

Technical Program

1998 TMS FALL MEETING

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'98 TMS Fall Meeting

O'Hare Hilton Hotel

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| | Zurich/Tokyo | Texture in Films & Coatings I | Texture in Films & Coatings II | Texture in Films & Coatings III | | GA-Phase Transformations | | |
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Technical Program

MONDAY AM

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; ¹Carnegie 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 as to 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; ¹University 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 Li¹; ¹University 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 parameters 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. Ojha¹; L. Yu¹; J. Reye¹; S. N. Tewari¹; S. N. Tewari¹; ¹Cleveland 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. Morral¹; ¹University 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. Dayananda¹; *Yong-Ho Sohn*¹; ¹Purdue University, School of Materials Engineering, 1289 MSEE Building, West Lafayette, IN 47907 USA

Multiphase diffusion was investigated in the Fe-Ni-Al system at 1000°C 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 [gamma] 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 Mitao¹; *Leonid A. Bendersky*²; ¹NKK Corporation, Advanced Materials Engineering Lab., Fukuyama 721 Japan; ²NIST, 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-DRAW ON THE GROWTH OF PROEUTECTOID FERRITE IN Fe-C-Mn ALLOYS: *Masato Enomoto*¹; ¹Ibaraki 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. Hackenberg¹; G. J. Shiflet¹; ¹University 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 re-examining 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 Snyder¹; Jens Alkemper¹; Peter W. Voorhees¹; ¹Northwestern 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. Wei¹; ¹Lehigh 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. Arora²; E. S. Dwarakadasa³; ¹University of Utah, Metallurgical Engineering, 135 South 1460 East Rm 412, Salt Lake City, UT 84112 USA; ²Indian Institute of Science, Department of Mechanical Engineering, Bangalore, Karnataka, 560 012 India; ³Indian 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 ($\beta = 90^\circ$) and two mixed mode I-II ($\beta = 60^\circ$ & $\beta = 45^\circ$) 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 $\beta = 90^\circ$ (pure mode I) to $\beta = 0^\circ$ (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 $\beta = 60^\circ$ is higher than that under $\beta = 45^\circ$. Roughness induced crack closure alters the kinetics of near threshold crack propagation significantly in both $\beta = 60^\circ$ & $\beta = 45^\circ$. Oxide induced crack closure is also observed under $\beta = 60^\circ$.

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. Wei¹; ¹Lehigh 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 100 μ m. 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; ¹IIT, 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. Thouless²; John W. Halloran³; ¹University of Texas at Austin, Department of Mechanical Engineering, Building ETC, Rm. 9.158, MC C2201, Austin, TX 78712 USA; ²University of Michigan, Mechanical Engineering and Applied Mechanics Department, 2282 G.G. Brown Building, Ann Arbor, MI 48109-2125 USA; ³University 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. Murty¹; V.N. Shah²; ¹North Carolina State University,

Department of Nuclear Engineering, Raleigh, NC 27695 USA; ²Idaho 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 delta-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. Ludrosky¹; John J. Lewandowski¹; ¹Case 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 Chen¹; ¹National 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 dislocation is located at the interface, the stress field, stress intensity factor 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. Sikaddour¹; M. Temar¹; S. Zidemel¹; ¹University 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 re-austenitise 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 Wieszorek¹; Paul Bonniwell²; William Baeslack³; Daniel Evans⁴; Daniel Evans⁴; Hamish Fraser¹; ¹The Ohio State University, Dept. Mater. Sci. Eng., 2041 College Rd, Columbus, OH 43210 USA; ²The Ohio State University, Dept. Ind. Sys. Welding Eng, 2041 College Rd, Columbus, OH 43210 USA; ³The Ohio State University, College of Engineering, 2070 Neil Ave, Columbus, OH 43210 USA; ⁴Wright 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. Hultgren¹; Charles A. Greene¹; *Sreeramamurthy Ankem*¹; ¹University 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. Pollock¹; ¹Carnegie 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. Gouma²; G. B. Viswanathan²; Glenn S. Daehn²; Michael J. Mills²; ¹Metals Technology Inc., 19801 Nordhoff Street, Northridge, CA 91324 USA; ²Ohio 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. Fuller¹; David N. Seidman¹; David C. Dunand¹; ¹Northwestern 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 Al₃Sc precipitates within coarse aluminum grains, as observed by transmission electron microscopy and optical microscopy. Tensile creep properties of the dispersion-strengthened Al-Sc alloys were investigated in the temperature range of 200 - 350° 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. Pollock¹; ¹Carnegie 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 1~2% 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 Matić*¹; Richard K. Everett¹; Robert J. Bonenberger²; Anthony J. Kee³; ¹Naval Research Laboratory, Mechanics of Materials Branch, Code 6382, Washington, DC 20375 USA; ²FM Technologies, Inc., 10529-B Braddock Road, Fairfax, VA 22032 USA; ³Geo-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; ¹The 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 dis-

location. 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; ¹Oregon 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 T_m, 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 Jensen*¹; M. T. Lytle¹; N. Hansen¹; ¹Riso 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. Godfrey¹; ¹Sandia 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; ¹FAMU-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. Lloyd²; ¹Uni-

versity of British Columbia, Centre for Metallurgical Process Engineering, Department of Metals and Materials Engineering, 309-6350 Stores Road, Vancouver BC V6T 1Z4 Canada; ²Alcan 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. Morris¹; ¹University 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. Morris¹; ¹University 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 multi-pass 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. Savage¹; R. Srinivasan¹; R. D. Noebe²; T. Lograsso³; M. S. Daw⁴; ¹The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; ²NASA, Lewis Research Center, Cleveland, OH 44135 USA; ³Ames Laboratory, 111A Metals Development, Ames, IA 50011 USA; ⁴Clemson 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 \leq 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 $\langle 001 \rangle$ 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; ¹University 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 treat-

ments, 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 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. Pike¹; ¹Oak 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 reproducibilities 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. Li¹; H. Xiao¹; O. Klein¹; C. Nelson¹; R. L. Carleton²; E. P. George²; ¹Thayer School of Engineering, Dartmouth College, Hanover, NH 03755 USA; ²Oak 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, $D_s/(D_c \times \epsilon)$ increases with increasing aluminum concentration, but when vacancies are present (> 45 at. % Al), $D_s/(D_c \times \epsilon)$ 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. Yoo¹; S. Hanada²; ¹Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA; ²Tohoku 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. Sockel²; ¹Siemens AG, Power Generation (KWU), 45466 Mülheim a.d. Ruhr, F.R. Germany; ²Universität 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 ($D0_3 \longleftrightarrow B2$; $B2 \longleftrightarrow A2$), 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 1000°C and 1200°C, related to disordering processes due to the diffusion of Ni- and Al-atoms. These results are in good agreement with investigations on high γ' -[Ni₃(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. George¹; R. H. Zee²; ¹Oak Ridge National Laboratory, Metals and Ceramic Division, Oak Ridge, TN 37831-6093 USA; ²Auburn 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 $\sim 10^3$ 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 H₂O is more detrimental than H₂ 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, Lawrence 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. Bloyer¹; K. T. Venkateswara Rao¹; R. O. Ritchie¹; ¹Lawrence 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. Lewandowski¹; S. Soly'vev¹; W. A. Zinsser¹; ¹Case 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. Carpenter¹; W. W. Liang²; G. H. Paulino²; Z. A. Munir¹; Jeffery C. Gibeling¹; ¹University of California, Department of Chemical Engineering and Materials Science, One Shields Avenue, Davis, CA 95616 USA; ²University 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 arrester 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. Rigney¹; R. W. Bush¹; J. Teply¹; J. J. Lewandowski²; C. K. Syn³; D. R. Lesuer³; ¹Alcoa Technical Center, (Now at General Electric Aircraft Engines, Cincinnati, OH 45215-6301), Alcoa Center, PA 15069 USA; ²Case Western University, Cleveland, OH 44106 USA; ³Lawrence 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. Mao¹; ¹The 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 μm 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. Ye¹; W. O. Soboyejo¹; ¹The 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 Zhao¹; Peter K. Liaw¹; Ronald Belardinelli²; David C. Joy³; Charlie R. Brooks¹; N. Allen Yu⁴; Carl J. McHargue¹; ¹University of Tennessee at Knoxville, Dept. of Materials Science and Engineering, Dougherty Engr. Bldg., 1512 Middle Dr., Knoxville, TN 37996-2200 USA; ²Northrop Grumman, 9314 W. Jefferson Blvd., Dallas, TX 75211 USA; ³University of Tennessee, Dept. of Biochemistry, Cellular and Molecular Biology, 232 Sci. & Engr. Facilities, Knoxville, TN 37996 USA; ⁴University 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

October 12, 1998

Location: O'Hare Hilton Hotel

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. Moody¹; ¹Sandia 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. Nairn¹; ¹University 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; ¹Purdue 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; ¹University 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; ¹University 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; ¹Air Force Research Laboratories, Materials and Manufacturing Directorate, WPAFB, OH 45433 USA; ²UES, 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; ¹The 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; ¹Sandia National Laboratories, Livermore, CA 94551 USA; ²University 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; ¹Brown 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. Smith¹; ¹Argonne 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 Sastri¹; ¹Surmet 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. Svedberg¹; ¹AMPS, 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. Briant¹; ¹Brown 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 $\langle 110 \rangle \{001\}$ 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 $\langle 110 \rangle \{001\}$, $\langle 110 \rangle \{111\}$, and $\langle 110 \rangle \{112\}$. Annealing of rolled samples produced a texture in which the $\langle 111 \rangle$ direction was parallel to the normal to the rolling plane. Drawing of tantalum produced a texture in which the $\langle 110 \rangle$ direction was parallel to the drawing direction.

10:00 AM

THE MECHANICAL PROPERTIES OF HIGH PURITY CHROMIUM: Clyde L. Briant¹; ¹Brown 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. Chan¹; David L. Davidson¹; ¹Southwest 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 has 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; ¹University 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; ¹Los Alamos National Laboratory, Materials Science & Technology Div., P.O. Box 1663, Mail Stop G770, Los Alamos, NM 87545 USA

A Mo-W¹⁸² 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 W¹⁸² 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; ¹Oak 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; ¹H. 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; ¹Elatec 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; ¹Northeastern University, Barnett Institute, Boston, MA 02115 USA; ²Northeastern University, Chemistry Department, Boston, MA 02115 USA; ³United 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 316L 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 BCl₃-H₂-Ar ATMOSPHERE: *E. Rodriguez Cabo*¹; *K. T. Rie*²; ¹Volkswagen AG, Zentrallabor, Wolfsburg Germany; ²Institut für Oberflächentechnik und 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; ¹Implant Sciences Corporation, Wakefield, MA 01880 USA; ²Colorado State University, Department of Mechanical Engineering, Fort Collins, CO 80523 USA; ³Colorado School of Mines, Physics Department, Golden, CO 80401 USA; ⁴Hughes 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 500°C are 1.21 eV/atom and $2.5 \times 10^{-3} \text{ cm}^2/\text{s}$, while ion implantation boriding yields 0.76 eV/atom and $7.0 \times 10^{-8} \text{ cm}^2/\text{s}$ 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; ¹Institut für 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-boriding 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 900°C 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 IHK 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; ¹Diesel 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; ¹Northeastern University, Barnett Institute, Boston, MA 02115 USA; ²Advanced Heat Treat Corp., Monroe, MI 48162 USA; ³United 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 1450°F. 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 sur-

faces 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 Kotwal¹; Paul Nowil¹; 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. Meletis¹; ¹Louisiana 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. Varin¹; J. Bystrzycki¹; ¹The*

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 severe 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) L1₂ 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 Oey¹; R. Mahapatra¹; S. K. Varma¹; W. E. Frazier²; ¹The University of Texas at El Paso, Department of Metallurgical and Materials Engineering, El Paso, TX 79968-0520 USA; ²Naval 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 1000°C 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. Manoharan¹; G. Muralidharan²; ¹Nanyang Technological University, Division of Materials Engineering, School of Applied Science 639798 Singapore; ²Institute 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. Zhang¹; V. Shah¹; J. M. K. Wiezorek¹; H. A. Lipsitt¹; G. Meyrick¹; H. L. Fraser¹; F. C. Gresing²; ¹The Ohio State University, 2041*

College Road, Columbus, OH 43210 USA; ²Brush 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 been 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. Li¹; M. B. Robinson¹; T. J. Rathz²; G. Williams²; ¹NASA/Marshall Space Flight Center, Space Sciences Laboratory, Huntsville, AL 35899 USA; ²University 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. Srivatsan¹; P. C. Lam¹; M. Manoharan²; ¹The University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA; ²Nanyang 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 Ibrahim¹; Sainsiah Sulaiman¹; Mazli Mustapha¹; Patthi Hussain¹; Azmi Idris¹; ¹Advanced 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. Boutorabi¹; ¹Iran 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 austenitized at 850, 900 and 956YC, and austempered in the temperature range 300Y-450YC, 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 austenite 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. Sadrnezhaad¹; ¹Sharif 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. Seidel¹; M. Stauber¹; W. Dreier²; ¹Dornier GmbH, Daimler-Benz Aerospace, Friedrichshafen D-88039 Germany; ²Deutsches Zentrum für 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. Egrý*¹; G. Lohoefer¹; S. Schneider¹; I. Seyhan¹; B. Feuerbacher¹; ¹German 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. Samwer¹; ¹University 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 led 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 $6 \times 10^{-5} \text{ K}^{-1}$ measured on earth in our laboratory in the highly undercooled state of $\text{Zr}_{65}\text{Al}_{17.5}\text{Cu}_{17.5}\text{Ni}_{10}$. 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. Sagel²; C. Ettl²; H. -J. Fecht²; ¹Technical University of Berlin, Institute for Metallic Materials - Metal Physics, 36, PN 2-3, D-10623, Hardenbergstr, Berlin Germany; ²University of Ulm, Faculty of Engineering, 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-Moritz¹; D. M. Herlach¹; D. M. Matson²; M. C. Flemings²; ¹German Aerospace Center (DLR), Institute of Space Simulation, DLR, Wb-RS, Köln D-51170 Germany; ²Massachusetts 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. Morton²; M. B. Robinson³; R. J. Bayuzick¹; ¹Vanderbilt University, Department of Chemical Engineering, Nashville, TN 37235 USA; ²Teledyne Advanced Materials, Laverne, TN USA; ³Space 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 UNDERCOOLED Fe-Cr-Ni STEEL ALLOYS IN MICROGRAVITY: *Douglas M. Matson*¹; Merton C. Flemings¹; ¹Massachusetts 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 METALS UNDER MICROGRAVITY CONDITIONS: RESULTS FROM MSL-1: *Robert W. Hyers*¹; Gerardo Trapaga¹; ¹MIT, 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; ¹German 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 principle 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. Yalosome¹; ¹University 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 non-destructively 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 organized after a film thickness of 1.0 μm 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 metallic films.

