MICROSCOPY STUDY OF THE DEFORMATION AND FRACTURE OF NANO-LAMINATE MULTILAYER MATERILAS: *T. W. Barbee*[1]; M. A. Wall[1]; T. P. Weihs[2]; [1]Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; [2]Johns Hopkins University, Baltimore, MD 21218 USA

The microstructural scale of multilayer nano-laminate materials is consistent with the resolution capabilities of transmission electron microscopy. Therefore, in-situ observations of their stability and of other physical processes have the potential to identify mechanisms which define the relationships between these synthetic microstructures and their physical properties. In this effort we sought to directly observe in cross-section in the high voltage transmission electron microscope the plastic deformation and the failure of multilayer nano-laminate materials having periods of 5 nm to 500 nm. A special sample preparation process was developed that enabled the localization of deformation and fracture facilitating observation of the sample during deformation and fracture in the microscope. Both static and continuous dynamic imaging was performed so that the deformation and failure processes could be temporally recorded. The samples studied were synthesized by magetron sputter deposition and had total thicknesses of 25 to 250 nm. The material pairs in these synthetic microstructure samples were Cu/Zr, Al/amorphous(A) Al2O3, Cu/A CuxZr1-x , Al/Ti, and V/Si. Brittle failure of one set of layers and subsequent ductile failure of the other material layers was observed and continuously recorded video tapes of this process will be shown. These results will be related to scanning electron microscopy observations of fracture surfaces on "bulk" samples of the materials studied by this in-situ technique.

2:30 PM

SLIP PROPAGATION IN METALLIC MULTILAYERED THIN FILMS: *Peter M. Anderson*[1]; Tim Foecke[2]; [1]The Ohio State University, Department of Materials Science and Engineering, 2041 College Street, Columbus, OH 43210 USA; [2]National Institute of Standards and Technology, 2 Metallurgy Division, Gaithersburg, MD 20899 USA

Observations from a novel in situ TEM straining experiment on a Cu/Ni multilayered sample reveal details of dislocation generation and motion. Slip is seen to occur predominantly as the operation of interfacial Frank-Read like sources that produce spanning 'Orowan' bows within the layer. These propagate within the layer, forming an extremely intense pileup at the intersection of the slip band with the surface of the sample. Slip propagates from layer to layer not as the movement of a single array of dislocations, but rather as a series of saturation-renucleation events. Elementary models of dislocation arrays are applied to multilayered thin films to predict the critical stress required to propagate the 'Orowan' bows within individual layers and to model critical conditions for the propagation of slip from one layer to another.

2:50 PM

DISLOCATION STRUCUTRES IN NANO-LAMINATE MULTI-LAYER MATERIALS: *T. W. Barbee*[1]; M. A. Wall[1]; [1]Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

Transmission electron microscopy studies of a broad range of nano-laminate multialyer structures in our laboratory have revealed that the dislocation densities in the individual layers in these synthetic structures appears anomalously small. Our observations indicate that as the individual layer thicknesses increase beyond 80 to 100 nm the dislocation density in the layers may also start to increase. It is likely that image forces imposed on the dislocations in the layers by the very high interface concentration in these synthetic microstructure materials results in the motion of dislocations to the interlayer interfaces and intralayer grain boundaries. The dislocations may be annihilated at these surfaces or reside in the near vicinity being localized by the image forces. This behavior is believed consistent with the formation of deformation cell strucutre in ductile metals at moderate to high strains. These observations will be discussed and related to predictions of the effects of image forces on dislocations in fine particles having diameters in the range 50 to 500 nm.

3:10 PM

HIGH TEMPERATURE STABILZATION OF LAYERED GRAIN STRUCTURES IN METALS BY SELECTIVE ZENER PINNING: *Gerhard F. Welsch*[1]; Paul Szozdowski[1]; Ricard Collins[1]; [1]Case Western Reserve University, Cleveland, OH 44106, 2 RIST USA

Arrays of pinning particles incorporated along embedded thin layers inside a composite enable the formation of stabilized overlapping 'bricklayer' or 'pancake' grain structures upon recrystallization. Grain growth is permitted within the channels delineated by the pinning layers but not across the pinning layers. In 'plane stress' loading so-stabilized microstructures have the potential of being creep and fracture resistant. They may be applied to coatings, thin foils, thin walled metal structures, fibers and wires to enhance their creep and rupture strength properties. Acknowledgment: NSF-DMR 9315755.

3:30 PM BREAK

4:00 PM

MECHANICAL BEHAVIOR OF COLD ROLLED NANOSCALE BRASS-STEEL MULTILAYER COMPOSITES:SIGNIFICANCE OF TEXTURE AND LAYER THICKENESS ON STRENGTH: *F. H. Kavaranan*[1]; S. S. Sahay[2]; K. S. Ravichandran[1]; [1]University of Utah, Department of Metallurgical Engineering, 412 WBB, Salt Lake City, UT 84112-2 USA; [2]Tata Research Development and Design Centre, Pune India

Microstructure and mechanical behavior of nanoscale brass-steel multilayer composites made by cold rolling were investigated. The evolution of texture as a function of deformation was investigated by determining pole figures and measuring elastic moduli by a precision laser interferometric displacement measurement technique. The values of elastic moduli were correlated with the bilayer thicknesses (or the percentage cold-work) and the degree of texturing. Tensile strength and ductility at room temperature of these multilayers were also determined and correlated with the bilayer thickness and the degree of texture. The elastic modulus and strength increased dramatically with an increase in the percentage of cold rolling or a decrease in the bilayer thickness. It was found that due to heavy deformation, texture-induced strengthening was the other dominant strengthening factor in addition to the Hall-Petch type strengthening in these multilayers.

4:20 PM

MODELING THE MECHANICAL BEHAVIOR OF Al LAMINATED METAL COMPOSITES DURING HIGH TEMPERATURE DEFORMATION: *R. B. Grishaber*[1]; R. S. Mishra[2]; A. K. Mukherjee[2]; [1]W. L. Gore and Associates, Inc, Medical Products Division, 3450 W. Kiltie Lane, Flagstaff, AZ 86002-0500 USA; [2]University of California, Department of Chemical Engineering and Material Science, Davis, CA 44106 USA

A constitutive model for deformation of a novel laminated metal composite (LMC) which is comprised of 21 alternating layers of Al 5182 alloy and Al 6090/SiC/25p metal matrix composite (MMC) has been proposed. The LMC as well as the constituent or neat structures have been deformed in uniaxial tension within a broad range of strain-rates (i.e. 10-6 to 10+1 s-1) and moderate to high homologous temperatures (i.e. 0.3 to 0.95 Tm). The stress exponent, n, of the Al 5182 layers increases from 3 to 5 with increasing strain-rate (i.e. >10-3 s-1). The MMC layer's apparent n value decreases from 7 to 5 with increasing temperature. The results of these experiments have lead to a characterization of the neat layer's mechanical behavior and a semi-empirical constitutive rate equation for both the Al 5182 and Al 6090/SiC/25p, respectively. These predictive relations for the neat layers have been coupled with a proposed model which takes into account the dynamic load sharing between the elastically stiffer and softer layers when loaded in parallel during isostrain deformation of the LMC. This model has led to the development of a constitutive relationship between flow stress and applied strain-rate for the laminated structure which has been compared with experimentally determined data. This work is supported in part by a grant: NSF-DMI-96-15540.

4:40 PM

CREEP DEFORMATION OF COPPER/SOLDER BRICKLAYER COMPOSITES: Tai-Yuan Wu[1]; *Gerhard Welsch*[1]; [1]Case Western Reserve University, Cleveland, OH 44106 USA

Bricklayer composites made by solder-boding of rectangular copper coupons (the bricks) served as physical models for the study of the role of composite architecture on the creep deformation and strength. The solder is a relatively soft material that creeps whereas the copper bricks represent the stronger component that deforms either just elastically or plastically. Creep strength depends on the composites' architecture. Results from creep deformation studies will be presented that illustrate the effects of the widths, overlap lengths and the symmetry of arrangement of copper bricks and solder bond layers. Acknowledgment: NSF-DMR 9315755.

MECHANISMS AND MECHANICS OF COMPOSITE FRACTURE: Fiber Reinforced Composites

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

Program Organizers: Bhaskar S. Majumdar, UES, Inc., Dayton, OH 45432-1894 USA; S. Krishnamurthy, Allied Signals Inc., Phoenix, AZ 85034 USA; Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

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Session Chair: S. Krishnamurthy, Allied Signals Inc., Phoenix, AZ 85034 USA

2:00 PM KEYNOTE

Al/Al_2O_3 FIBER COMPOSITES THROUGH INTERFACE PARTICLE COARSENING: EXPERIMENTS AND MODELING: *Ali S. Argon*[1]; M. L. Seleznev[1]; C. F. Shih[2]; X. H. Liu[2]; [1]M.I.T., Cambridge, MA 02139 USA; [2]Brown University, Division of Engineering, Providence, RI 02912 USA

In Al alloy matrix composites reinforced with Al2O3 fibers, an effective means of optimizing axial strength and work of fracture in parallel with transverse strength and toughness is possible through coarsening of Al2Cu precipitates on fiber/matrix interfaces. This controls the all important ductile work of debonding by plastic cavity growth which affects all tensile properties. An approach combining experiments and micro-mechanical modeling will be described.

2:30 PM INVITED PAPER

FRACTURE, TENSILE STRENGTH AND STRENGTH VARIABILITY IN CONTINUOUS FIBER REINFORCED ALUMINUM MATRIX COMPOSITES: *Colin McCullough*[1]; [1]3M Company, MMC Program, St. Paul, MN 55144-1000 USA

Aluminum unidirectionally reinforced with 3M NextelTM 610 alumina fibers represents a simple example of a strongly bonded interface system. For this type of system it is believed that local load sharing mechanisms operate, and thus the strength is controlled by microstructural features present in very small volumes of the composite. As with all continuous fiber composite systems, there is a wide variation in the measured tensile strength. Examination of the fracture behavior shows the variation to be composed of both process related features and to variations in the constituent arrangement. These are characterized and presented as a hierarchy of microstructural factors that control the composite fracture and thus the composite strength and strength variability. Emphasis will be placed on the size of these features, the role of matrix phases, misaligned fibers, and uniformity of fiber arrangement.

2:55 PM INVITED PAPER

DAMAGE AND FRACTURE IN METAL MATRIX COMPOSITES: *W. A. Curtin*[1]; G. C. Foster[1]; [1]Virginia Polytechnic Institute and State University, Department of Engineering Science and Mechanics, Blacksburg, VA 24061 USA

The ultimate tensile strength in fiber-reinforced MMCs is controlled by the statistical strength distribution of the reinforcing fibers and the stress transfer from broken to unbroken fibers, which depends on the fiber/matrix interfacial mechanics. Recently, models of the tensile strength of composites have been developed which include local load transfer in a controlled manner. Here, we show that the predicted failure strength and its size scaling are largely independent of the fiber arrangement by comparing results for square, hexagonal, "random", and thin-tape arrays of fibers. The weak dependence of failure on fiber geometry arises because of the diffuse nature of the fiber damage prior to failure. We also consider the failure of thin tapes under pure bending, and show that the discrete nature of the fibers and the thin specimen width lead to a very stable progression of damage and substantially increased composite strength in comparison to failure under tension. In all cases, the predicted results are compared to experimental data and very good agreement is demonstrated.. G. Foster, Master's Thesis, VPI&SU (1998)

3:20 PM

FRACTURE OF METAL/CERAMIC LAMINATES:TRANSITION FROM SINGLE TO MULTIPLE FRACTURE: K. L. Hwu[1]; *B. Derby*[1]; [1]University of Oxford, Department of Materials, Parks Road, Oxford OX1 3PH UK

We have investigated the transition from single to multiple fracture in laminates of Al, Cu and Ni/alumina with a range of metal/ceramic thickness ratios. Multiple cracking occurs above a critical metal/ceramic thickness ratio, (tm/tc)crit with the critical value increasing as the ratio of constrained metal flow stress to mean ceramic fracture stress, sm/sc, decreases. As sm/scdecreases, the predictions of (tm/tc)crit. given by existing models of the transition from single to multiple fracture diverge significantly from experiment. These models use linear elastic fracture mechanics formulations, which ignore the effects of metal plasticity away from the plane of a propagating crack. We propose a modified model which describes the stress distribution in the ceramic laminates in the wake of a crack using a shear-lag formulation. The modified model is shown to predict better the transition from single to multiple fracture in the lower yield Al and Cu/alumina laminates.

3:45 PM BREAK

4:00 PM

INTERFACE EFFECTS ON LOAD SHARING BEHAVIOR IN TITANIUM MATRIX COMPOSITES UNDER LONGITUDINAL TENSION: *B. S. Majumdar*[1]; T. Matikas[2]; D. B. Miracle[3]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]University of Dayton Research Institute (UDRI), Dayton, OH 45469 USA; [3]Air Force Research Laboratories, WPAFB, OH 45433 USA

The mechanisms of failure of longitudinally loaded fiber-reinforced titanium matrix composites (TMCs) were investigated using single-ply and multi-ply composites with different types of coatings. Initially, fiber fragmentation tests were performed and analyzed using the elegant model of Curtin, to assess the effective shear strength of the interface under highly constrained conditions around a fiber break. Those results indicated very high friction stress (compared with a push-out test), as well as damage of the fiber coating. Single-ply tests indicated local load sharing behavior even for the weakest interface. Although such is expected for single-ply material, what was unexpected was the configuration of fiber breaks that were strongly influenced by macroscopic slip bands, with interface strength having an insignificant influence. Results from on-going experiments on multiply composites, as well as from single-ply composites at elevated temperatures, will also be presented. The strength and elongation data will be compared with the prediction of available models.

4:25 PM

MODELING OF MICROCRACK TOUGHENING IN TWO-PLASE MULTILAYERED MEDIA: *Peter M. Anderson*[1]; Sandeep Muju[2]; [1]Ohio State University, Dept. MSE, 2041 College Road, Columbus, OH 43210 USA; [2]Allied Signal Aerospace Engines, 111 S. 34th Street, Phoenix, AZ MS 201-228 USA

This work presents a numerical model of the toughening behavior exhibited when a Mode I macrocrack propagates through alternating tough and brittle layers of a composite. The toughening occurs due to microcracking in brittle layers. Microcracks nucleate at periodic flaw sites in brittle layers when the stress at a site is sufficient to propagate a crack within the layer. Established microcracks are modeled by a dislocation dipole, with Burgers vector chosen to render the normal component of stress equal to zero at the dipole midpoint. R-curves for layered media are predicted as a function of bi-layer thickness, volume fraction, density of flaw sites, and inhomogeneity in fracture toughness and elastic modulus. A critical combination of properties for the onset of R-curve behavior is identified, and features which optimize the upper plateau of the R-curve are discussed.