9:05 AM

EPITAXIAL POTASSIUM NIOBATE THIN FILMS: *Barbara M. Nichols*¹; *Bruce W. Wessels*¹; ¹Northwestern 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 Ji¹; ZhenQiang Ma¹; ¹University 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 50Y; 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. Szpunar¹; ¹McGill 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 polycrystalline 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. Czerwinski¹; J. A. Szpunar¹; ¹McGill 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 Lee¹; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 730-701 Korea

Textures and microstructures of Ni electrolytically deposited 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. Recrystallization texture is compared with texture of as-deposits.

10:55 AM

TEXTURE FORMATION IN ELECTRODEPOSITED COATINGS: *Oleg B. Girin*¹; ¹State Metallurgical Academy of Ukraine, Dnipropetrovsk 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 behavior of axial texture and texture-related substructure anisotropy is dealt with in relation to electrocoating crystal lattice type, supercooling in electrocrystallization, and deposition 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. Kalinushkin¹; Oleg B. Girin¹; ¹State 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 electrocrystallization on quantitative characteristics of texture and texture-related 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; ¹Seoul 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 <100>. The result cannot be explained by a 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. Field¹; ¹TexSEM 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 diffusive 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; ¹General 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 litera-

ture 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; ¹Lockheed 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; ¹FASM 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; ¹Air 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; ¹Aluminum 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.

Technical Program

MONDAY PM

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. Mason¹; G. C. Kaschner¹; S. R. Chen¹; ¹Los 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 stability, 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 suppression 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; ¹University 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. Vecchio¹; *Aashish Rohatgi*¹; George T. Gray²; ¹UC San Diego, Materials Science Group, Dept. of Applied Mechanics, La Jolla, CA 92093 USA; ²Los 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. 10⁹ /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-Lücke 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. Gray¹; S. Hecker¹; D. Thoma²; D. Korzekwa²; A. Patterson²; P. Dunn²; ¹Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA; ²Los 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 martensite formation, and similar to shape-memory behavior. The stress-strain response of U-6Nb under quasi-

static uniaxial tension 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. Molinari¹; Y. Estrin²; S. Mercier¹; ¹LPMM, Universite de Metz, F-57045, Metz, Cedex France; ²The 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

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DEFORMATION BEHAVIOR OF TANTALUM AT HIGH AND LOW STRAIN RATES: Christopher Bull¹; Clyde L. Briant¹; David Lassila²; ¹Brown University, Division of Engineering, P.O. Box D, Providence, RI 02912 USA; ²Lawrence 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 Chen¹; Bin Zhang¹; Binling Zhou¹; ¹The 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.

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COMPARISON BETWEEN HIGH STRAIN RATE AND LOW STRAIN RATE DEFORMATION IN TANTALUM: Rajeev Kapoor¹; Sia Nemat-Nasser¹; ¹UCSD, 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 Milella¹; ¹ANPA, 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 $\dot{\epsilon}$. 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 $\dot{\epsilon}$ deriving what is known as the mechanical equation of solid state $\sigma_y = f(\dot{\epsilon}, T)$. 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 $\ln\epsilon$ 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: $\sigma_y = A(\exp B/T \epsilon^{C/T})$ (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 $\ln\epsilon$ 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 $\ln\epsilon$ plot. The coefficients of the equation of this line will provide the B and C values.

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THE DYNAMIC COMPRESSIBILITY OF SOFT SOILS IN THE PRESSURE RANGE OF 0.05-1.5 GPa: *Anatoly M. Bragov¹; Andrey K. Lomunov¹; Sergey A. Lomunov¹*; ¹Research Institute of Mechanics, State University of Nizhny Novgorod, 23, Gagarin Ave, Nizhny Novgorod, Nizhegorodskaya 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. Gratias¹*; ¹LEM-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 Dahmen¹*; ¹Lawrence 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-ACO3-76SFOO098.

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3:50 PM INVITED PAPER

THE THEORY OF GENERALISED CAHN-HOFFMAN XI-VECTORS: *A. A. Wheeler*¹; ¹University 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 inter-phase 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 ξ 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 ξ vector which may be associated with anisotropic diffuse interface models. We will demonstrate that the generalised ξ vector provides a natural theoretical setting for the diffuse interface models and provides a connection to the sharp interface ξ vector theory. We build on this idea to show that the generalised ξ 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 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. Boyce¹; J. P. P.¹; O. Roder¹; A. W. Thompson¹; R. O. Ritchie¹; ¹University 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 ($\nu \sim 1-2$ 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 ($a < 500 \mu\text{m}$) and long-crack ($a > 5 \text{ mm}$) techniques are employed on a Ti-6Al-4V blade alloy ($\sim 50\%$ primary α + transformed β) to estimate lower-bound values of ΔK_{TH} for $\nu = 50-1000$ 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.

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MICROCRACK PROPAGATION IN Ti-6Al-4V: *J. A. Hines*¹; J. O. Peters¹; G. Lutjering¹; ¹Technische Universität 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 α phase in a lamellar structure and an alloy containing 60% primary α . Microcracks (approximately 20-40 μm) 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.

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RELATIONS BETWEEN MICROSTRUCTURE AND FATIGUE PROPERTIES IN TITANIUM ALLOYS: *Anthony W. Thompson*¹; ¹Lawrence Berkeley 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 α phase, and the size of both individual α plates, and of α 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. Albrecht¹; G. Lutjering¹; K. -D. Folkers¹; Ch. Liesner¹; ¹Technische Universität 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, particularly in Ti-6Al-4V, in comparison 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 treat-

ment 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.

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ROLE OF MICROSTRUCTURE AND TEXTURE IN THE MEAN STRESS DEPENDENCE OF Ti-6Al-4V: *Svetlana G. Ivanova*¹; Frederic Cohen²; Richard D. Sisson¹; Ronald R. Biederman¹; ¹Worcester Polytechnic Institute, 100 Institute Road, Worcester, MA 01609-2280 USA; ²Pratt & 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.

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MEAN STRESS EFFECTS ON FATIGUE STRENGTH IN TITANIUM ALLOYS: *J. Lindermann*¹; L. Wagner¹; ¹Technical 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 most 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.

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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. Jha¹; ¹The 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.

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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. Mercer¹; W. O. Soboyejo¹; ¹The 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.

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FATIGUE CRACK GROWTH AT HIGH MEAN STRESS IN Ti-6Al4V AT 1.5 KHZ: *David L. Davidson*¹; ¹Southwest 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 Sizak, Inco Alloys International, Mechanical Testing, Huntington, WV 25705

Monday PM

Room: Rome

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

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MATERIALS-RELATED ADVANCES IN RE-BRAZEABLE SNM PACKAGES: *John J. Stephens*¹; James D. Pierce¹; Jeffrey G. Bobbe¹; Charles A. Walker¹; F. Michael Hosking¹; Charles V. Robino¹; ¹Sandia 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.

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THE BEHAVIOR OF A319 DURING HEAT TREATMENT: *Matt L. Newman*¹; Jonathan A. Dantzig¹; Huseyin Sehitoglu¹; ¹University 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.

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DENSITY FIELD VISUALIZATION IN CONVECTING METALLIC ALLOYS: *Jean N. Koster*¹; ¹University 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 radiosopic 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/cm³ 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 concentration 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.

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UTILIZING VIBRATION TO PROMOTE UNIFORM COMPOSITE GROWTH IN HYPERMONOTECTIC ALLOYS: *Richard N. Grugel*¹; Shinwoo Kim²; Shinwoo Kim²; ¹Universities Space Research Association, c/o MSFC, MS ES-75, Huntsville, AL 35812 USA; ²Hoseo University, Dept. of Materials Science and Engineering, Chung-Nam, Korea Korea; ²Hoseo 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.

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GLASS FORMATION AND CHARACTERIZATION OF AlNiFeRE ALLOYS BY MECHANICAL ALLOYING: *Weigang Meng*¹; Davin O'Neill¹; Gary Shiflet¹; ¹University 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.

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PULSED LASER WELDING OF TRICLAD METAL: A COMPARISON OF EXPERIMENTAL AND COMPUTATIONAL RESULTS: *Marcelino Essien*¹; Joseph A Romero¹; David Noble¹; Adonis C. Kassinos¹; Gerald A. Knorovsky¹; ¹Sandia 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 occurs 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.

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MACRO-PARTICLE DISTRIBUTION AND DEPOSITION RATE VARIATION OF TiAlN COATING BY MAGNETIC FILTERED CATHODIC ARC DEPOSITION: *Y. W. Lee¹; M. S. Leu¹; I. C. Hou¹; W. C. Lih¹;* ¹Industrial Technology Research Institute, Materials Research Laboratories, Bldg. 52, 195 Chung Hsing Road Section 4, Chunging, Hsinchu, Taiwan 310 ROC

The TiAlN 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.

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STRIP-EDGE PHENOMENA IN THE GAS WIPING OF A CONTINUOUS HOT-DIP GALVANIZING LINE: *Jeong-Real Park¹; Sun-Ho Jeon¹; Ro-Bum Park¹;* ¹POSCO, POSCO Technical Research Labs., Rolling Products Research Team, 699 Kumho-dong, Kwangyang, Chonnam 545-090 Republic of Korea

Turbulence 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 accommodate 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

Monday PM

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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 Mabuchi¹; Kenji Higashi²;* ¹National Industrial Research Institute of Nagoya, Hirate-cho, Nagoya, Kita-ku 462 Japan; ²Osaka 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.

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SUPERPLASTICITY OF 1N90 PURE ALUMINUM AND THE THEMOMECHANICAL PROCESSING: *Tsunemichi Imai¹; Sumito Kojima²; Daming Jiang³; Jianfu Mao³; Gilles L'Esperance⁴; Bande Hong⁴;* ¹National Industrial Research Institute of Nagoya, 1 Hirate-cho, Kita-ku, Nagoya 462 Japan; ²Nagoya Municipal Industrial Research Institute, 3-4-41 Rokuban-cho, Atsuta-ku, Nagoya Japan; ³Harbin Institute of Technology, Harbin 150001 P.R. China; ⁴Ecole 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.

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EQUAL CHANNEL ANGULAR EXTRUSION-A TURN IN THE RIGHT DIRECTION: *R. E. Goforth*¹; K. Theodore Hartwig¹; Roy Cornwell¹; ¹Texas 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.

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PROCESSING OF ALUMINUM ALLOYS FOR HIGH STRAIN RATE SUPERPLASTICITY: Patrick B. Berbon¹; Minoru Furukawa²; Zenji Horita³; Minoru Nemoto³; Nikolai K. Tsenev⁴; Ruslan Z. Valiev⁵; *Terence G. Langdon*¹; ¹University of Southern California, Department of Materials Science and Mechanical Engineering, Los Angeles, CA 90089-1453 USA; ²Fukora University of Education, Department of Technology, Munakata, Fukuoka 811-41 Japan; ³Kyushu University, Department of Materials Science and Engineering, Faculty of Engineering, Fukuoka 812-81 Japan; ⁴Ufa State Petroleum Technical University, Institute of Chemical Technology, Ufa 450062 Russia; ⁵Ufa 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).

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SUPERPLASTICITY OF ULTRAFINE-GRAINED Al-Mg-Sc ALLOYS PRODUCED BY EQUAL-CHANNEL ANGULAR PRESSING: Shogo Komura¹; Patrick B. Berbon²; Atsushi Utsunomiya¹; *Minoru Furukawa*³; Zenji Horita¹; Minoru Nemoto¹; Terence G. Langdon²; ¹Kyushu University, Fukuoka 812-81 Japan; ²University of Southern California, Los Angeles, CA 90089-0241 USA; ³Fukuoka University of Education, Munakata 811-41 Japan

An Al-3%Mg-0.2%Sc alloy with a grain size of ~0.2 μm 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 3.3×10^{-2} s⁻¹. These results demonstrate the potential for using ECA pressing to produce superplastic structure.

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CREEP BEHAVIOR OF POWDER METALLURGY Al-6092 AND Al-6061 ALLOYS: *Yong Li*¹; Terence G. Langdon¹; ¹University 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.

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A HEAT FLOW MODEL FOR FRICTION STIR WELDING: *Zhili Feng*¹; Jerry E. Gould²; *Thomas J. Lienert*²; ¹Edison Welding Institute, Engineering and Materials Team, 1250 Arthur E. Adams Drive, Columbus, OH 43221-3585 USA; ²Edison 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.

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DEFORMATION MICROSTRUCTURES IN FRICTION STIR WELDS ON 6061-T6: *Thomas J. Lienert*¹; *Richard J. Grylls*²; Jerry E. Gould¹; *Hamish L. Fraser*²; ¹Edison Welding Institute, Resistance and Solid-State Welding Team, 1250 Arthur E. Adams Drive, Columbus, OH 43221-3585 USA; ²The 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 testing. 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

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EFFECTS OF DEVIATIONS FROM STOICHIOMETRY ON THE MECHANICAL PROPERTIES OF SINGLE-CRYSTAL Ni₃Al:

*Toshiyuki Hirano*¹; Dmitri V. Golberg¹; Masahiko Demura¹; ¹National Research Institute for Metals, Mechanical Properties Division, 1-2-1, Sengen, Tsukuba, Ibaraki 305 Japan

Ni₃Al 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, Ni₃Al. 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 Ni₃Al 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.