4:50 PM

MICROMECHANICS OF THE INTERFACIAL DEBONDING IN TITANIUM MATRIX COMPOSITES HAVING GRADIENT INTERPHASE: A. Sinharoy[1]; *R. B. Bhagat*[1]; [1]The Pennsylvania State University, Engineering Science and Mechanics Department, 226 Hammond Bldg., University Park, PA 16802 USA

The mechanisms of damage that are likely to occur in a composite, and the extent and sequence of their occurrence, depend upon the mechanics of damage initiation and propagation which are strongly influenced by the physico-mechanical characteristics of the fiber-matrix interface (interphase). This paper deals with the analysis of the debonding process considering the strain energy release rate (SERR) in a composite plate (SiC/Ti-15-3) with a transverse center through crack. The composite is modeled with distinct fiber, matrix and multilayered gradient interphase regions in the local area surrounding the crack-tip, and as homogeneous orthotropic away from the crack. For the simulated composite, the strain energy release rate at the debond reaches to 15% of that at the main crack before stress concentrations in the fiber adjacent to the debond leads to stresses high enough to cause fiber fracture. The SERR at the tip of a debond ahead of the main crack reached a maximum of 0.5 kJ/m2 with increasing debond length, while the SERR at the tip of the main crack was 3.35 kJ/m2, before the fiber ahead of the crack experienced stresses close to its tensile strength (3200 MPa). To obtain high tensile strength and fracture toughness, the predicted results are of significance in providing guidelines in the design of tailored interphase favoring interfacial debonding in the composites.

5:15 PM

EFFECT OF VOLUME FRACTION ON THE FRACTURE BEHAVIOR OF Nb-1%Zr/218W COMPOSITES AT ELEVATED TEMPERATURES: *S. V. Raj*[1]; Louis J. Ghosn[1]; [1]NASA Lewis Research Center, Materials Division, MS 24-1, 21000 Brookpark Road, Cleveland, OH 44135 USA

Nb-1Zr/218 W long fiber composites are candidate materials for fabricating components for molten lithium-cooled, space-based nuclear reactors. The present study was part of a larger investigation on optimizing the processing conditions for fabricating these composites and characterizing their elevated temperature tensile properties. Nb-1Zr/218 W composites containing 0 to 70 vol.% of 218 tungsten fibers were fabricated by arc-spraying and consolidated by hot-pressing and hot-isostatic pressing techniques. Tensile tests were conducted on pin and clevis specimens between 1400 and 1600 K under engineering strain rates varying between 1.5 x 10-5 to 1.5 x 10-3 s-1. Characteristics of the deformation behavior are discussed. Post-fracture analysis of the fracture surfaces and polished sections revealed that the fracture morphology was dependent on the volume fraction of the fibers and the test temperatures.

5:40 PM

CORRELATION OF TENSILE STRENGTH WITH FRACTURE MODES OF KAOWOOL AND SAFFIL REINFORCED 339 ALUMINUM: *William J. Baxter*[1]; Anil K. Sachdev[1]; [1]GM Research & Development Center, 30500 Mound Road, Warren, MI 48090 USA

The tensile strengths of 339 aluminum composites reinforced with randomly oriented Saffil or Kaowool fibers depend upon the interfacial bond strength. The Kaowool fibers form a strong bond so failure occurs in the matrix at 300ΥC and by fiber cleavage at 20ΥC. In the T5 condition the Saffil interface is weak and the transverse fibers delaminate. Thus, although Saffil is stronger than Kaowool, the T5 composites have the same tensile strength. A T6 heat treatment promotes an interfacial reaction with magnesium. This strengthens the Saffil interface so failure occurs primarily in the matrix. The reaction with Kaowool is extensive and weakens the matrix. When account is taken of the different fracture modes, together with the matrix strengths as determined by nanoindentation, the calculated composite strengths agree with experiment.

NEW & EMERGING APPLICATIONS FOR REFRACTORY METALS & MATERIALS: Refractory Metal Based Intermetallics

Sponsored by: Structural Materials Division, Refractory Metals Committee

Program Organizers: Joseph W. Newkirk, Univ of Missouri-Rolla, Dept of Metallurgical Engineering, Rolla, MO 65409-0340; Edward N.C. Dalder, Lawrence Livermore National Lab, PO Box #808, Livermore, CA 94551-0808

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October 12, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Edward Dalder, Lawrence Livermore National Labs, Livermore, California 94531 USA; Joseph W. Newkirk, U. of Missouri-Rolla, Dept of Metallurgical Eng., Rolla, Missouri 65409 USA

2:00 PM

WEAR RESISTANCE OF REFRACTORY METAL SILICIDE COMPOSITES: *Joseph W. Newkirk*[1]; [1]University of Missouri-Rolla, Dept. of Metallurgical Eng., 282 McNutt Hall, Rolla, MI 65409 USA

Silicides based on refractory metals can have a very high hardness, similar to ceramic materials, but with the high thermal and electrical conductivity of metals. This allows these materials to be machined by EDM, unlike ceramic materials. The toughness of these silicides can be improved by adding ductile second phases to the matrix. The resulting composites have good wear properties, good high temperature properties, and should have good corrosion resistance in many harsh environments. The processing and properties of silicides based on Cr and Ta will be presented, with emphasis on their wear resistance and some corrosion results.

2:30 PM

METHODS OF IMPROVING TOUGHNESS IN Cr-Cr_3Si COMPOSITES: *Joseph W. Newkirk*[1]; [1]University of Missouri-Rolla, Dept. of Metallurgical Eng., 282 McNutt, Rolla, MI 65409 USA

Chromium silicide has a very high elastic modulus, good oxidation resistance, good elevated temperature strength, and a lower density than nickel based superalloys. Applications for this material are limited by its low toughness at room temperature. Composites of Cr and Cr_3Si are thermodynamically stable and are tougher than the monolithic silicide. This paper describes efforts to improve the toughness of this composite through alloying and processing. Molybdenum significantly improves the toughness of the composites. The effects of alloying and other methods of improving the toughness will be discussed, and possible applications outlined.

3:00 PM

DECOMPOSITION OF THE BETA PHASE IN A Nb-25Ti-25Al ALLOY: *Keith J. Leonard*[1]; Vijay K. Vasudevan[1]; [1]University of

Cincinnati, Department of Materials Science and Engineering, 515 Rhodes Hall, PO Box 210012, Cincinnati, OH 45221-0012 USA

The phase transformations and low temperature phase equilibria resulting from the decomposition of the beta phase in Nb-25Ti-25Al, were examined as part of an investigation of the potential structural applications of Nb-rich alloys of the Nb-Ti-Al system. The as-cast microstructure revealed a primary β solidification path, further extending the known range of primary β solidification. Site occupancy preferences in the B2 ordered structure of the β phase, were examined through atom location channeling enhanced microanalysis (ALCHEMI). Subsequent decomposition of the β phase was observed at various temperatures and conditions. The existence of a β to σ + δ eutectoid transformation, was identified by differential thermal analysis and confirmed through microstructural analysis of heat treated samples. In conjunction with high temperature cooling experiments, electrical resistivity versus temperature measurements were conducted to further evaluate the eutectoid transformation and its cooling rate dependence. Isothermal treatments of the alloy at 1100, 900 and 700ºC were performed; a comparison of the results to those of calculated models are discussed. The formation and stability of orthorhombic Ti_2AlNB, which dominates the microstructure at low temperatures, was also evaluated. As a result of this research work, addition information on the phase transformations in the Nb-rich portion of the Nb-Ti-Al system will allow for further exploitation of these stable and metastable microstructures.