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SOLUTE EFFECTS ON THE CREEP OF NiAl: *W. D. Nix*¹; P. H. Kitabjian¹; R. D. Noebe²; A. Garg²; ¹Stanford University, Department of Materials Science and Engineering, Stanford, CA 94305-2205 USA; ²NASA, 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 850°C to 1250°C. 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.

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EFFECTS OF PURITY ON THE PROPERTIES OF BETA-NiAl SINGLE CRYSTALS: *M. J. Kaufman*¹; J. Hu¹; V. I. Levit²; ¹University of Florida, Materials Science and Engineering, Gainesville, FL 32611 USA; ²AS&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 that, 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.

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STATIC AND DYNAMIC STRAIN AGING IN MICROALLOYED NiAl-BASED ALLOYS: *M. L. Weaver*¹; R. D. Noebe²; ¹University of Alabama, Department of Metallurgical and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; ²NASA, 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 its 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.

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SOLUTE HARDENING AND SOFTENING EFFECTS IN B2 NICKEL ALUMINIDES: *L. M. Pike*¹; C. T. Liu¹; I. M. Anderson¹; Y. A. Chang²; ¹Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA; ²University 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.

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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. Crimp¹; ¹Michigan 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).

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MECHANISMS OF DISCONTINUOUS YIELDING IN A Nb-Ti-Al ALLOY: *Richard J. Grylls*¹; R. Wheeler²; S Banerjee³; S Perungulam¹; D.-H. Hou¹; H.L. Fraser¹; ¹The Ohio State University, Department of Materials Science and Engineering, 2041 College Road, Columbus, OH 43210 USA; ²UES Inc., 4401 Dayton-Xenia Road, Dayton, OH 45432 USA; ³Bhabha 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 environ-

ments. 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 MATERIALS: *T. W. Barbee*¹; M. A. Wall¹; T. P. Weihs²; ¹Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; ²Johns 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 magnetron 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 Cu_xZr_{1-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 obser-

variations of fracture surfaces on "bulk" samples of the materials studied by this in-situ technique.

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SLIP PROPAGATION IN METALLIC MULTILAYERED THIN FILMS: *Peter M. Anderson*¹; *Tim Foecke*²; ¹The Ohio State University, Department of Materials Science and Engineering, 2041 College Street, Columbus, OH 43210 USA; ²National 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-reenucleation 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.

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DISLOCATION STRUCTURES IN NANO-LAMINATE MULTILAYER MATERIALS: *T. W. Barbee*¹; *M. A. Wall*¹; ¹Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

Transmission electron microscopy studies of a broad range of nano-laminate multilayer 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 structure 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.

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HIGH TEMPERATURE STABILIZATION OF LAYERED GRAIN STRUCTURES IN METALS BY SELECTIVE ZENER PINNING: *Gerhard F. Welsch*¹; *Paul Szozdowski*¹; *Ricard Collins*¹; ¹Case 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 welded metal structures, fibers and wires to enhance their creep and rupture strength properties. Acknowledgment: NSF-DMR 9315755.

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MECHANICAL BEHAVIOR OF COLD ROLLED NANOSCALE BRASS-STEEL MULTILAYER COMPOSITES: SIGNIFICANCE OF TEXTURE AND LAYER THICKNESS ON STRENGTH: *F. H.*

*Kavaranan*¹; *S. S. Sahay*²; *K. S. Ravichandran*¹; ¹University of Utah, Department of Metallurgical Engineering, 412 WBB, Salt Lake City, UT 84112-2 USA; ²Tata 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.

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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;

¹W. L. Gore and Associates, Inc, Medical Products Division, 3450 W. Kiltie Lane, Flagstaff, AZ 86002-0500 USA; ²University 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 T_m). 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.

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

Bricklayer composites made by solder-bonding 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

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

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

2:00 PM KEYNOTE

Al/Al₂O₃ FIBER COMPOSITES THROUGH INTERFACE PARTICLE COARSENING: EXPERIMENTS AND MODELING: *Ali S. Argon*¹; M. L. Seleznev¹; C. F. Shih²; X. H. Liu²; ¹M.I.T., Cambridge, MA 02139 USA; ²Brown 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; ¹3M 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. Foster¹; ¹Virginia 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)

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FRACTURE OF METAL/CERAMIC LAMINATES: TRANSITION FROM SINGLE TO MULTIPLE FRACTURE: K. L. Hwu¹; B. Derby¹;

¹University 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/sc decreases, 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.

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INTERFACE EFFECTS ON LOAD SHARING BEHAVIOR IN TITANIUM MATRIX COMPOSITES UNDER LONGITUDINAL TENSION: *B. S. Majumdar*¹; T. Matikas²; D. B. Miracle³; ¹UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; ²University of Dayton Research Institute (UDRI), Dayton, OH 45469 USA; ³Air 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.

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MODELING OF MICROCRACK TOUGHENING IN TWO-PLASE MULTILAYERED MEDIA: *Peter M. Anderson*¹; Sandeep Muju²; ¹Ohio State University, Dept. MSE, 2041 College Road, Columbus, OH 43210 USA; ²Allied 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.

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MICROMECHANICS OF THE INTERFACIAL DEBONDING IN TITANIUM MATRIX COMPOSITES HAVING GRADIENT INTERPHASE: A. Sinharoy¹; R. B. Bhagat¹; ¹The 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.

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EFFECT OF VOLUME FRACTION ON THE FRACTURE BEHAVIOR OF Nb-1%Zr/218W COMPOSITES AT ELEVATED TEMPERATURES: S. V. Raj¹; Louis J. Ghosn¹; ¹NASA 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 1.5×10^{-5} to 1.5×10^{-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.

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CORRELATION OF TENSILE STRENGTH WITH FRACTURE MODES OF KAOWOOL AND SAFFIL REINFORCED 339 ALUMINUM: William J. Baxter¹; Anil K. Sachdev¹; ¹GM 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 300°C and by fiber cleavage at 20°C. 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

Monday PM Room: Dublin
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

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WEAR RESISTANCE OF REFRACTORY METAL SILICIDE COMPOSITES: Joseph W. Newkirk¹; ¹University 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.

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METHODS OF IMPROVING TOUGHNESS IN Cr-Cr₃Si COMPOSITES: Joseph W. Newkirk¹; ¹University 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 Cr₃Si 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.

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DECOMPOSITION OF THE BETA PHASE IN A Nb-25Ti-25Al ALLOY: Keith J. Leonard¹; Vijay K. Vasudevan¹; ¹University 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 700°C were performed; a comparison of the results to those of calculated models are discussed. The formation and stability of orthorhombic Ti₂AlNb, 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.

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SOLIDIFICATION PATHWAYS AND SOLID STATE TRANSFORMATIONS IN Nb-Ti-AL INTERMETALLIC ALLOYS: *Keith John Leonard*¹; Joe C. Mishurda¹; Vijay K. Vasudevan¹; ¹University 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 .

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A STUDY OF PHASE EQUILIBRIA AND TRANSFORMATIONS IN THE Nb-Ti-AL SYSTEM: *Joseph C. Mishurda*¹; Vijay K. Vasudevan¹; ¹University 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. Ter-

nary 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 plethral 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 occur 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

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THE APPLICATION OF PLASMA-ASSISTED SURFACE TREATMENTS - A COMMERCIAL HEAT TREATER'S EXPERIENCE: *Peter C. Lidster*¹; Vladimir I. Gorokhovskiy²; Deepak G. Bhat³; ¹Exactatherm Limited, Mississauga, Ontario L5S 1 C1 Canada; ²Arcomac Plasma Processing Lab, Toronto, Ontario M6K 1W8 Canada; ³UES, 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.

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PLASMA NITRIDING AND DLC COATING AS A DUPLEX TREATMENT FOR HIGH LOADED STAINLESS STEELS: *J. Olfe¹; T. Michler¹; K. Bewilogua¹; K. -T. Rie²;* ¹Fraunhofer Institut für Schicht- und Oberflächentechnik, Braunschweig Germany; ²Institut für 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.

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PLASMA-ASSISTED COATINGS OF METALS: *O. T. Inal¹;* Tezer Brattal¹; John Hardy¹; ¹New 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 200Y. 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.

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EFFECT OF POWER ON THE SPUTTER DEPOSITED Cr COATINGS: *Z. Fan¹;* J. Seok¹; M. Pakala¹; R. Y. Lin¹; ¹University 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.

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SURFACE TREATMENT ON NON-FERROUS METALS BY PLASMA-ASSISTED CVD AND PLASMA NITRIDING: *Myoung-Jae Lee¹;* Duk Jae Kim¹; Jung Mi Hong¹; Yoon-Kee Kim¹; Hun Kyu Kim²; Keun-Ho Lee¹; ¹Plasma Technology Center, Institute of Advanced Engineering, Yonin, Kyungki-Do 449860 Korea; ²Daewoo 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 AlN layers.

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CORROSION RESISTANCE OF ION NITRIDED POWDER METAL PARTS: *Pramod Kotwal¹;* Paul Nowil¹; ¹Nitron 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 to 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.

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PLASMA NITRIDING UNIFORMITY IMPROVEMENT BY FINITE ELEMENT ANALYSIS MODELING: *Leon Duo¹;* Tony Wu¹; ¹Diesel 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.

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NONDESTRUCTIVE EVALUATION ON PLASMA NITRIDATION IN Fe-Cr SYSTEM: *Tatsuhiko Aizawa¹;* Hideyuki Kuwahara²; ¹University of Tokyo, Department of Metallurgy, 7-3-1 Hongo, Bunkyo, Tokyo 113-8656 Japan; ²Institute of Applied Science, Tanakaookubocho 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 struc-

ture, 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 nitrated structure is compared with the predicted system by the present model.

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SOURCE OF MULTICOMPONENT ATOMIC FLUXES: *I. N. Churkin*¹; *V. I. Volossov*¹; *A. G. Steshov*¹; ¹Budker 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

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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; ¹University 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; ¹University of Siegen, Inst. of Materials Technology Laboratory of Surface Engineering, Siegen D-57068 Germany; ²Zoz 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; ¹Zoz GmbH, Wenden D-57482 Germany; ²Fukuda Metal Foil & 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 1 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 deformed 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-SnO₂ ELECTRICAL CONTACT MATERIAL PRODUCED BY MECHANICAL ALLOYING: *H. Zoz*¹; *D. Ernst*¹; *N. Spath*²; ¹Zoz GmbH, Wenden Germany; ²Engelhard-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 conduc-

tivity, 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-SnO₂ was produced on the powder metallurgical route by reactive milling. The starting powders - the silver tin alloy Ag₃Sn and silver oxide (Ag₂O) - 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 SnO₂-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 properties 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; ¹Gazi 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 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 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; ¹Gazi University, Metallurgy Education Department, Tenik Okullar, Ankara Turkey

Reactive mechanical alloying of TiH₂-B (sample #1), Ti-TiH₂-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 1200°C 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 N₂ cause to the formation of TiN in different levels in all the samples studied. Annealing of the mechanically alloyed powders under purified Ar to 1200°C 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 TiH₂-B, Ti-TiH₂-B and Ti-B is a promising way for producing ultrafine grained Ti-TiN-TiB composite material with reactive mechanical alloy processing under N₂.

4:40 PM

TiN FORMATION IN THE TiH₂-B SYSTEM DURING REACTIVE MECHANICAL ALLOYING TO PRODUCE Ti-TiN-Ti₂-TiB METAL-MATRIX COMPOSITES: *Sedat Ozbilen*¹; ¹Gazi 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 1200°C 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 N₂; 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 1200°C for one hour leads to the formation of Ti-TiN-TiO₂-TiB with RMA under air; and only Ti-TiB with mechanical alloying under purified Ar.

5:00 PM

MECHANICALLY ALLOYED AND REACTIVELY SINTERED TiH₂-Al COMPACTS TO PRODUCE IMC (TiAl-Ti) MATERIALS: *Sedat Ozbilen*¹; ¹Gazi University, Metallurgy Education Department, Tenik Okullar, Ankara Turkey

TiH₂-Al powder mixtures with different TiH₂ 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; ¹McGill 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 crystal 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, and 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 Battaile¹; D. J. Srolovitz¹; P. Smereka²; ¹University of Michigan, Dept. of Materials Science and Engineering, 3074 H.H. DOW, 2300 Hayward Av, Ann Arbor, MI 48105 USA; ²University 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. Szpunar¹; ¹McGill 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 foils with the $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ textures were manufacturing an electrodeposition technique. The initial textures and 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 $\langle 100 \rangle$ texture followed by annealing leads to an Ni layer with a $\langle 111 \rangle$ texture and a low density of structural defects. Such a substrate of Ni promotes the formation of epitaxially oriented, slow growing and highly protective NiO scales with the $\langle 111 \rangle$ texture.

3:30 PM

AFM STUDY OF SURFACE TOPOGRAPHY AND MORPHOLOGY OF A NITROGEN PLASMA TREATED STEEL: *F. Mahboubi*¹; G. Collins²; ¹Amir Kabir University of Tech., P.O. Box 15875, -4413, Tehran Iran; ²Australian 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 550°C in a 75%N₂-25%H₂ atmosphere. The morphology and texture of the surface of the samples treated by P13 were totally 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 Zhu¹; Nobuya Iwamoto¹; ¹Ion 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; ¹Metz University, LETAM URA CNRS, 2090, Metz, Cedex BP 80794 - F-57012 France; ²Union Miniere, Research, B-, 2250 Olen; ³Cockerill Sambree, B, -4400, Flemalle France

Many commercial galvanized steel sheets have been characterized 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; ¹McGill 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-nickel 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; ¹Rutgers

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

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

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. Guo¹; ¹University 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-Al₃Ni-graphite composites and b) aluminum-alumina composite melts to synthesize Al-Al₂O₃-Al₃Ni-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. Rack²; ¹Air Force Research Laboratory, Wright-Patterson Air Force Base, OH 45433 USA; ²Clemson 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; ¹Institute of Advanced Manufacturing Sciences, Machining Excellence 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; ¹Edison 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; ¹Case 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.

Technical Program

TUESDAY AM

DISCONTINUOUSLY REINFORCED ALUMI-NUM-COMPOSITES: PRESENT & FUTURE: Secondary Processing and Performance of Discontinuously Reinforced Aluminum

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

Tuesday AM Room: Dublin
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433

8:30 AM INVITED PAPER

DESIGN OF MICROSTRUCTURES FOR OPTIMUM FRACTURE TOUGHNESS: *D. L. Davidson*¹; ¹Southwest Research Institute, San Antonio, TX USA

The fracture toughness of particulate reinforced composites are below those desired for useful engineering materials. Models that account for the effects of microstructure have been developed and used to optimize fracture toughness. Constraint of deformation by particles is identified as an important influence of microstructure on fracture, and is incorporated in the models. The purpose of the models is to focus processing efforts on those issues that can enhance fracture toughness. Finally, the issue of using shearable particles to enhance fracture toughness is addressed.

9:00 AM

THERMAL FATIGUE OF ALUMINUM-BASED MMCS STUDIED BY MECHANICAL SPECTROSCOPY: *E. Carreno-Morelli*¹; R. Schaller¹; ¹Ecole Polytechnique Federale de Lausanne, Institut de Genie Atomique, PHB-Ecublens, Lausanne CH-1015 Switzerland

The mechanical behaviour of aluminium based composites reinforced with short Al₂O₃ SAFFIL fibres has been investigated by mechanical loss and elastic shear modulus measurements during thermal cycling. A mechanical loss maximum which is not present in the unreinforced alloys has been observed during cooling near 120K. It originates in the relaxation of thermal stresses at the interfaces due to the differential thermal expansion between matrix and reinforcement. The maximum height increases with the volumetric fibre content. In addition, if the matrix strength is increased by the appropriated choice of alloy and thermal treatment, the maximum diminishes and shifts to lower temperatures. No damage accumulation at the interfaces has been detected during long period thermal cycling in the range 100-500K. A description of the damping behaviour is made in terms of the development of microplastic zones which surround the fibres. Such zones, in which dissipation takes place, would be beneficial for increasing the life time of MMC components in service conditions.

9:30 AM

MATRIX EFFECTS ON DEFORMATION AND FRACTURE OF DISCONTINUOUSLY REINFORCED ALUMINUM (DRA) COMPOSITES: *I. Dutta*¹; T. R. McNelley¹; ¹Naval Postgraduate School, Monterey, CA USA

This talk will present a review of the impact of matrix microstructure and flow behavior on deformation and fracture of DRA. Results of experimental studies delineating how the matrix grain structure and grain boundary character, dislocation sub-structure and precipitate state can influence both ambient and high temperature flow properties will be discussed. Additionally, the roles of these parameters on fracture toughness will also be addressed. In particular, it has been found that a number of unconventional heat treatments resulting in unusual precipitate states may be advantageously utilized to produce significant changes in both flow and fracture properties of DRA. The microstructural changes associated with these heat treatments will be discussed, and their impact on the mechanical properties of DRA will be presented.

10:30 AM

EXTRUSION MODELING OF DISCONTINUOUSLY REINFORCED ALUMINUM COMPOSITES: *Marvin McKimpson*¹; T. Loftin²; ¹Michigan Technology University, Institute of Materials Processing, Houghton, MI 49931 USA; ²DWA Aluminum Composites, 21130 Superior Street, Chatsworth, CA 91311 USA

A major factor limiting the introduction of wrought discontinuously reinforced aluminum composites into new applications is the high cost of developing and demonstrating the secondary processing required for these applications. More extensive use of numerical metal forming simulation tools has excellent potential for reducing this cost. Accordingly, a program has been initiated to utilize DEFORM, a commercial finite element simulation code, to model hot extrusion of 180 mm dia. 6092/SiC/17.5p rounds into simple shapes. The primary objective of this work is to develop an improved understanding of the factors controlling process parameter selection for extrusion of these commercial composites. The program involves developing appropriate high temperature flow data on the 6092/SiC/17.5p material, constructing an appropriate geometric and thermal model for the extrusion press being used, and then combining the two to simulate the extrusion operation. The status of this program, as well as recent research results, will be reviewed.

11:00 AM

MILLING AND DRILLING OF GrA-Ni : A Graphitic-SiC MMC: V. Songmene¹; M. Balazinski²; R. Maranzana¹; ¹Industrial Research and Development Institute, P.O. Box 518 L4R 4L3 Canada; ²Ecole Polytechnique de Montreal, C.P. 6079, Centreville, Montreal QC H3C 3A7 Canada

A new MMC consisting of an aluminum matrix reinforced with SiC and nickel-coated graphite particles, GrA-Ni, was developed two years ago. This composite has a low weight/volume ratio, and higher wear resistance, properties that make it suitable for brake rotors and liners as replacement for grey cast iron. The addition of nickel-coated graphite particles to SiC particulate in the aluminum matrix has improved the machinability of the SiC-reinforced MMC. But the tool performance is still poor compared to that obtainable on high silicon content aluminum alloys. The expansion of the use of MMCs in engineering applications requires more machining data on strategies to cost effectively cut these composites. GrA-Ni is relatively new and there is a very limited quantity of published data on its machinability. The paper investigates machining strategies to cost effectively rough GrA-Ni with carbide tools. A design of experiment type of approach was first used to compare the machining behavior of

GrA-Ni and existing aluminum SiC-reinforced MMCs at different cutting conditions. Several machining parameters (speed, feed, depths of cut) were used to machine the GrA-Ni composite and their impact on tool wear, tool life, volume of metal removed and unit machining cost were analyzed.

11:30 AM

COMMERCIAL DEVELOPMENT OF DRAS FOR PERFORMANCE AND LOW COST: *J. C. Withers¹; S. M. Pickard¹; ¹MER Corporation, Tucson, AZ USA*

Commercial applications of DRA for thermal management, structural applications and sporting goods are reviewed with case studies of MERs activities in these areas. Microstructure and property interrelationships for specific material applications with best selection of reinforcement phase and matrix alloy by performance and cost criteria are discussed.

DYNAMIC BEHAVIOR OF MATERIALS: Dynamic Synthesis, Processing, And Experimentation

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

Tuesday AM Room: Sydney
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Marc A. Meyers, University of CA, Dept. of AMES, La Jolla, CA 92093-0411 USA; Naresh N. Thadhani, Georgia Institute of Technology, Materials Science and Engineering, Atlanta, GA 30332-0245 USA

8:30 AM

REACTION SYNTHESIS OF SHOCK-DENSIFIED TITANIUM-BASED INTERMETALLIC AND CERAMIC COMPOUNDS: *Shantanu Ashok Namjoshi¹; Naresh N. Thadhani¹; ¹Georgia Institute of Technology, Materials Science and Engineering, 778 Atlantic Drive NW, Atlanta, GA 30332-0245 USA*

Shock densification was used to obtain dense unreacted compacts of Ti-Si (Ti₅Si₃), Ti-B (TiB₂), and Ti-Al (TiAl) powders mixed in stoichiometric ratios. The compacts (~ 85-90% dense) were characterized to determine the defect densities and crystallite size reduction due to the large deformations and fracture introduced during dynamic densification process. Systematic heat treatments were carried out on these systems to determine the reaction synthesis mechanisms and their enhanced kinetics. The highly-activated and dense-packed state of these materials resulted in complete chemical reactions at much lower temperatures than that at which reactions normally initiate with the melting of one of the components. The synthesis process was dominated by defect-enhanced solid-state diffusion reactions followed by a small amount of combustion reaction. Modeling of the heat and mass transfer processes occurring during reaction synthesis of these materials, was performed to determine the balance between the rate of heat generation and dissipation, thereby obtaining the limiting conditions for the prevention of the onset of the combustion reaction. Consequently the reaction synthesized compacts had higher

hardness, and were dense and free from defects, typically associated with combustion synthesis. In this presentation, the experimental and modeling results and characteristics of the synthesized Ti-Al, Ti-Si, and Ti-B based intermetallics, ceramics, and their composites will be presented. Funded by ARO Grant Number: DAA G55-97-1-0163

8:50 AM

LASER-DRIVEN SHOCK LOADING DEVICE FOR TIME RESOLVED MEASUREMENT OF SHOCK INDUCED REACTION: *Tatsuhiko Aizawa¹; ¹University of Tokyo, Dept. of Metallurgy, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113 Japan*

Through the systematic studies in the recent years, it has been found that the shock induced reaction should have its intrinsic feature as compared with the shock assisted reaction, where the thermal transients during shock loading governs the whole process in reaction. Furthermore, use of mechanically alloyed precursor (MA precursor) enables us to reduce the shock pressure condition for ignition of reactions. Although the reaction process can be described through the recovery experiments for the shock induced reaction into aluminides and silicides from the starting constituent elements by varying the shock condition and the premixing parameters in preparation of MA precursor, no direct information can be obtained with respect to the shock reaction mechanism during shock loading. In the present paper, new shock loading procedure is proposed for time resolved measurement to describe the shock reaction process on the route of the laser-driven shock loading. Three points to be noticed here are: 1) Adaptive shock cell to laser-driven shock loading, 2) Investigation of shock induced reaction from the MA precursors, and 3) Time resolved measurement of shock Hugoniot by using the VISAR. Using the two types of shock cell for time resolved measurement and shock recovery experiments, we can describe the time history of particle velocity by using the VISAR and evaluate the synthesized materials by XRD and microstructure analysis. Mo - Si system is employed for a targeting material to investigate the shock ignition condition from MA precursor with different premixing level to molybdenum di-silicides.

9:10 AM

DYNAMIC POWDER COMPACTION BY THE ION-BEAM DRIVEN SHOCK LOADING: *Minoru Tasuchida¹; Tatsuhiko Aizawa²; Kozuhiko Horioka³; ¹University of Tokyo, Graduate School of Engineering, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113 Japan; ²University of Tokyo, Department of Metallurgy, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113 Japan; ³Tokyo Institute of Technology, Department of Energy Science, Nagatsuda 4259, Yokohama, Kanagawa 226 Japan*

New methodologies have been studied to improve the number of event actions for shock loading and to make repetitive shock compaction instead of the powder or gas guns and the explosive detonation methods. Use of powder laser becomes the first candidate to make time-resolved evaluation on the dynamic behavior during the shock loading. Since the yield of recovered sample is limited to very small amount, however, another candidate method is waited especially for shock recovery experiments. Authors have been developing the ion-driven shock loading device both for higher-velocity experiments and for shock cold and hot compactions. Different from the laser-driven shock loading devices, relatively large samples can be shock consolidated by repetitive application of high pressure pulses. The flyer can be accelerated to the targeting velocity by tuning the shock loading conditions: the carrier gas species for generation of plasmas, and the voltage of condenser bank for acceleration of plasmoids. In the present paper, our developed ion-beam driven shock loading system is briefly introduced with some comments on its capacity for acceleration and the real time measurement of flyer velocity. Two targeting material systems are employed for cold and hot compaction: 1) Rapidly quenched Al-based amorphous alloys powders, and 2) Ne-Fe-B magnetic amorphous alloys powders. Parametric study is first done to determine the adequate shock condition for successful recovery and to investigate the effect of shock pressure on the change of XRD profiles for the recovered samples. The

holding temperature is also varied to understand the effect of temperature on the compaction behavior during the shock loading.

9:30 AM

INVESTIGATION OF COMBINED REACTION PROCESSING AND DYNAMIC DENSIFICATION IN Mo + 2 Si POWDER MIXTURES: *Kevin S. Vandersall*¹; Naresh N. Thadhani¹; ¹Georgia Institute of Technology, Materials Science and Engineering, 778 Atlantic Drive, Atlanta, GA 30332-0245 USA

Statically pressed Mo-Si powder mixtures (~50-55% dense) were densified employing shock and magnetic implosion techniques. Reaction was observed in some of the recovered compacts and the resulting microstructure revealed differences associated with whether the reaction occurred due to shock pressure or temperature. These differences provide information on the mechanism of reaction, which can be characterized as shock induced (occurring during the high pressure shock state) or shock assisted (occurring subsequent to the shock event, but due to bulk temperature increases). The un-reacted compacts (~78-95%) were subsequently reacted by thermal treatments under controlled conditions. The influence of densification parameters including initial packing density, pressure utilized, and loading conditions (which in turn control the compact density, defect concentration, and minimization of macroscopic cracking) on the reaction behavior was investigated using x-ray diffraction (XRD) and optical and scanning electron microscopy (SEM). This paper will outline the methods applied and microstructures of MoSi₂ intermetallic formed by reaction processing under different conditions. Work supported by ARO/AASERT Grand No. DAAH04-95-1-0235.

9:50 AM

A CRITICAL ANALYSIS OF THE SHPB THROUGH SIMULATION AND EXPERIMENT: *David A. Gorham*¹; Xiaojun J. Wu¹; ¹The Open University, Faculty of Technology, Walton Hall, Milton Keynes, Buckinghamshire MK7 6AA UK

The paper will present results from a project in which we have examined the reliability of the compressive split Hopkinson pressure bar (SHPB) test by exploring material models in experimentally-validated simulations. The experimental work was with a 6.7 mm SHPB system, using copper specimens. For these measurements we have paid careful attention to dispersion, friction and calibration. The numerical work has been carried out with axisymmetric models in DYNA2D. Very good agreement with experiment can be obtained using a simulation model in which the input bar is truncated at the strain gauge position and is loaded at that point with an experimentally measured incident pulse. The simulation is fast enough for material parameters in an appropriate model to be adjusted by trial and error until the total force on the output face of the simulated specimen matches closely with the transmitted pulse from the experiment. This agreement with experiment has given us confidence that the detailed numerical history and mapping of stress and strain within the specimen are realistic. Such simulation alongside experimental work gives a great deal of information about the stress and strain distribution that would be impossible to obtain any other way. Using this technique we have examined the uniformity of stress, strain and strain rate over a range of specimen geometries and loading rates. This has shown that inhomogeneous deformation can be significant in areas of operation of the SHPB test that are accepted as valid. A number of examples will be presented. The close agreement between simulation and experiment means that the fitted material parameters can be regarded as the results of the test. The simulation model we have developed is fast enough, with current computer technology, to be used on a routine basis, and so a possible approach to improve the reliability of stress-strain results from the SHPB is to parallel each experiment by fitting such a simulation. An important point is that valid material characteristics can be obtained by such simulation-supported tests even when deformation is not uniform, and so provides the opportunity to obtain reliable results over a wider range of SHPB operating conditions than can be achieved at present. These possibilities will be discussed in the paper.