3:20 PM

SOLIDIFICATION PATHWAYS AND SOLID STATE TRANSFORMATIONS IN Nb-Ti-Al INTERMETALLIC ALLOYS: *Keith John Leonard*[1]; Joe C. Mishurda[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Materials Science and Engineering, 515 Rhodes Hall, P.O. Box 210012, Cincinnati, OH 45221-0012 USA

The Nb-Ti-Al liquidus surface has recently been re-examined as part of a larger study of the phase transformations and equilibria in the Nb-Ti-Al system. The as-cast microstructures of fifteen alloy compositions were characterized through optical microscopy, X-ray diffraction, differential thermal analysis, scanning electron microscopy and transmission electron microscopy. The beta primary solidification field, has been determined to extend further within the ternary system than those of the currently existing calculated and experimental liquidus surfaces. Differential thermal analysis has identified the existence of a β to σ + γ transformation in three alloys where it was not previously thought to exist; confirmation was provided by high temperature vacuum heat treatments in the single phase β region followed by oil quenching. The location of the boundaries between the β and the δ, σ and γ primary solidification fields have been redefined. Trends with the β phase with variations in composition were established for both lattice parameters and microhardness. As a result of this wider extent of the primary β solidification field, a greater possibility exists for microstructural control through thermal processing for alloys consisting of either σ + γ, β + σ, or β + δ.

3:40 PM

A STUDY OF PHASE EQUILIBRIA AND TRANSFORMATIONS IN THE Nb-Ti-Al SYSTEM: *Joseph C. Mishurda*[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, MS&E, ERC 560, ML 0012, Cincinnati, OH 45221-0012 USA

The Nb-Ti-Al phase equilibria has been re-examined as part of a larger investigation of Nb-Ti-Al intermetallic alloys. Eight alloy compositions were produced by arc melting. Differential thermal analysis (DTA) was initially used to examine alloy behavior with temperature. All transformation temperatures were determined and transformations tentatively identified. All samples were homogenized in the identified beta single phase field and rapidly quenched to retain as much of the metastable beta phase as possible. Heat treatments were then conducted at selected temperatures based on DTA results to characterize the phase equilibria and transformations within the system. The equilibrated structures were characterized by X-ray diffraction, metallography, scanning electron microscopy (BSEI), electron microprobe analysis and transmission electron microscopy. Ternary isothermal diagrams were established for 1360, 1270, and 1100(1090) °C. Close agreement was observed for the currently existing experimental phase equilibria, although the results differed from the calculated diagram by higher Ti solubility in the sigma and delta phases than predicted at lower temperatures (970 and 920°C). A beta to sigma + gamma eutectoid type transformation, passing through some of the alloys studied at temperatures in excess of 1180°C was observed to yield a desirable lamellar structure of sigma and gamma phases. A plethal section was found for the sigma + beta alloys, with a microstructure of sigma dispersed in a beta matrix and a desirable alloy composition was identified. The possibility of a four phase plane involving the O + beta +sigma + delta phases was found to occurr at 981°C.

PLASMA CASE HARDENING & RELATED PROCESSES FOR SURFACE MODIFICATION: Session II

Sponsored by: Materials Design and Manufacturing Division, Surface Modification & Coatings Technology Committee
Program Organizers: Krassimir Marchev, Barnett Institute, Northeastern University, Boston, MA 02115 USA; Clark V. Cooper, United Technologies Research Ctr, East Hartford, CT 02618 USA; Bill Giessen, Barnett Institute, Northeastern University, Boston, MA 02115 USA; Osman T. Inal, New Mexico Inst of Mining & Tech, Matl & Met Dept., Socorro, NM 87801 USA; Tali Spalvins, NASA, Lewis Research Center, Cleveland, OH 44135-3191 USA; Kumar Sridharan, University of Wisconsin, Madison, WI 53706 USA

Monday PM Room: London
October 12, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Bill Giessen, Barnett Institute, Northeastern University, Boston, MA 02115 USA; Kumar Sridharan, University of Wisconsin, Madison, WI 53706 USA

2:00 PM

THE APPLICATION OF PLASMA-ASSISTED SURFACE TREATMENTS - A COMMERCIAL HEAT TREATER'S EXPERIENCE: *Peter C. Lidster*[1]; Vladimir I. Gorokhovsky[2]; Deepak G. Bhat[3]; [1]Exactatherm Limited, Mississauga, Ontario L5S 1 C1 Canada; [2]Arcomac Plasma Processing Lab, Toronto, Ontario M6K 1W8 Canada; [3]UES, Inc., Dayton, OH 45432 USA

Many advances have taken place in recent years in the technology of plasma-assisted surface treatment of materials. The traditional plasma-assisted ion nitriding processes are reviewed from a historical perspective. The application of a newly emerging technology based on cathodic arc plasma surface engineering is discussed from the perspective of heat treatment applications. The vacuum arc discharge plasma has been used successfully in the last two decades for the deposition of hard coatings for cutting tools and machine parts. In this process, the jet of a highly ionized metal ion plasma, flowing from the cathodic arc spots, transfers the coating material from the target to the substrate surface. A significant disadvantage of this technology is the formation of droplets, also known as macro-particles, in the cathodic arc jets, which limit the application of the process to surface treatments that do not require high precision or surface finish. The filtered arc sources allow deposition of droplet-free coatings by deflecting the plasma flow along the curvilinear magnetic lines of force towards the substrate, while the droplets, having straight trajectories, are captured on the baffles. Thus, a fully ionized metal plasma flow is directed to the substrate to be coated. The vacuum arc cathode is also a theoretically unlimited electron emitter, thus providing an efficient source of high-density electron current. In this mode, it facilitates the generation of a uniform, high-density plasma cloud in the process chamber. This results in a "plasma-immersed" environment which provides a uniform condition for plasma ion etching, ion nitriding, low energy ion implantation and plasma-assisted chemical vapor deposition. Large area dual filtered cathodic arc sources (LAFAS|), recently developed by Arcomac, are now available to support the multi-stage surface treatment technology in a single process cycle. In this presentation, the highlights of this technology will be reviewed, and applications of this process for plasma heat treatment processes will be examined.

2:20 PM
PLASMA NITRIDING AND DLC COATING AS A DUPLEX TREATMENT FOR HIGH LOADED STAINLESS STEELS: *J. Olfe*[1]; T. Michler[1]; K. Bewilogua[1]; K. -T. Rie[2]; [1]Fraunhofer Institut fur Schicht- und Oberflächentechnik, Braunschweig Germany; [2]Institut fur Oberflächentechnik und Plasmatechnische Werkstoffentwicklung, Braunschweig Germany

Nitriding of stainless steels by means of plasma assisted diffusion treatment (PDT) is carried out to improve the hardness and wear resistance without affecting the corrosion properties. A lower coefficient of friction which is required in many applications is achieved by a subsequent coating with DLC. The present paper deals with the mechanical behavior of duplex treated stainless steels. Plasma diffusion treatment is carried out by means of the pulsed DC in a H2-H2 gas mixture. The DLC coatings are produced by DC magnetron sputtering and in a PACVD process using medium frequency. The influence of different nitriding conditions on the adhesion of DLC is determined. Furthermore, the influence of nitriding and duplex treatment on the fatigue life is investigated.

2:40 PM
PLASMA-ASSISTED COATINGS OF METALS: *O. T. Inal*[1]; Tezer Brattal[1]; John Hardy[1]; [1]New Mexico Tech., Materials and Metallurgical Engineering, Socorro, NM 87801 USA

Metallic coatings (Cu, Ni, Ti and Zr) have been deposited onto stainless steel and aluminum substrates in a 2 kV plasma at 30 m torr. The metals, in each case, are vaporized in the argon plasma which assists in their deposition to the substrate maintained at 200ϒ. It is believed that the excellent adhesion and high purity of these coatings are afforded by the superimposed plasma in this case. The above application pertains to having coatings that retard tritium permeation in the accelerator production of tritium (APT) design. This procedure also affords coatings for subsequent plasma treatment. In this regard, chromium and titanium coatings were deposited onto tool steel substrates and subsequently ion-nitrided to a uniform hardness. Details of the study will be given in the presentation.