10:10 AM BREAK

10:20 AM

ATOMISTIC FINITE DEFORMATION HIGH STRAIN RATE SIMULATIONS OF SINGLE CRYSTALS: *Mark F. Horstemeyer*¹; Mike Baskes¹; ¹Sandia National Labs, MS9405, 7011 East Ave, Livermore, CA 94550 USA

Atomistic simulations employing the Embedded Atom Method are used to determine length scale effects related to macroscopic mechanical quantities under high strain rates. Various orientations of single crystal Nickel and Copper have been deformed numerically under compression and simple shear loading up to 30% strain. The motivation for this study is to develop mathematical expressions for bridging length scales within the context of internal state variable viscoplasticity. In order to bridge scales, we believe that a systematic method of determining hardening mechanisms from a bottom-up approach is key. As such, we examine the misorientation, microtexture, and heterogeneous hardening that occurs within the grain. These simulations indicate that determining the continuum effects related to spatial length scales is feasible up to the micron level based on the recent improvements of parallel computing.

10:40 AM

SIMULATION OF SHEAR PLUGGING THROUGH THIN PLATES USING THE GRIM EULERIAN HYDROCODE: Philip Church¹; Ian Cullis¹; Nick Lynch¹; ¹DERA, Warheads Division, Fort Halstead, Sevenoaks, Kent TN14 7BP United Kingdom

Ballistic experiments have been performed using aluminium spheres against 10mm RHA, MARs 270, MARs 300 and titanium alloy plates to investigate the influence of plugging mechanism on material properties. The experiments have measured the threshold for plug mass and velocity as well as the recovered aluminium sphere mass over a range of velocities. Some of the experiments have been simulated using the in-house second generation Eulerian hydrocode GRIM. The calculations feature advanced material algorithms derived from interrupted tensile testing techniques and a triaxial failure model derived from notched tensile tests over a range of strain rates and temperatures. The effect of mesh resolution on the results has been investigated and understood. The simulation results illustrate the importance of the constitutive model in the shear localisation process and the subsequent plugging phenomenon. The stress triaxiality is seen as the dominant feature in controlling the onset and subsequent propagation of the crack leading to the shear plug. The simulations have demonstrated that accurate numerics coupled with accurate constitutive and fracture algorithms can successfully reproduce the observed experimental features.

11:00 AM

THE USE OF TRANSIENT X-RAY DIFFRACTION FOR SHOCK WAVE AND HIGH PRESSURE MATERIALS SCIENCE: *Allan A Hauer*¹; G. A. Kyrala¹; R. A. Kopp¹; D. J. Thoma¹; K. Chen¹; F. Chu¹; D. C. Wilson¹; P. Gobby¹; L. Foreman¹; J. S. Wark²; A. Loveridge²; D. H. Kalantar³; B. Remington³; J. Colvin³; M. A. Meyers⁴; J. Asay⁵; C. Hall⁵; T. Tracano⁵; ¹Los Alamos National Laboratory, Group P-24, E 526, Los Alamos, NM 87545 USA; ²Oxford University, Dept. of Physics, Oxford, Oxfordshire UK; ³Lawrence Livermore Nat. Lab., L-473, Livermore, CA 94551 USA; ⁴Univ. of Calif. San Diego, Dept. of Ames, San Diego, CA 92093 USA; ⁵Sandia Nat. Lab., Pulsed Power Sciences Dept, Albuquerque, NM 87544 USA

Transient x-ray diffraction is a powerful tool for the study of shock wave propagation and materials effects in the condensed state. Some of the phenomena that can be studied are plastic wave propagation, generation of dislocations, and the dynamics of phase transitions. By observing the angular deflection of the diffracted beam due to the passage of a shock or stress wave a real time measurement of strain can be made (with 10 ps temporal resolution) we have recently performed a series of experiments on the Trident laser at Los Alamos and the Nova laser at Livermore that illustrate the value of transient diffraction when used in laser-based experiments. For example, by

observing the diffraction from 2 or more planes simultaneously information can be obtained on plastic wave propagation. For purely elastic solid response, uniaxial compression in the direction of shock propagation would be expected. If, however, significant plastic deformation occurs a diffracted signal from planes orthogonal to the shock propagation might be expected. We have observed such a signal in experiments with LiF crystals. Similar experiments with silicon have not yet shown this behavior but exhibit other interesting phenomena such as the splitting of diffracted lines which may give temporally resolved information on phase changes. In recent work we have applied transient diffraction to the study of Beryllium. Beryllium is one of the most promising materials for the fuel containing capsule in inertial confinement fusion (ICF) target designs that may achieve ignition. Beryllium has many unusual properties such as extremely high tensile strength, and high density for such a low x-ray opacity that make it valuable as an ICF capsule material. Fabrication into a spherical capsule, however, presents a number of challenges that must be overcome through materials properties research. In addition, the performance of a Be ignition capsule in the early stages of implosion may also depend on specific Be materials properties such as grain structure or the details of melting. As part of the capsule fabrication effort, high quality single crystal Be samples have been made. We have obtained time resolved diffraction measurements using these crystals that will help to clarify the behavior of Be in laser driven implosions. Recent results from these experiments will be presented.

11:20 AM

TRANSIENT X-RAY DIFFRACTION OF SHOCK COMPRESSED CRYSTALS USING THE NOVA LASER: *Daniel H. Kalantar*¹; E. A. Chandler¹; J. D. Colvin¹; K. Mikaelian¹; B. A. Remington¹; S. V. Weber¹; L. G. Wiley¹; A. A. Hauer²; J. S. Wark³; *M. A. Meyers*⁴; G. Ravichandran⁵; ¹Lawrence Livermore National Lab, L-473, 7000 East Ave, Livermore, CA 94550 USA; ²Los Alamos National Lab., P.O. Box 1663, Los Alamos, NM 87545 USA; ³University of Oxford, Clarendon Laboratory, Parks Road, Oxford OX1 3PU UK; ⁴University of California, San Diego, Dept. of Applied Mechanics and Engineering Sciences, MC-0411, San Diego, CA 92093 USA; ⁵California Institute of Technology, Pasadena, CA 91125 USA

We are conducting experiments on the Nova laser to investigate the Rayleigh-Taylor instability in metal foils maintained in the solid state. In these experiments, we use a hohlraum x-ray drive to ablatively accelerate a thin Cu foil that has a preimposed modulation at the ablator-metal interface. We use x-ray backlighting to characterize the instability growth. By temporally shaping the drive history, the foil is kept below the melt temperature at peak pressures up to 3 Mbar under extreme strain rates (10^7 - 10^9). In support of this effort, we are developing dynamic diffraction as a diagnostic of material state for shock compressed samples. The crystal samples are shock compressed with an x-ray drive, and x-rays from a point backlighter are used to measure the lattice compression by diffraction. We have observed shock compression of more than 10% in 1-D in a Si crystal. In addition, the crystal is probed by simultaneous Bragg and Laue diffraction of orthogonal lattice planes [1] to study the transition from elastic to plastic compression in a shocked solid. We are also working to use dynamic Bragg diffraction as a way to verify that the metal foils used in the instability growth experiments remain solid at peak compression. We will present results from the instability growth measurements of Cu foils, and dynamic Bragg experiments using shocked Si. [1] R. R. Whitlock and J. S. Wark, Phys. Rev. B 52, 8 (1995).

11:40 AM

DEFORMATION LOCALIZATION OF 8090 Al-Li ALLOYS DURING DYNAMIC LOADING AT ROOM TEMPERATURE AND LIQUID NITROGEN: *Y. B. Xu*¹; W. L. Zhong²; Y. J. Chen³; Y. L. Bai²; M. A. Meyers³; ¹Chinese Academy of Sciences, Institute of Metal Research, Shengyang, P.R. China; ²Chinese Academy of Sciences, Institute of Mechanics Research, Beijing, P.R. China; ³Univer-

sity of California, San Diego, Materials Science Group, 9500 Gilman Drive, La Jolla, CA 92093-0411 USA

Shear localization at a strain rate of 10^3 s⁻¹, was studied in 8090 Al-Li alloys at 298 and 77 K in the (1)naturally-aged, (2)peak-aged, (3)under-aged, (4)over-aged conditions. The peak-aged Al-Li alloy had a higher tendency to form a shear the other three other conditions. In the peak-aged Al-Li alloy, double shear bands and kink bands were also observed. SEM revealed damage of the microstructure within the shear band. The sharp drop in the load is associated with growth and coalescence of microcracks and microvoids rather than the occurrence of shear localization, but shear localization is seen to accelerate their growth and coalescence. TEM of matrix and transition region showed an elongated structure with high dislocation densities. Structure becomes fragmented in the margin of shear band. In center, fine equiaxed grains with lower dislocation densities are observed, due to the effects of the large temperature rise and the highly localized deformation in an extremely short time. Evidence for dynamic recovery and dynamic recrystallization is found.

EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Surface Energy Effects in Microstructure Formation; 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; Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Dept.; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Tuesday AM
October 13, 1998

Room: International Ballroom
Location: O'Hare Hilton Hotel

Session Chair: Robert Balluffi, MIT, Cambridge, MA 02139-4307

8:30 AM INVITED PAPER

MORPHOLOGY: THERMODYNAMICS AND KINETICS: *W. Craig Carter*¹; ¹MIT, Dept. of Materials Science, 77 Massachusetts Ave, Cambridge, MA 02139-4307 USA

John Cahn's career has influenced a remarkable breadth of topics in materials science. The excitement of discovery and quality of instruction that he has provided for his colleagues cannot be overestimated. This talk will illustrate some work with Cahn which lead to the convergence of fundamental principles from his previous, and seemingly disparate, contributions to materials science: morphology, kinetics and thermodynamics. The principles behind the development of corners in a Wulff shape and the thermodynamics of a miscibility gap and the conditions for a steady-state growth shape all have the same underlying theoretical structure. The common tangent rule is related to the Wulff construction through convexification of a free energy surface. Because the choice of the metric for convexification has traditionally been different for these two topics, their connection has not been obvious. Likewise the principles be-

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hind the Gibbs-Duhem equation and the Cahn-Hoffman equations are the same; furthermore, they are equivalent to the method of characteristics which can be applied to growth shapes for particular physical models. The depth of understanding which is established by such connections will be discussed as well as applications to observed materials phenomena.

9:15 AM INVITED PAPER

TRIPLE JUNCTIONS: *Jean E. Taylor*¹; ¹Rutgers University, Mathematics, Hill Center, Busch Campus, 110 Frelinghuysen Road, Piscataway, NJ 08854 USA

It was John Cahn's interest in soap bubbles and soap films, in particular my proof that the only singularities were smooth triple junction curves and isolated tetrahedral points (as observed by Plateau a century earlier), that drew him to a lecture I gave 25 years ago and eventually led to our long collaboration. About that time he, together with Hoffman, was investigating which multiple junctions might be possible for anisotropic surface free energies. Both he and I have continued to be interested in multiple junctions and how they move. Much of our joint and separate work on this topic will be reviewed, together with observations concerning positive and negative triple junction line energies.

10:00 AM BREAK

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WETTING AND ADSORPTION TRANSITIONS IN Ga-Pb ALLOYS: *Paul Wynblatt*¹; Dominique Chatain²; ¹Carnegie Mellon University, Dept. of Mater. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; ²CMRM2-CNRS, Campus de Luminy, Case 913, F-13288 Marseille Cedex 9, Bouches du Rhone France

In 1977, Cahn predicted that a transition from incomplete to complete wetting should occur, at some temperature below the critical temperature, in binary systems displaying a liquid miscibility gap. Results will be described which establish the complete wetting of Ga-rich liquids by Pb-rich liquids, by means of Auger electron spectroscopy (AES) measurements at the surface of two-phase liquid mixtures. The measurements also demonstrate, as expected from Cahn's theory, that the thickness of the Pb layer on the Ga surface diverges as liquid-liquid coexistence is approached. Recent determinations of adsorption at single phase liquid Ga surfaces, performed by both surface energy and AES measurements, will be discussed in the light of so-called prewetting transitions also predicted by Cahn.

10:25 AM

BOUNDARY MOTION BY CURVATURE IN THE FERROMAGNETIC POTTS MODEL WITH TRIPLE JUNCTIONS: *Mark A. Miodownik*¹; Anthony D. Rollett²; Elizabeth A. Holm¹; ¹Sandia National Laboratories, Materials Theory & Computation, P.O. Box 5800, MS 1411, Albuquerque, NM 87185-1411 USA; ²Carnegie Mellon University, Matls Sci & Eng Dept, Pittsburgh, PA 15213-3890 USA

The ferromagnetic Potts model has been used extensively to simulate grain growth in polycrystalline materials. While excellent agreement is found in many cases, the limits of applicability of the model are not well-known. In this study, we examine the evolution of a simple polycrystalline system of known, constant driving force: columnar grains with a single crystal cap. This system contains only grain boundaries and triple junctions, and no topological changes occur during evolution. For isotropic boundary energy and uniform boundary mobility, this system evolves with dynamics equal to the continuum case, except at very small grain widths, where lattice and stochastic effects cause deviations. When boundary mobilities vary, the system finds a steady state in which the grains shrink uniformly; that is the product of mobility and curvature is constant for all the moving boundaries in the system. For anisotropic boundary energies, the system evolves correctly as well, but it becomes much more sensitive to anisotropy in the underlying lattice. Similar results are

found in three dimensions. The case of four-grain junctions is briefly discussed.

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BOUNDARY MOTION BY DISCRETIZED MEAN CURVATURE IN THE FERROMAGNETIC ISING MODEL: *Elizabeth A. Holm*¹; John W. Cahn²; *Mark A. Miodownik*¹; ¹Sandia National Laboratories, Materials Theory & Computation, P.O. Box 5800, MS 1411, Albuquerque, NM 87185-1411 USA; ²NIST, 223/A153, Gaithersburg, MD 20899 USA

The ferromagnetic Ising model is used to study coarsening of systems driven by surface energy minimization, such as magnetic domain coarsening. However, the relationship between the motion of a discretized boundary with energy determined by neighbor counting and the motion of a continuous boundary by curvature has not been elucidated. We show that for a ferroelectric Ising model with Metropolis dynamics at $T=0$, a boundary discretized on a two-dimensional lattice moves with velocity proportional to its discrete mean curvature, independent of the magnitude of the surface free energy per unit length. Simulations of isolated boundaries (i.e., shrinking circle and hairpin bicrystal) confirm this prediction. Similar results are found in three dimensions, although artificial pinning may occur for lattices of low coordination number. Boundary segments in ferromagnetic Potts models used to simulate grain growth also move by discretized mean curvature, in contrast to grain boundaries, which move by weighted mean curvature. The presence of domain junctions also influences boundary motion in such systems.

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THE EQUILIBRIUM SHAPE OF LIQUID Pb AT TRIPLE JUNCTIONS IN Al: *Heike Gabrisch*¹; ¹Lawrence Berkeley National Laboratory, National Center for Electron Microscopy, Building 72, 1, Cyclotron Road, Berkeley, CA 94720 USA

The effect of liquid metals on the properties of polycrystalline materials is well established. However, most investigations of the underlying wetting phenomena rely on indirect evidence. Here we present direct observations of the behavior of liquid Pb at grain boundaries in Al. Using in-situ transmission electron microscopy, we have determined the shape of liquid Pb inclusions in solid Al at the intersection of grain boundaries with the foil surfaces and at grain boundary triple junctions. It was found that dihedral angles along triple junctions are smaller than along grain boundaries, leading to a greater tendency for wetting. The observed shapes could be used to determine relative interfacial energies and may be understood in terms of a simple model. 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-ACO3-76SF00098.

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TRIPLE JUNCTIONS IN ANISOTROPIC MATERIALS: *Alexander H. King*¹; ¹State University of New York, Materials Science & Engineering, Nicolls Road, Stony Brook, NY 11794-2275 USA

The measurement of dihedral angles at triple junctions is a time-honored method of determining interfacial energies, but is only used under the assumption of isotropic interfacial energy. The Cahn-Hoffmann capillarity vector provides a mechanism for extending the analysis to anisotropic cases. We demonstrate some applications of this approach in this presentation, concentrating upon cases in which it is known that at least one boundary plane lies in an energy-minimizing orientation. It is shown that the dihedral angles are not uniquely defined for these cases, and that they can instead fall anywhere within specified "allowed ranges." This allows for a variability of dihedral angle that can be used to minimize interfacial curvature, and thus the driving force for migration. These effects are all demonstrated through TEM observations Acknowledgement: This work is supported by NSF Grant # DMR 9530314

10:45 AM

INTERATOMIC POTENTIAL AND ATOMIC STRUCTURE OF A MODEL CERAMIC/METAL INTERFACE: {222}MgO/Cu: *Roy Benedek*¹; *David N. Seidman*²; L. H. Yang³; L. H. Yang³; L. H. Yang³; A. Alavi⁴; ¹Argonne National Laboratory, Building 223, Argonne, IL 60439 USA; ²Northwestern University, Materials Science and Engineering Dept., 2225 North Campus Drive, Evanston, IL 60208-3108 USA; ³Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; ⁴University of Belfast

Ab initio local-density-functional-theory (LDFT) calculations (with the plane-wave-pseudopotential method) and classical molecular statics simulations are applied to {222}MgO/Cu, a model interface that has been the subject of extensive experimental observation. This interface is polar, has a misfit of about 1/6, and is invariably oxygen terminated. Relatively small-cell (less than approximately 30 atoms) LDFT total-energy calculations are performed for coherent and semicoherent {222}MgO/Cu interfaces, as a function of interface spacing and translation state, to elucidate the interface potential. The results suggest a simple short-range interface interatomic potential that includes an attractive contribution and a repulsive Born-Mayer contribution. Molecular statics calculations with this potential predict a misfit dislocation network with trigonal symmetry and no standoff dislocations. This potential, however, is able to address atomic relaxation only on the metallic (Cu) side of the interface. LDFT calculations for large supercells that include misfit (with up to 360 atoms) enable the validity of the dimple interface potential to be assessed. *Supported by the U.S. Department of Energy.

10:50 AM

MICROSTRUCTURAL STABILITY AND SEGREGATION IN Al-Sc-Mg ALLOYS: *Dmitry Gorelikov*¹; *David N. Seidman*¹; ¹Northwestern University, Materials Science and Engineering Dept./MLSB, 2225 North Campus Drive, Evanston, IL 60208-3108 USA

Dilute Al-Sc alloys have excellent mechanical properties at room temperature and a high yield-strength at temperatures up to 673 K, due to the presence of elastically hard coherent and unsharable Al₃Sc precipitates that can be obtained at a high number density [R.W. Hyland, Jr. Metall. Trans. A 23A, 1947 (1992)]. These Al₃Sc precipitates remain coherent to a diameter of 20-25 nm, since the lattice parameter mismatch is only about 1%. The addition of Mg prevents recrystallization of Al-Sc alloys up to the solidus temperature. Precipitation hardening of an Al-0.2 wt.% Sc-Mg alloy is studied by atom-probe field-ion and transmission electron microscopies. The effects of segregation of Mg to {100} Al₃Sc/Al interfaces on the coarsening of coherent and semicoherent Al₃Sc precipitates is studied. The Gibbsian interfacial excess of solute at the {100} Al₃Sc/Al interface is studied as a function of ageing temperature, time, and precipitate diameter. *This research is supported by the National Science Foundation/DMR.

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LOCAL STRESSES AT INTERFACES AND THEIR EFFECTS ON SEGREGATION: *Olof C. Hellman*¹; *David N. Seidman*¹; ¹Northwestern University, Materials Science and Engineering Dept./MLSB, 2225 North Campus Drive, Evanston, IL 60208-3108 USA

Solute segregation to interfaces is often driven by stress effects; a solute atom's placement at or near a grain boundary (GB) can relieve both the stress associated with a solute atom in the host crystal structure and the local stress intrinsic to the grain boundary. Frequently, the GB structure and its local state of stress is simple enough that a one-dimensional model of segregation behavior suffices for explaining the effect of stress on segregation. This has been fortunate, because in the past, experimental examination of segregation to GBs was only possible on a one-dimensional level. Many GBs, however, are expected to have very complex structures for which a one-dimensional treatment does not suffice. Now that experimental techniques, such as three-dimensional atom-probe microscopy, are providing richer experimental data, our theoretical treatment of local stress at interfaces needs to be expanded to examine stress distributions in three-dimensions near interfaces, and to resolve each compo-

nent of the local stress. We are investigating the interrelation of the effects of atomic structure, local strain and impurity segregation in a series of crystallographically distinct GBs. We use an embedded-atom method (EAM) type potential for modeling atomic interactions, and Monte Carlo and energy minimization techniques for calculating atomic positions and solute segregation. We attempt to draw general conclusions about segregation at interfaces by correlating segregation behavior with each of the components of the local stress tensor. *This research is supported by the National Science Foundation/DMR.

11:00 AM

ATOMIC-SCALE STUDIES OF EARLY-STAGE DECOMPOSITION MICROSTRUCTURES IN SYSTEMS WITH A LARGE TRANSFORMATIONAL MISFIT: *Dieter Isheim*¹; *Didier Blavette*²; *David N. Seidman*¹; ¹Northwestern University, Materials Science and Engineering Dept./MLSB, 2225 North Campus Drive, Evanston, IL 60208-3108 USA; ²Universite de Rouen, Laboratoire de Microscopie Ionique, URA CNRS 808, Faculte des Sciences et Techniques, F-76821, Mont-Saint-Aignan, Cedex France

Elastic interactions are known to cause an alignment of coherent precipitates with a large misfit with the matrix, in elastically anisotropic matrices. A periodic arrangement of precipitates can also be produced by a spinodal decomposition process. Simply observing the spatial arrangement of precipitates may not be sufficient to reveal the actual operating mechanism. In order to obtain additional insight, atomic scale analyses of the precipitate's compositions, as well as the chemical sharpness of their interfaces, during the early stages of the decomposition is necessary. This contribution presents the results of a study of the decomposition of an Fe-20 at.% Mo alloy, employing conventional and high resolution electron microscopies, as well as atom-probe field-ion microscopy including three-dimensional atomic tomography. *Research supported by the National Science Foundation/Division of Materials Research and the Deutsche Forschungsgemeinschaft.

11:05 AM

EXPLORING GRAIN-BOUNDARY PHASE SPACE ON AN ATOMIC SCALE: SIMULATIONS AND EXPERIMENTS: *David N. Seidman*¹; ¹Northwestern University, Materials Science and Engineering Dept./MLSB, 2225 North Campus Drive, Evanston, IL 60208-3108 USA

The concept of grain boundary (GB) phase space (with the 5 macroscopic geometric variables serving as state variables leading to a local phase rule for GBs) represents a basic thermodynamic framework for understanding GB segregation [J.W. Cahn, J. Phys. (Paris) 43, C6-199 (1982)]. We employ Monte Carlo techniques (Metropolis algorithm and overlapping distributions MC) to explore systematically the 8-dimensional GB phase space for both twist and tilt boundaries in dilute single-phase binary f.c.c. alloys; the atomic interactions are described by Baskes-Daw-Foiles embedded-atom method (EAM) potentials. We first use lattice statics calculations to determine the lowest energy GB structures and then MC simulation to calculate the solute-atom distribution and Gibbsian interfacial excess at elevated temperatures for each GB structure studied. Accurate segregation free energies for atomic sites in GBs are calculated via the ODMC technique and segregation entropies are determined and found to be a linear function of the segregation internal energies for the same GB sites. The effects of both the 5 macroscopic and 3 microscopic degrees of freedom (DOFs) are studied and it is demonstrated that the Gibbsian interfacial excess is a function of these DOFs. Also a GB's atomic structure determines the partitioning of segregating solute atoms between the cores of dislocations and in the elastic stress fields of GB dislocations. We demonstrate that none of the geometric criteria frequently suggested in the literature is capable of predicting the propensity for GB segregation, much less the magnitude. Experimental segregation results have been obtained on selected binary alloys employing combined transmission electron and atom-probe microscopies, which demonstrate the importance of the macroscopic DOFs in determining the value of the Gibbsian interfacial excess for a

specific GB. *This research is supported by the National Science Foundation/DMR.

11:10 AM

FIRST-PRINCIPLES STUDY OF ORDERING AND PRECIPITATION IN ALUMINUM ALLOYS: *Christopher M. Wolverton*¹; Alex Zunger¹; ¹NREL, 1617 Cole Blvd., Golden, CO 80401 USA

We demonstrate a first-principles theoretical approach for studying the ordering and precipitation in aluminum alloys. The method involves mapping atomically-relaxed first-principles total energies onto an effective Hamiltonian. The effective Hamiltonian is composed of coherency strain and chemical energy terms. By combining this Hamiltonian (which is comprised of both real- and reciprocal-space terms) with a mixed basis Monte Carlo algorithm, we are able to study the thermodynamic and structural properties of Al-rich alloys, both at high and low temperatures. The method has been applied to three Al-rich alloy systems which makes for an interesting comparison, due to the different relative contributions of strain and chemical energies: Al-Mg, Al-Cu, and Al-Ni. We find that anomalously soft coherency strain for certain preferred orientations leads to the observed precipitate shape [e.g., (001)-planar in Al-Cu and Al-Ni and more spherical in Al-Mg]. Furthermore, we demonstrate the importance of anharmonic contributions to the coherency strain for large size-mismatched alloys.

11:15 AM

COHERENCY STRAIN AND VEGARD'S LAW: *Jong K. Lee*¹; ¹Michigan Technological University, Dept. of Metallurgical and Materials Engineering, 1400 Townsend Drive, Houghton, MI 49931-1295 USA

Using the Discrete Atom Method (DAM) to examine the elastic strain energy associated with arbitrarily-shaped, elastically-inhomogeneous, coherent precipitates, both miscibility gap and lattice parameter are investigated as a function of solute composition in a binary model fcc system. The solubility limit and lattice parameter depend not only on the misfit strain but also on the solvent-solute bond length. For a given positive misfit strain, as the solvent-solute bond length approaches that of the matrix phase, the solubility limit increases and the lattice parameter shows a negative deviation from Vegard's law. To take advantage of the interphase boundary area, the minor phase tends to form a network of isolated precipitates. Vegard's law is observed when the solvent-solute bond length is equal to the average of the matrix and the precipitate phase, and a positive deviation when the bond length moves toward that of the precipitate phase. Some features of coherent phase equilibria will be compared between the DAM predictions and the previous results of Cahn and other investigators based on continuum elasticity.

11:20 AM

EQUILIBRIUM OF COHERENT PHASES IN A FILM: *Alexander L Roytburd*¹; *John W Cahn*²; ¹University of Maryland, Materials and Nuclear Engineering, College Park, MD 20742 USA; ²NIST, Materials Science and Engineering, Bld.223, Gaithersburg, MD 20899 USA

The elastic energy of two coherent phases in a film can be reduced to a minimum if the phases are arranged as two layers and the film is bent. This fact results in the appearance of a metastable two-phase equilibrium for a single-component system and in the modification of phase equilibria for a two-component system. The coherent phase diagrams for a bending film are presented and they are compared to the phase diagrams for a constrained film.