3:00 PM
EFFECT OF POWER ON THE SPUTTER DEPOSITED Cr COATINGS: *Z. Fan*[1]; J. Seok[1]; M. Pakala[1]; R. Y. Lin[1]; [1]University of Cincinnati, Department of Materials Science and Engineering, M.L. #12, Cincinnati, OH 45221 USA

Coatings of Cr on steels have been investigated with RF and pulsed DC magnetron sputter deposition at ambient temperature with powers in the range of 2.19 and 8.77 W/cm2. X-ray diffraction analysis and microhardness measurements have been used to characterize the coatings. It was observed that increasing the sputtering power increased the coating hardness. However, due to the residual compressive stress, hillock formation was observed for coatings at high power deposition. X-ray diffraction analysis and TEM (Transmission Electron Microscope) analysis indicated that all as-deposited films were microcrystalline in nature and exhibited a BCC (body centered cubic) Cr phase. Pulsed DC deposition resulted in higher deposition rates than PF deposition It would be a desirable technique for industrial applications.

3:20 PM
SURFACE TREATMENT ON NON-FERROUS METALS BY PLASMA-ASSISTED CVD AND PLASMA NITRIDING: *Myoung-Jae Lee*[1]; Duk Jae Kim[1]; Jung Mi Hong[1]; Yoon-Kee Kim[1]; Hun Kyu Kim[2]; Keun-Ho Lee[1]; [1]Plasma Technology Center, Institute of Advanced Engineering, Yongin, Kyungki-Do 449860 Korea; [2]Daewoo Precision Industries, Ltd., Kumjeoung, Pusan Korea

The formation of nitrided layers on the non-ferrous metals such as titanium alloys and aluminum alloys has been very attractive process in the surface treatment industry. Although Ti, Al and their alloys possess excellent strength-to-weight ratio, their wear performance is not satisfactory, hence require surface hardening to achieve high physical stability. In the present work, TiN layers were developed on Ti-alloys by pulsed DC plasma-assisted CVD after plasma nitriding It was shown that the nitrided Ti-alloys significantly improved their wear resistance and surface hardness. In the case of Al-alloys, low process temperature was required due to their low melting points. The processed Al-alloys resulted in a significant increase of the resistance against wear and corrosion. To develop optimal process conditions, optical emission spectroscopy (OES) and mass spectroscopy were used for the characterization of the dissociation processes of nitrogen in the plasma. A number of techniques including SEM and XRD were conducted for the analyses of surface morphology and microstructure of the TiN and AIN layers.

3:40 PM BREAK

4:00 PM
CORROSION RESISTANCE OF ION NITRIDED POWDER METAL PARTS: *Pramod Kotwal*[1]; Paul Nowil[1]; [1]Nitron Inc., Lowell, MA USA

The use of sintered powder metal parts made out of inexpensive iron powder containing small amounts of copper is increasing due the economics of near net shape production. Ion nitriding increases the wear resistance of these parts while causing no distort ion. Ion nitrided parts are expected to show a higher corrosion resistance, thus increasing the incentive to use them in new applications. In this study we will ion nitride parts in Nitron's systems to create a thin Epsilon layer of Iron nitrides, document the kinetics of nitriding and evaluate the influence of ion nitriding on corrosion resistance.

4:20 PM
PLASMA NITRIDING UNIFORMITY IMPROVEMENT BY FINITE ELEMENT ANALYSIS MODELING: Leon Duo[1]; *Tony Wu*[1]; [1]Diesel Technology, HT Business Unit, Kentwood, MI 49512 USA

Plasma nitriding technology is challenged by high volume automotive components in uniformity and consistency. It has been found that plasma power (or current density) distribution is a key variable for nitriding uniformity. Finite Element Analysis (FEA) modeling was used to optimize plasma current distribution FEA results along with actual tests, showed that plasma power lead location is vital to the plasma uniformity.

4:40 PM
NONDESTRUCTIVE EVALUATION ON PLASMA NITRIDATION IN Fe-Cr SYSTEM: *Tatsuhiko Aizawa*[1]; Hideyuki Kuwahara[2]; [1]University of Tokyo, Department of Metallurgy, 7-3-1 Hongo, Bunkyo, Tokyo 113-8656 Japan; [2]Institute of Applied Science, Tanaka-ookubochou 49, Kyoto 606 Japan

Authors have been developing a new quantitative nondestructive methodology for elastic characterization and diagnosis of nitrided surface structure by using the acoustic spectro-microscopy. This methodology was successfully applied to materials evaluation on the gas-nitrided Fe-Cr system; elastic properties for each nitrided layer can be obtained through the multi-layered media model, and the growth of nitrided layers can be traced by the change of dispersion curves for the surface wave velocity. In the present paper, this method is further modified and promoted to deal with the plasma nitrided structure, where the volume fraction of nitride precipitates is continuously distributed in the matrix. The unknown elastic properties of a nitride precipitate and its distribution of volume fraction are determined by the inverse analysis in order that the measured dispersion curves of the surface wave velocity should coincide with the theoretical one within the prescribed tolerance of error. Fe-Cr system is employed to experimentally demonstrate the validity and effectiveness of the developed methodology. In parallel with description on the difference of nitridation mechanism between the gas and the plasma nitridation, the observed nitrided structure is compared with the predicted system by the present model.

5:00 PM
SOURCE OF MULTICOMPONENT ATOMIC FLUXES: *I. N. Churkin*[1]; V. I. Volossov[1]; A. G. Steshov[1]; [1]Budker Institute of Nuclear Physics, Novosibirsk Russia

Source of atomic fluxes on the basis of the trap with crossed fields is considered. Source can generate intensive polyenergetic fluxes of various atoms including metals and gasses. Accelerated atoms are formed from accelerated by radial electrical field within plasma volume ions by means of their charge-exchanging. Application of atomic fluxes for surface treatment is discussed in detail. Different surface treatment modes (implantation at high flux intensity and accelerated atom beam assisted deposition and magnetron sputtering) depend on operating modes of the source.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VII: Mechanical Alloying

Sponsored by: Structural Materials Division, Structural Materials Committee
Program Organizers: T. Srivatsan, Univ of Akron, Dept. of Mech. Engr., Akron, OH 44325-3903; K. A. Khor, Nanyang Technological University, Sch of Mech & Prod Engrg, Singapore 639798 Singapore

Monday PM — Room: Chicago
October 12, 1998 — Location: O'Hare Hilton Hotel

Session Chairs: Dr. J. W. Newkirk, University of Missouri-Rolla, Metallurgical Engineering, Rolla, MO USA; Dr. H. Zoz, Zoz Gmbh, Wenden D-574S2 Germany

2:00 PM INVITED PAPER
METAL INJECTION MOLDING OF MECHANICALLY ALLOYED POWDERS: *Joseph William Newkirk*[1]; [1]University of Missouri-Rolla, Metallurgical Eng., 282 McNutt Hall, Rolla, MI 65409-0340 USA

Advanced Materials can be fabricated by mechanical alloying. However, the small size of the mechanically alloyed powders makes them poorly suited for many consolidation methods. Metal Injection Molding (MIM) has the capability of making net-shape parts with a high final density. This paper describes research on the compatibility of mechanical alloying with MIM. Powders of 17-4PH stainless steel and NiTi intermetallic alloys have been produced by mechanical alloying. These powders have been injection molded, debound, and sintered. Results of feedstock characterization, injection molding parameters, sintering and final mechanical properties will be presented.