11:25 AM

MODELING KINETICS OF PHASE GROWTH WITH NON-EQUILIBRIUM INTERFACES: Brian Richard Hinderliter¹; *William C. Johnson*¹; ¹University of Virginia, Material Science and Engineering, Thornton Hall, Charlottesville, VA 22903-2442 USA

We model the kinetics of multiple phase growth via diffusion in a stressed binary alloy in which the interfaces are not in local thermo-

dynamic equilibrium. The numerical model employed uses a fine grid overlay in the region of the interface that tracks the interface, while using a coarser grid over the remainder of the material. The interface is assumed to be sharp, planar and coherent. Elastic deformation can result from misfit strain or applied stresses. The numerical model is compared to analytic solution in the limit of equilibrium boundary conditions for a binary regular solution. This work was supported by the National Science Foundation under Grant DMR-9496133.

11:30 AM POSTER VIEWING

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

Tuesday AM Room: Athens/Berlin
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: A. W. Thompson; J. Petit

8:30 AM KEYNOTE

DWELL AND ENVIRONMENTAL ASPECTS OF FATIGUE IN ALPHA/BETA TITANIUM ALLOYS: *W. J. Evans*¹; *M. R. Bache*¹; ¹University of Wales Swansea, Interdisciplinary Research Centre, Department of Materials Engineering, Singleton Park, Swansea SA2 8PP U.K.

In 1973, the Rolls Royce RB-2121 engine experienced several in-service catastrophic fan disc failures which had significant repercussions throughout the titanium industry. It became clear after major research efforts both in Europe and the United States that a number of factors were involved. Several of the problems such as inadequate work during forging, poor microstructural control and inherent defects were removed relatively quickly through changes in material – the beta processed IMI 685 was replaced with Ti-Ti-6Al-4V – improved melt practice and tighter control over working and heat treatment procedures. Other issues, such as a sensitivity to dwell under fatigue loading, have continued up to the present day and have even been implicated in recent in-service problems. The objective of the paper is to review the dwell sensitivity situation with a view to highlighting the mechanisms and arriving at a consensus understanding of the phenomenon and its implications. Over the intervening years many factors have been implicated in dwell fatigue including hydrogen and other interstitials, weak microstructural links, time dependent deformation, stress redistribution and the degree of biaxiality. The paper will explore the published work on these issues and will bring to the front unpublished research at Swansea and within the UK. This work encompasses mean stress effects, HCF/LCF interactions, tension/torsion fatigue response, 'internal' and external environments and the application of EBSD in the assessment of fatigue characteristics. It will be shown that the processes involved have important implications for fatigue crack initiation and short crack growth in general. Furthermore, the paper will explore the 'dwell' effect beyond the usual low temperature regime in which it is generally placed.

The implications for forging and microstructural control are assessed and the impact on component lifing philosophy discussed.

9:00 AM

EFFECT OF MICROSTRUCTURE ON THE DWELL FATIGUE BEHAVIOR OF Ti-6242: *Andrew P Woodfield*¹; Mark D Gorman¹; John A Sutliff²; ¹GE Aircraft Engines, MPED, 1 Neumann Way, M-89, Cincinnati, OH 45215 USA; ²GE Corporate Research and Development, 1 Research Circle, Schenectady, NY 12309 USA

This work describes the relationship between microstructure, and dwell low cycle fatigue life and fracture morphology in alpha-beta processed Ti-6242. It has been found that aligned alpha colonies that form during a beta quench in the billet cycle can survive during subsequent alpha-beta billet, forge and heat treat processing. The surviving aligned alpha colonies in forgings often cannot be recognized using conventional metallographic procedures and a new experimental technique for observing these features has been developed. This technique uses electron back scattered patterns (EBSP) generated from a sample in a scanning electron microscope and combines crystallographic information from these EBSPs to form an image of the microstructure based on crystallographic orientation of the basal pole. A method for quantifying the microstructure in terms of aligned alpha colony/alpha grain parameters has been developed and a model constructed which relates the microstructural parameters to dwell fatigue life.

9:20 AM

LOW CYCLE DWELL TIME FATIGUE IN Ti-6242: *R. Faber*¹; M. E. Kassner¹; Y. Kosaka²; B. Bristow¹; S. H. Reichman²; J. A. Hall²; ¹Oregon State University, Dept. of Mech. Engr., 204 Rogers Hall, Corvallis, OR 97331-6001 USA; ²Oremet Titanium, P.O. Box 580 530 34 Ave. SW, Albany, OR 97321

Ambient temperature low cycle dwell time and conventional low cycle fatigue tests were performed on Ti-6.0Al-2.0Sn-4.0Zr-2.0Mo-0.1Si (Ti-6242). Specimens were solution annealed at various temperatures below the beta transus to control the volume fraction of primary alpha. The influence of the changes in primary alpha phase on low cycle dwell time fatigue life (DCF) were determined and compared to the conventional low cycle fatigue (LCF) properties of the alloy. A dwell significantly decreased the number of cycles to failure. Increasing primary alpha associated with lower solution temperatures significantly increased susceptibility to low cycle dwell time fatigue although this effect diminished with decreasing stress. It is believed that the susceptibility to dwell fatigue may be associated with ambient temperature time dependent plasticity or (creep).

9:40 AM

CHARACTERISTICS OF FATIGUE SOFTENING AND HARDENING IN TITANIUM ALLOYS: *Fumio Morito*¹; Junji Takahashi¹; Seiichi Muneki¹; Toshio Kainuma¹; ¹National Research Institute for Metals, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

Details of fatigue softening behavior have been reported for the materials such as aluminum, copper, silver and iron. However fatigue properties in titanium alloys have scarcely been examined in connection with the characteristics of crystal structure and microstructure. We studied the fatigue behavior in the rolled and the recrystallized state of pure titanium, Ti-6Al-4V and Ti-3Al-8V-6Cr-4Mo-4Zr under the stress ratio(R) between 0.1 and -1. In pure titanium and Ti-3Al-8V-6Cr-4Mo-4Zr, the maximum stress of fatigue fracture in the rolled state was higher than that in the recrystallized one. In Ti-6Al-4V, however, the maximum stress of fatigue fracture under R=0.1 or -1 was nearly same irrespective of the rolled state or the recrystallized one. The maximum stress of fatigue fracture was higher under R=0.1 than that under R=-1. Fatigue softening was recognized in pure titanium and Ti-3Al-8V-6Cr-4Mo-4Zr. Fatigue hardening was very small in Ti-3Al-8V-6Cr-4Mo-4Zr. But the fatigue softening or hardening in Ti-6Al-4V was hardly recognized due to a small difference of the strength between the recrystallized state and the rolled one. Layered and elongated substructure due to fatigue deformation was observed in

pure titanium. But such a substructure was difficult to observe in Ti-6Al-4V and Ti-3Al-8V-6Cr-4Mo-4Zr.

10:00 AM

FATIGUE STRENGTH OF TITANIUM WELDS-DEFECT SENSITIVITY: *Stig Berge*¹; ¹Norwegian University of Science and Technology, Department of Marine Structures, Trondheim 7034 Norway

Compared to steel, titanium has a very good strength/weight ratio, low modulus of elasticity, and excellent corrosion resistance. Titanium is therefore well suited as a material for risers for offshore oil and gas production. Risers are subjected to significant fatigue loading. Whereas the fatigue properties of base material have been extensively studied, very little data is available on fatigue strength and defect sensitivity of heavy section titanium fusion welds. Fatigue data are presented for TIG welded plates and pipe coupons of two different medium strength alloys, with a discussion on defect sensitivity.

10:20 AM BREAK

10:40 AM INVITED PAPER

NUCLEATION AND PROPAGATION OF FATIGUE CRACKS IN B-TITANIUM ALLOYS: *J. O. Peters*¹; G. Lutjering¹; ¹Technische Universität Hamburg-Harburg, Eissendorfer Str. 42, Hamburg, FRG 21073 Germany

The influence of the microstructure on the fatigue properties of the two β -titanium alloys β -CEZ (developed by CEZUS, France) and VT 22 (Russia) was investigated. For the β -CEZ alloy a comparison between lamellar microstructures (β -processed) and bi-modal microstructures ($\alpha+\beta$ processed) at a yield stress level of 1200 MPa was performed: Bi-modal microstructures showed a higher ductility, higher LCF and HCF strength level and a higher resistance against microcrack propagation whereas lamellar microstructures showed a higher resistance against macrocrack propagation and a higher fracture toughness. These findings could be explained on the basis of β -grain size and α -plate dimensions. In the second part of this work a comparison between the bi-modal condition of the β -CEZ alloy and the VT 22 alloy was made. For this comparison the $\alpha+\beta$ processed VT 22 alloy was heat treated to the same yield stress level of 1200 MPa. The differences in mechanical properties will be discussed in terms of microstructural differences between the two alloys.

11:00 AM

MICROSTRUCTURAL ASPECTS OF FATIGUE CRACK INITIATION AND GROWTH IN Ti-10V-2Fe-3Al: *P. S. Shankar*¹; K. S. Ravichandran¹; ¹The University of Utah, Department of Metallurgical Engineering, 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

The influence of microstructure on fatigue crack initiation and growth response of the beta titanium alloy Ti-10V-2Fe-3Al was investigated. Three beta-heat-treated microstructures with nearly the same tensile strength levels were employed. One was unaged, one was $a\delta$ aged and the other was $w\delta$ -aged after furnace cooling from above the beta transus. Fatigue crack nucleation studies were performed using electropolished specimens under a stress ratio R of 0.1. Fatigue crack growth behavior at $R=0.1$ was determined using compact tension specimens. Fractography was employed to identify sites of crack nucleation and the mechanism of crack growth. Replication studies were performed to identify the relative importance of crack nucleation versus crack growth of fatigue life. The effects of microstructure on fatigue resistance are rationalized in terms of deformation characteristics, size and location of fatigue crack initiation site and the intrinsic resistance to fatigue crack growth.

11:20 AM

EFFECT OF AGING ON THE DEPENDENCE OF FATIGUE CRACK GROWTH BEHAVIOR ON MEAN STRESS (STRESS

RATIO) IN THE BETA TITANIUM ALLOY: Ti-10V-2Fe-3Al: S. K. Jha¹; K. S. Ravichandran¹; ¹The University of Utah, Department of Metallurgical Engineering, 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

Effect of mean stress (stress ratio, R) on fatigue crack growth (FCG) behavior of β - aged and ω - aged microstructures of the beta titanium alloy Ti-10V-2Fe-3Al was investigated. While the mean stress had a negligible effect on the FCG behavior of the β - aged microstructure, a strong effect was observed for the ω - aged microstructure. Crack closure levels for the β - aged microstructure were found to be very low compared to the ω - aged microstructure. Transmission and scanning electron microscopic studies of microstructures and fracture surfaces were performed to gain insight into the deformation characteristics and crack propagation mechanisms in these microstructures. The results are rationalized in terms of the effect of aging on slip and crack closure.

11:40 AM

EFFECTS OF ALPHA CASE ON FATIGUE STRENGTHS IN TITANIUM ALLOYS: H. Fukui¹; H. Iizumi¹; K. Minakawa²; ¹NKK America, 1 Kokan-cho, Fukuyama, Hiroshima 721-8510 Japan; ²NKK America, 450 Park Avenue, New York, NY 10022 USA

Formation of alpha case occurs during hot working, even during superplastic forming which is generally performed in argon gas. When Ti-6Al-4V is superplastically formed at approximately 900°C, alpha case of 50-100 μ m in thickness typically forms at the surface of the alloy. Since alpha case significantly degrades fatigue properties of titanium alloys, this oxygen contamination must be removed by chemical milling which is an expensive and rather environmentally unfriendly process. In recent years, considerable attention has been given to titanium alloys with lower superplastic forming temperatures, because these alloys should be able to reduce the degree of alpha case formation. It was reported that little alpha case (less than 5 μ m in thickness) was observed in Ti-4.5Al-2Mo-2Fe after superplastic forming at 775°C. However, oxygen enrichment took place in the region approximately 30 μ m in depth from the surface. It is not clear whether or not such very thin alpha case or the oxygen enriched region affects the fatigue strength of titanium alloys. The effect of alpha case on bending fatigue strength ($R = -1$) is being examined for Ti-4.5Al-2Mo-2Fe and Ti-6Al-4V in the present study. Specimens are carefully heat treated in argon gas at various temperatures in order to develop a variety of thickness in α case and the oxygen enriched region at the specimen surface. Fatigue test results will be discussed in terms of thickness of alpha case and depth of oxygen enriched region.

12:00 PM

CHARACTERIZING THE CRACKING BEHAVIOR OF HARD ALPHA DEFECTS IN ROTOR GRADE TiTi-6Al-4V ALLOY: P. C. McKeighan¹; R. C. McClung¹; A. E. Nicholls¹; L. Perocchi¹; ¹Southwest Research Institute, P.O. Drawer 28510, San Antonio, TX 78228-0510 USA

Improved methods for life management of aircraft turbine engine rotors are being developed with an initial focus on fatigue cracking at hard alpha defects in titanium (FAA Grant 95-G-041). Specimens manufactured from Ti-6-4 and containing small artificial hard alpha defects (with diffusion zones) at surface or subsurface locations were tested both statically and under fatigue loading. The goal was to determine the stresses required to nucleate and grow cracks in the hard alpha core, the surrounding diffusion zone, and the matrix material. The onset and development of cracking was detected during the tests using visual and nonvisual (potential drop, acoustic emission, and ultrasonic) techniques, along with post-test destructive sectioning. Results indicate large differences in cracking behavior depending upon defect size, defect position, and loading type.