2:30 PM INVITED PAPER
MECHANICAL ALLOYING OF Fe-BASED SOLID LUBRICANT COMPOSITE POWDERS: *D. Ernst*[1]; H. Weiss[1]; H. Zoz[2]; [1]University of Siegen, Inst. of Materials Technology Laboratory of Surface Engineering, Siegen D-57068 Germany; [2]Zoz GmbH, Wenden D-57482 Germany

Tribological problems are of very high interest in modern research fields of mechanical engineering. In order to obtain low friction coefficients and an efficient self lubrication behaviour under high and low temperature conditions as well as under vacuum which is problematic in case of using conventional oil lubricants, several alloying systems were produced and investigated with respect to their structural properties. As previous experiments have shown, the mechanical alloying process in a ball mill is a convenient technique to get highly dispersed phase distributions on a nanocrystalline scale. Therefore the powder processing was carried out in a Simoloyer (Zoz - horizontal rotary ball mill) to achieve a homogeneous phase distribution of the solid lubricants in a strong metallic matrix. The chosen solid lubricant powders were pure graphite (C) and molybdenum disulfide (MoS2) combined with elemental iron (Fe) powder in various concentration ranges. The evolution of the microstructure was followed by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray diffraction (XRD) methods. Differential scanning calorimetry (DSC) will be applied to get further information about the stability and crystallization behaviour of the powders under the influence of increasing temperature.

3:00 PM INVITED PAPER
SEMI-CONTINUOUSLY MECHANICAL ALLOYING IN A PRODUCTION SCALE USING CYCLE OPERATION - PART II (ZOZ-HORIZONTAL ROTARY BALL MILL) - SEMOLOYER CM100S.: *H. Zoz*[1]; D. Ernst[1]; R. Reichardt[1]; T. Mizutani[2]; H. Okouchi[2]; [1]Zoz Gmbh, Wenden D-57482 Germany; [2]Fukuda Metal Fuil & Powder Co. Ltd., Kyoto 607 Japan

The production of large quantities of powders for industrial application, e.g., in paints or soldering materials is an aim followed by Fukuda Metal Foil and Powder Co. Ltd. in Japan. For these application, Cu-and Ag-particles with a special geometry (flakes) are needed. Based on milling experiments resolved by the Simoloyer CM-1-1/2 l with a grinding unit capacity of 0.5 l for laboratory purpose, a new grinding device has been developed using the same principle. This Simoloyer CM100s1, suitable for a semi-continuously production of mechanically alloyed and mechanically particle deformated powders, has been designed and already been described in part I of this work. Part II focuses on testing for and on industrial application of the processing and the equipment. The batch operation procedure has already been done in Germany in September 1997. The efficiency of the system was even much higher than expected. The well-known problems when processing CMB-materials (ductility etc.) were solved by using the Cycle Operation procedure controlled by the Maltoz-software. A production capability of 600 kg of ready product per day with this one machine only has been achieved. The testing regarding the semi-continuous operation will be done in Kyoto in November 1997 and consequently these data will be available for the current paper. If, what is expected, the semi-continuously procedure and the use of the corresponding equipment will be successful and the main processing parameters can be kept, the expected continuous capability is calculated to be about four tons of powder per day. The already proved application in the batch process is already a revolutionary step for Fukuda, for the process and for mechanical alloying. Depending on the coming work, it will be even more.

3:30 PM
IMPROVED Ag-SnO_2 ELECTRICAL CONTACT MATERIAL PRODUCED BY MECHANICAL ALLOYING: H. Zoz[1]; *D. Ernst*[1]; N. Spath[2]; [1]Zoz GmbH, Wenden Germany; [2]Engelhard-CLAL, Noisy-le-Sec 93130 France

Silver cadmium oxide used as a conventional materials for electrical contacts and other electrical components during a couple of years exhibits a lot of advantageous properties like a good thermal conductivity, a low contact resistance, a low welding force and a high ability arc quenching [1]. However, especially concerning the problem of the toxicity of cadmium oxide, the application of this materials will be reduced to a minimum in the near future and has to be replaced by a suitable materials with similar but minor harmful properties. A very promising candidate for such an application is represented by the system silver tin oxide [2]. Following a new processing technique [3], the basic powder component Ag-SnO_2 was produced on the powder metallurgical route by reactive milling. The starting powders - the silver tin alloy Ag_3Sn and silver oxide (Ag_2O) - were mechanically alloyed in a Simoloyer (Zoz - horizontal rotary ball mill) in a specific concentration ratio. During processing, a chemical reaction takes place which leads to a high dispersed phase distribution of nanoscaled SnO_2-particles in a silver matrix. Due to the applied temperature conditions and the milling parameters a completed reaction can be observed. Being compacted and sintered to a dense bulk material and brazed on copper contacts the silver tin oxide should exhibit the above mentioned propertied which are currently tested by further experiments. Results will be available in the near future. A characterization of the mechanically alloyed and sintered powders will be done by optical and scanning electron microscopy (SEM) and also by X-ray diffraction (XRD) studies.

4:00 PM
MECHANICALLY ALLOYED AND REACTIVELY SINTERED TI-AL COMPACTS USED BY THE PRODUCTION OF TiAl BASED INTERMETALLIC MATRIX COMPOSITES: *Sedat Ozbilen*[1]; [1]Gazi University, Metallurgy Education Department, Tenik Okullar, Ankara Turkey

Ti-Al powder mixtures with different Ti contents were mechanically alloyed by a high energy apex ball mill. Resultant powders was reactively sintered at different temperatures. The microstructure and properties of the reactively sintered IMC (TiAl-Ti) compacts were determined by SEM, XRD and destructive testing. Results will be presented to explain the encouraging ductility levels observed in this class of composite materials after mechanical alloying and reactively sintering cycle.

4:20 PM
Ti-TiN-TiB MMC'S BY REACTIVE MECHANICAL ALLOYING (RMA): *Sedat Ozbilen*[1]; [1]Gazi University, Metallurgy Education Department, Tenik Okullar, Ankara Turkey

Reactive mechanical alloying of TiH_2-B (sample #1), Ti-TiH_2-B (sample #2) powder mixes and mechanical alloying (MA) of Ti-B (sample #3) powder mix for 10 uninterrupted hours with sealed vial was carried out under nitrogen. This was followed by annealing of the mechanically alloyed powders under purified Ar to 1200ΥC for thermal treatment to determining the stability of nitride and boride formations. Representative samples of each powder mix mechanically alloyed and thermally treated were characterized by XRD using CuK radiation and examined by Cambridge ST40 Stereoscan SEM operating under 25kV to determine morphology and crystal structure change of the powders. It is observed that reactive mechanical alloying under N_2 cause to the formation of TiN in different levels in all the samples studied. Annealing of the mechanically alloyed powders under purified Ar to 1200ΥC for 1 hour leads to the formation of Ti-TiN-TiB multi-phase material in samples (albeit with different ratios.) This shows that sintering of the MA'ed powders of TiH_2-B, Ti-TiH_2-B and Ti-B is a promising way for producing ultrafine grained Ti-TiN-TiB composite material with reactive mechanical alloy processing under N_2.

4:40 PM
TiN FORMATION IN THE TiH2-B SYSTEM DURING REACTIVE MECHANICAL ALLOYING TO PRODUCE Ti-TiN-Ti)2-TiB METAL-MATRIX COMPOSITES: *Sedat Ozbilen*[1]; [1]Gazi University, Metallurgy Education Department, Tenik Okullar, Ankara Turkey

Reactive mechanical alloying (RMA) of TiH2-B powder mix for ten hours with sealed vial was carried out under N2 and air together with MA under purified Ar for comparison. Reactive mechanical alloyed samples were then examined by EDAX analysis for their oxygen and nitrogen contents. This was followed by annealing of mechanically alloyed powders under purified Ar to 1200ΥC to determine the stability of nitrides, oxides, and borides forming. Powder samples mechanically alloyed and sintered were characterized by XRD and SEM. It is observed that RMA cause to the formation of TiN when it is carried out under N_2; to that of (Ti,O) N with RMA under air. The f.c.c. (Ti,O)N phase forms as a result of the preferential reaction of nitrogen with Ti to form TiN with which any oxygen present can then react to form (Ti,O)N. This indicates reversed reaction kinetics of Ti with oxygen and nitrogen during RMA. Mechanical alloying under purified Ar prevents the formation of any Ti-nitride or Ti-oxy-nitride. Annealing under purified Ar to 1200ΥC for one hour leads to the formation of Ti-TiN-TiO_2-TiB with RMA under air; and only Ti-TiB with mechanical alloying under purified Ar.

5:00 PM
MECHANICALLY ALLOYED AND REACTIVELY SINTERED TiH_2-Al COMPACTS TO PRODUCE IMC (TiAl-Ti) MATERIALS: *Sedat Ozbilen*[1]; [1]Gazi University, Metallurgy Education Department, Tenik Okullar, Ankara Turkey

TiH_2-Al powder mixtures with different TiH_2 contents were mechanically alloyed (MA) by a high energy apex ball mill. Resultant powder were reactively sintered at different temperatures. The microstructure and properties of the reactively sintered IMC(TiAl-Ti) compacts were determined by SEM, XRD, and destructive testing. Results will be presented to explain the encouraging findings observed in this class of composite materials after mechanical alloying, thermohydrogen processing and reactively sintering cycle.

TEXTURE IN FILMS AND COATINGS: Session II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Texture & Anisotropy Committee
Program Organizer: J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

Monday PM — Room: Zurich/Tokyo
October 12, 1998 — Location: O'Hare Hilton Hotel

Session Chair: Hasso Weiland, Alcoa Technical Center, Alcoa Center, PA 15069

2:00 PM INVITED PAPER
TEXTURE INFLUENCE ON PROPERTIES OF FILMS: *J. A. Szpunar*[1]; [1]McGill University, Department of Metallurgical Engineering, 3610 University Street, Montreal, QC H3A 2B2 Canada

Texture influences various physical, chemical and mechanical properties of films. Often, the relationship between properties and texture can be easily formulated as the case of elastic properties or magnetic properties, like the magnetocrystalline energy or magnetostriction. In these cases, the anisotropic properties of a single crytal are averaged over the orientation distribution function to obtaining an average property for a given direction of the specimen. However, there are also correlations between the texture and properties that are not at all obvious nor fully understood. Examples of the different types of properties of films that are affected by texture are presented. In particular, texture influence on oxidation resistance of nickel, properties of magnetic films electromigration failure in electronic interconnects, arid the corrosion properties of zinc coatings is discussed.

2:35 PM INVITED PAPER

MODELS FOR TEXTURE AND MICROSTRUCTURE EVOLUTION DURING CVD DIAMOND FILM GROWTH: *Fnu Paritosh*[1]; Corbett Battaile[1]; D. J. Srolovitz[1]; P. Smereka[2]; [1]University of Michigan, Dept. of Materials Science and Engineering, 3074 H.H. DOW, 2300 Hayward Av, Ann Arbor, MI 48105 USA; [2]University of Michigan, Dept. of Mathematics, Ann Arbor, MI 48105 USA

The temporal evolution of the texture, microstructure and morphology of polycrystalline diamond films during chemical vapor deposition have been simulated. The surfaces of polycrystalline diamond films exhibit {111} and/or {100} facets or {10} and {11} facets in the two dimensional analog. The ratio of the growth rate of these two surfaces determines the characteristic crystal shapes or idiomorphs. The simulation proceeds by tracking the evolution of junction where pairs of facets meet. Both the average grain size and surface roughness increase as the square root of the film height h. The film texture increases rapidly as the film grows. The dominant texture component is a function of the ratio of the facet velocities. The preferred orientation and the exposed surface facets can be predicted in terms of idiomorphs. An analytical model that self-consistently accounts for the grain size and texture evolution will be presented.

3:10 PM

CONTROLLING THE SURFACE TEXTURE OF NICKEL FOR HIGH TEMPERATURE OXIDATION INHIBITION: *F. Czerwinski*[1]; J. A. Szpunar[1]; [1]McGill University, Dept. of Metallurgical Engineering, Montreal, H3A 2B2 Canada

The possibility of controlling the crystallographic texture of Ni surface to increase the resistance against high-temperature oxidation is studied Polycrystalline Ni coatings and toils with the <100>, <110> and <111> textures were manufacturing an electrodeposition technique. The initial textures arid microstructures of Ni were modified using annealing in an inert atmosphere of argon Textures of a variety of NiO scales grown on the electrodeposits at 1073 K in air were measured and compared to the textures of the corresponding Ni substrates. It was found that NiO grown on as-deposited Ni has a random distribution of grain orientations for all types of the substrate textures. When Ni electrodeposits were annealed prior to oxidation, there was a strong influence of substrate texture on the texture of NiO. In particular, a surface treatment composed of the electrodeposition of Ni primer with a strong <l00>texture followed by annealing leads to an Ni layer with a <111> texture and a low density of structural defe cts. Such a substrate of Ni promotes the formation of epitaxially oriented, slow growing and highly protective NiO scales with the <111> texture.

3:30 PM

AFM STUDY OF SURFACE TOPOGRAPHY AND MORPHOLOGY OF A NITROGEN PLASMA TREATED STEEL : *F. Mahboubi*[1]; G. Collins[2]; [1]Amir Kabir University of Tech., P.O. Box 15875, -4413, Tehran Iran; [2]Australian Nuclear Science and Technology Organization (ANSTO), Private Mail Bag 1, Menai, NSW 2234 Australia

Nitriding of steels using plasma environments has been practiced for many years. A lot of effort has been put into developing new methods, such as plasma immersion ion implantation (P1), which uses high ion energy bombardment, and low pressure radio frequency plasmas for mass transfer of nitrogen into the surface of the workplace. This article presents results obtained from an in-depth investigation of the surface texture of samples treated by P13 and RF plasma nitriding techniques using an atomic force microscope. It particularly discusses layer formation mechanisms in these processes. Samples of a microalloyed steel were treated by PP and RF plasma nitriding for 5 hours at temperatures ranging from 350 to 550C in a 75%N2-25%H2 atmosphere. The morphology and texture of the surface of the samples treated by P13 were totality different from those obtained by the other process. This was attributed to the fundamentally different nature of the processes.

3:50 PM BREAK

4:00 PM

Si-N COATING ON CARBON MATERIALS BY ION PLATING METHOD: *Yutaka Sato*[1]; *Saburo Ohtani*[1]; Yao-Can Zhu[1]; Nobuya Iwamoto[1]; [1]Ion Engineering Research Institute Corporation, 2-8-1, Tsuda-yamate, Hirakata, Osaka 573-0128 Japan

Si-N coating by ion plating method was applied to improve oxidation/corrosion resistance of carbon materials. Si-N thin film of 3 micrometer thickness was coated on graphite and C/C composite. Nitrogen content in Si-N film was increased up to ca.50at.% with the increase of voltage applied to anode and also discharge current. Obtained Si-N film was amorphous and crystallized to alpha silicon nitride after heat treatment at 1373 to 1773K in nitrogen atmosphere. Si-N film with low nitrogen content of 20at.% showed crystallized Si phase after the heat-treatment. In Si-N film with 50at.% nitrogen content, no Si phase was determined and beta silicon nitride formation was observed after 1773K heat-treatment.

4:20 PM

TEXTURE AND MICROSTRUCTURE CONTROL IN ZINC COATED STEEL SHEETS, EFFECT ON FORMABILITY OF THE COATINGS: *S. Gaignard*[1]; S. Lazik[1]; J. Wegria[2]; M. Dubois[3]; M. J. Philippe[1]; C. Esling[1]; [1]Metz University, LETAM URA CNRS, 2090, Metz, Cedex BP 80794 - F-57012 France; [2]Union Miniere, Research, B-, 2250 Olen; [3]Cockerill Sambree, B, -4400, Flemalle France

Many commercial galvanized steel sheets have been characterised before deformation (grain sizes, grain distributions in the coatings, pre-cracking, individual orientations misorientations between neighbouring grains, local global textures) in relation to the zinc alloy which was used and the process parameters. The samples have been deformed by a biaxial expansion and examined after deformation. All the coatings are pre-cracked before deformation. The extent of the pre-cracking is dependent both on the intensity and the sharpness of the coating basal texture and on the boundary misorientations. In the same way, zinc primary dendrites and recrystallized grains can hinder the crack propagation, limiting their extent. A clear correlation can be made between texture and microstructure of the coatings (using a new concept that corresponds to the free mean path of a crack) and also with the formability. As we know the process parameters, it is now easy to control the texture of the coatings.

4:40 PM

THE MICROSTRUCTURAL CHARACTERIZATION OF ELECTROGALVANIZED ZINC-IRON AND ZINC-NICKEL COATINGS: *H. Park*[1]; J. A. Szpunar[1]; [1]McGill University, Department of Metallurgical Engineering, 3510 University Street, Montreal, QC H3A 2B2 Canada

In this study, the development of the microstructure of zinc-iron and zinc-nickel coatings on steel sheet electrodeposited at various deposition conditions is analyzed. The evolution of the coating microstructure, morphology, crystallographic lattice, microstress, alloy composition and phase of coatings is investigated at different current densities and deposition times. The microstructure of zinc alloy coatings varies significantly with the change of deposition parameters. It is proposed that the transformation of texture during the alloy codeposition is closely related to the morphological characteristics of the coating surface and the change alloy and phase composition of the deposit. Anomalous codeposition behavior is considered responsible for the change or transformation of the microstructure of castings. The texture of zinc-iron coatings changes from the combined texture of the basal fiber and non-fiber pyramidal component to the pyramidal fiber texture with the current density increase. In the case of zinc-nicke1 coatings, the intensity of the {110} texture of γ phase increases with increasing nickel content while the intensity of the {100} texture decreases. A correlation between the texture, morphology and alloy content in the deposits is discussed.

5:00 PM

TEXTURE ANALYSIS OF TAPE CAST CERAMICS BY EBSD: *Frederic Cosandey*[1]; P. Markondeya Raj[1]; W. R. Cannon[1]; [1]Rutgers

University, Dept. of Ceramic and Materials Engineering, College of Engineering, Piscataway, NJ 08854-8065 USA

This work reports results on the effect of shearing during tape casting on texture development in ceramic materials. High spatial resolution Electron Backscatter Diffraction (EBSD) in a field emission SEM has been used for orientation determination of sub-micron particles and for texture analysis. During tape casting, the degree of particle orientation has been found to vary with the shear rates and solid loading. This alignment of particle major axis in the flow direction changes significantly materials properties as well as anisotropic sintering rates and anisotropic shrinkage. It has been found that texture development during tape casting is caused primarily by particle shape anisotropy and has been observed for different crystal systems such as alumina, titania and ferrite.

DISCONTINUOUSLY REINFORCED ALUMINUM-COMPOSITES: PRESENT & FUTURE: Materials and Processes

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

Program Organizers: Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433; Warren H. Hunt, Aluminum Consultants Group, Inc., Murrysville, PA 15668 USA

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Session Chair: Dr. Warren H. Hunt, FASM, Aluminum MMC Consortium, FASM, Murrysville, PA 15668-2002 USA

2:00 PM

GRAPHITIC ALUMINUM METAL MATRIX COMPOSITES: *P. K. Rohatgi*[1]; R. Q. Guo[1]; [1]University of Wisconsin-Milwaukee, Department of Materials, P.O. Box 784, Milwaukee, WI 53201 USA

This paper presents a background of the field of cast metal matrix composites and an overview of processing, casting characteristics, structure, properties, and potential applications of graphitic aluminum metal matrix composites. Nickel-coated graphite particles are incorporated into a) aluminum-silicon carbide composites melts to synthesize Al-Si-SiC-Al_3Ni-graphite composites and b) aluminum-alumina composite melts to synthesize Al-Al_2O_3-Al_3Ni-graphite composites with improved casting characteristics, thermal properties, machinability, and wear resistance. These composites appear to be promising cast materials for several applications.

2:30 PM

HIGH TEMPERATURE, HIGH STRAIN DEFORMATION OF SiC REINFORCED ALUMINUM COMPOSITES: *J. Malas*[1]; Prof. H. Rack[2]; [1]Air Force Research Laboratory, Wright-Patterson Air Force Base, OH 45433 USA; [2]Clemson University, FASM, Department of Ceramic and Materials Engineering, 208 Rhodes Hall, Clemson, SC 29634-0907 USA

The successful fabrication of finished and semi-finished products from SiC reinforced Al billet requires careful control of strain, strain rate and temperature variables. This presentation will discuss the application of Dynamic Material Modeling for establishing this process control strategy. Stable and unstable flow will be defined based on the strain rate and temperature sensitivity of the flow stress at high strains and correlated with the microstructural changes [dynamic recovery, dynamic recrystallization, reinforcement cracking and flow localization] that have been observed in SiC reinforced Al composites during high temperature, high strain deformation. Examples will be given for both particulate and whisker reinforced composites contained within Al-Mg, Al-Mg-Si and Al-Cu-Mg-Si matrices.

3:00 PM

MACHINING Al/SiC METAL MATRIX COMPOSITES: *John Pfeiffer*[1]; [1]Institute of Advanced Manufacturing Sciences, Machining Xcellence Division, 1111 Edison Drive, Cincinnati, OH 455216-2112 USA

One barrier to wider application of discontinuously reinforced aluminum (DRA) metal matrix composites is the perceived difficulty of machining these materials. This presentation summarizes the best techniques, cutting tools, and operating parameters for machining silicon carbide-reinforced aluminum. The influence of material characteristics (matrix and reinforcement) and the process for evaluating the economics of machining these materials are also discussed.

3:30 PM

JOINING OF DRA COMPOSITES: *Thomas J. Lienert*[1]; [1]Edison Welding Institute, 1250 Arthur E. Adams Drive, Columbus, OH 43221 USA

The effective integration of discontinuously-reinforced aluminum (DRA) composites into useful structures often mandates that they be joined to similar and dissimilar materials. The purpose of this presentation will be to review the current understanding of joining of DRA. First, general joining considerations for DRA will be outlined. Subsequently, the advantages/disadvantages associated with joining of DRA with fusion welding processes, solid-state joining processes and brazing processes will be discussed via the use of case studies, and recommendations on joining of DRA will be given.

4:00 PM

EVOLVING RESEARCH AND DEVELOPMENT DIRECTIONS IN DRA MATERIALS ENGINEERING: *John J. Lewandowski*[1]; [1]Case Western Reserve University, Dept. of Materials Science and Engineering, 10900 Euclid Avenue, Cleveland, OH 44106-7204 USA

DRA materials have been successfully integrated into a number of first generation products. A number of other fruitful areas exist for such materials due to the ability to tailor their properties to the intended use. A review of some of the evolving research and development directions which includes topics such as low cost processes and toughened products will be covered.