HOT DEFORMATION OF ALUMINUM ALLOYS: Constitutive Modeling, Dislocations and Grain Boundaries

Sponsored by: Materials Design and Manufacturing Division, Structural Materials Division, Metal Processing & Fabrication of Met. 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

Tuesday AM

Room: Orchard

October 13, 1998

Location: O'Hare Hilton Hotel

Session Chairs: Lawrence Lalli, Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA; Jurgen Hirsch, VAW Aluminium AG, R&D, Bonn 53014 Germany

8:30 AM INVITED PAPER

MODELLING PLASTIC DEFORMATION OF ALUMINIUM ALLOYS: E. Nes¹; K. Marthinsen²; ¹Norwegian University of Science and Technology, Department of Metallurgy, Trondheim N-7034 Norway; ²SINTEF Materials Technology, Trondheim N-7034 Norway

A new approach to the modelling of work hardening during plastic deformation of metals has recently been proposed (E. Nes, Modelling work hardening and stress saturation in FCC metals, *Progr. Mater. Sci.*, in press; K. Marthinsen and E. Nes., A general model for metal plasticity, *Mater.Sci.Engineering. A234-236*, 1095, 1997). This model is based on a microstructural concept comprising three elements, the cell/subgrain size, d , the cell interior dislocation density, ρ_i , and the cell boundary dislocation density or the sub-boundary misorientation ρ_b or f . The model is further based on a statistical approach to the storage of dislocations and the model provides a solution to the basic "dislocation-book-keeping problem" by defining a differential equation which regulates the storage of dislocations into (i) a cell interior dislocation network, (ii) increases in boundary misorientation and (iii) the creation of new cell boundaries. By combining the solution for the dislocation storage problem with models for dynamic recovery of network dislocations and sub-boundary structures, a general internal variable solution is obtained. Recently the model has been further developed by including (i): the aspect of kinetics in the calculation of work hardening, (ii): the dynamic recovery aspect associated with the thermal stability of the sub-boundary structure, (iii): the grain aspect, i.e. polycrystalline metals as well as single crystals can be handled, and (iv): the alloying aspect associated with the interaction between migrating dislocations (and/or sub-boundaries) and both precipitate particles and atoms in solid solution. The result is a complete work hardening model and associated computer code capable of providing the stress strain behaviour for a given metal or alloy composition under any combination of constant strain rate and temperature. The model has been applied to the problems of work hardening of aluminium alloys. It is demonstrated that the model predictions, in terms of microstructure evolution and associated strengthening, are in good agreement with experimental observations.

9:00 AM INVITED PAPER

CONSTITUTIVE RELATIONSHIPS FOR MODELLING HOT ROLLING OF ALUMINUM-MAGNESIUM ALLOYS: C. M. Sellars¹; Q. Zhu¹; ¹The University of Sheffield, IMMPEUS, Institute of Microstructural and Mechanical Process Engineering, Mappin Street, Sheffield, S1 1 3JD UK

During industrial hot working operations, the deformation history of material is complex in terms both of changes in strain rate and changes in strain path. These changes can be accurately predicted as a function of position in the working stock by Finite Element Modelling, but their consequences on microstructural evolution are little understood. Most laboratory studies have been carried out at constant strain rate, with simple strain paths. Current research on Al-Mg alloys has employed systematic changes in strain rate and in strain path driving hot deformation. The as - deformed microstructures have been characterised in terms of internal dislocation, density, subgrain size and subgrain boundary misorientation. These have been used as internal state variables in modelling, in which the interrelationships between the evolution equations have been considered. The effect of the variations of deformation conditions on the subsequent static recrystallisation kinetics and the recrystallised grain size have also been measured and attempts have been made to incorporate the relationships in the microstructural evolution models.

9:30 AM INVITED PAPER

MULTISCALE MODELING OF THE PLASTICITY OF FCC CRYSTALS: *L. P. Kubin*¹; ¹LEM, CNRS-ONERA, 29 Av. de la Division Leclerc, BP 72, Chatillon Cedex 92322 France

The numerical modeling of crystal plasticity involves a combination of three approaches: microscopic (atomistic scale), mesoscopic (scale of the microstructure) and macroscopic (continuum modeling). The present state of the art is discussed in the case of fcc crystals in the domain of dislocation glide and at small strains. The connection between micro- and mesoscopic aspects is performed in series. The output expected from atomistic studies is essentially concerned with properties stemming from the core structure of dislocations. For instance, very promising results have been obtained in the past two years on cross-slip properties and the structure of dislocation junctions and locks. At the mesoscopic scale, the main issues are the formation of organised dislocation microstructures in monotonic and cyclic deformation and the structure of the corresponding internal stresses. Examples will be given of the output of a 3-D, mesoscopic, numerical model. Finally, the connection between meso- and macroscopic scales has to be performed in parallel by coupling a meso simulation with a FE code. Such connection between the discrete and continuum approaches of plasticity is now possible. It poses a few interesting questions that are discussed.

10:00 AM INVITED PAPER

CONSTITUTIVE MODELING OF A 5182 ALUMINUM AS A FUNCTION OF STRAIN RATE AND TEMPERATURE: *S. R. Chen*¹; *M. G. Stout*¹; *U. F. Kocks*¹; *S. R. MacEwen*²; *A. J. Beaudoin*³; ¹Los Alamos National Laboratory, MST-8: Structure/Property Relations, Material Science and Technology Division, Mail Stop G755, Los Alamos, NM 87545 USA; ²Alcan International; ³University of Illinois at Urbana-Champaign

We have measured the stress/strain response of a 5182 aluminum alloys as a function of strain rate and temperature. We have separated the response into two categories, when the material displays a yield drop and when it does not. The yield drop was only observed if the yield stress was below 70MPa. In this case the work-hardening curve was for practical purposes flat. Within this regime the deformation has been labeled "Class A" behavior. It occurs by continuous motion of dislocations accompanied by diffusion of solute. It is furthered shown that a constitutive relation such as is appropriate to describe deformation in this temperature/strain-rate regime where the solute drag mechanism dominates. In this expression QD is the activation enthalpy for self diffusion of Mg in aluminum, which is 131 kJ/mol. In the high-stress regime, where the yield stress is above 80MPa, there is positive work hardening associated with flow stress behavior of the 5182 alloy. The yield stress was nearly constant; however, the hardening and saturation flow stress increases with decreasing temperature and increasing strain rate. In this regime the deformation is dominated by dislocation accumulation and dynamic recovery. We have found that the Mechanical Threshold Strength (MTS) model accurately describes the constitutive response as a function of tem-

perature and strain rate. We have observed that at room temperature dynamic strain aging is the dominate mechanism and the 5182 exhibits negative strain-rate sensitivity. Sponsored by: Basic Energy Sciences, Division of Materials Sciences, US Department of Energy.

10:30 AM INVITED PAPER

FLOW STRESSES AND RHEOLOGICAL BEHAVIOUR OF MODEL Al-Mg ALLOYS DURING HOT PLANE STRAIN COMPRESSION: *J. H. Driver*¹; *F. Basson*¹; ¹Ecole des Mines de Saint Etienne, Department of Microstructures and Processing, 158 Cours Flauriel, Saint-Etienne, Cedex 2 42023 France

The final properties of non-heat-treatable aluminum alloys depend to a great extent on the thermomechanical conditions of the hot deformation process. Therefore, constitutive equations relating the flow stress of a material with a known microstructure to the strain, strain rate and deformation temperature are essential for modeling and controlling the thermomechanical processing operations. Four model Al-Mg alloys (AA1070 with 0, 1, 3 and 5 wt.% Mg) have been deformed in plane strain compression up to a strain of unity using a hot channel die device. Deformation temperatures were between 20 and 500C and strain rates between 10⁻³ and 10 s⁻¹. Low strain rate tests were used to derive the rheological parameters from the isothermal stress-strain curves. Different approaches have been used for the work hardening analysis; though satisfactory results are obtained with a combination of linear and power law hardening, the best agreement is given by a modified Kocks-Mecking analysis of the work hardening rate. The accuracy of the parameters determination is discussed.

11:00 AM INVITED PAPER

GRAIN BOUNDARY PROPERTIES FOR MICROSTRUCTURAL EVOLUTION AT HIGH TEMPERATURES: *A. D. Rollett*¹; *B. L. Adams*¹; *D. Kinderlehrer*¹; *W. W. Mullins*¹; *S. Ta'asan*¹; ¹Carnegie Mellon University, Materials Science & Engineering, Rm 3327, Wean Hall, 5000 Forbes Avenue, Pittsburgh, PA 15213-3890 USA

Detailed descriptions of the properties of grain boundaries as a function of misorientation (and inclination) would be of great value in many systems and applications. This is particularly relevant to the challenge of understanding microstructural evolution via grain growth and recrystallization at high temperatures. We describe a new approach to extracting grain boundary excess free energy and (curvature driven) boundary mobility as a function of crystallographic type. The method depends on measuring very large numbers of triple junction (TJ) configurations in order to obtain statistically valid samples of relationships between energies (or mobilities) of every combination of type. The method provides relative energies (mobilities) over the entire fundamental zone and therefore requires calibration with absolute measurements of at least one reference type that is obtained independently. The extraction of boundary energies assumes local equilibrium at each TJ thus permitting the application of Herring's relations. The method is illustrated by an application to the case of a material with a strict fiber texture for which the boundary type can be simplified to a one-parameter description. The extraction of mobilities assumes that boundary velocity is proportional to energy and curvature but does not require equilibrium at each TJ. As for boundary energies the set of relative mobilities must be calibrated by the measurement of the absolute mobility at least one boundary type. Preliminary results of serial sectioning experiments on magnesia, copper and aluminum polycrystalline samples are described.

11:30 AM INVITED PAPER

GRAIN BOUNDARY EVOLUTION DURING PROCESSING AND RECRYSTALLIZATION OF SUPERPLASTIC ALUMINUM ALLOYS: *T. R. McNelley*¹; *M. E. McMahon*¹; *M. T. Perez-Prado*²; ¹Naval Postgraduate School, Department of Mechanical Engineering, 700 Dyer Road, Monterey, CA 93943-5146 USA; ²Centro Nacional de Investigaciones Metalurgical (CENIM), Avda, De Gregorio del Amo 8, Madrid Spain

Microtexture and grain boundary misorientation distributions have been studied in several superplastic aluminum alloys by means of computer-aided electron backscatter diffraction (EBSD) analysis methods. Two distinct transformation paths from the deformation microstructure of as-processed material may enable superplastic response. In alloys such as Supral 2004, the development of a superplastically enabled microstructure occurs by a continuous reaction, i.e. in a homogenous manner, during elevated temperature annealing and superplastic straining of the material. Bimodal grain boundary misorientation distributions are frequently observed in association with such reactions; such distributions may be interpreted by models combining misorientations between symmetric variants of texture components with misorientations reflecting dislocation reactions during processing. Alternatively, in aluminum alloys such as 5083 and 7475 superplastic microstructures develop by recrystallization processes involving the heterogeneous formation and subsequent growth of grains by the migration of high-angle grain boundaries. The new grains typically form within the deformation zones surrounding coarse precipitate particles and the associated grain orientations tend to be random. Correspondingly, the grain boundary misorientations tend toward random. The evolution of these different misorientation distributions during elevated temperature straining will also be described in terms of slip and grain boundary sliding.

INTERSTITIAL AND SUBSTITUTIONAL SOLUTE EFFECTS IN INTERMETALLICS II: Unusual Intermetallics Site Occupancy and Modelling

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

Tuesday AM Room: Madrid
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: D. Mikkola, Michigan Technological University, Department of Metallurgical and Materials Engineering, Houghton, MI 49931-1295 USA; M. J. Kaufman, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611 USA

8:30 AM

SOLUTE EFFECTS IN EXOTIC INTERMETALLICS: David P. Pope¹; Yoshisato Kimura¹; David E. Luzzi¹; Alexander Goldberg²; ¹University of Pennsylvania, Materials Science and Engineering, 3231 Walnut St., Philadelphia, PA 19104 USA; ²Cercon, Howmet-Cercast Group, 201 Cercon Dr., Hillsboro, TX 76645 USA

This talk will emphasize the effects of substitutional solutes on the mechanical properties of cubic Laves phases, with special emphasis on twinning in HfV2 alloyed with Nb. Binary HfV2 is very brittle, showing plastic flow only at quite high homologous temperatures, but alloying with Nb greatly increases the deformability at low temperatures. The deformation at low temperatures is almost totally the result of twinning in the Laves phase. Even more interesting is the fact that as the temperature increases, a region is reached where neither twinning nor dislocation slip occurs. At still higher temperatures dislocation slip is observed. In an effort to increase the deformability of this alloy, we have considered various mechanisms

that could give rise to the increased deformability. This paper will concentrate on the mechanisms we have concluded are most likely to be responsible for the ease of twinning in Nb-alloyed HfV2.

9:00 AM

SITE OCCUPANCIES AND LONG RANGE ORDERING PARAMETERS USING THE ORDERING TIE-LINE METHOD: H. L. Fraser¹; I. P. Jones²; R. Banerjee¹; J. Nan²; S. Banerjee³; R. J. Grylls¹; ¹The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; ²The University of Birmingham, School of Metallurgy and Materials, Birmingham, B15 2TT United Kingdom; ³Bhabha Atomic, Research Center, Bombay, 400 085 India

Complex ordered intermetallic alloys often have compositions which are far from those corresponding to stoichiometry and also exhibit ordering schemes which result in the alloys being not fully ordered. To understand the properties of such materials and also to be able to supply an accurate physical description of these materials to those involved in computational efforts, it is important that the site occupancies and degree of long range order be characterized accurately and precisely. A relatively new technique for determining these quantities, the ordering tie line method, will be described and then applied to Nb and Ti based ordered intermetallics. Computational efforts aimed at determining these same quantities by solution thermodynamics have been undertaken, and the computational and experimental results will be compared and contrasted.

9:30 AM

APPLICATIONS OF ALCHEMI TO ORDERED INTERMETALLIC ALLOYS: Ian M. Anderson¹; L. M. Pike¹; A. J. Duncan¹; J. Bentley¹; ¹Oak Ridge National Laboratory, Metals & Ceramics Division, Oak Ridge, TN 37831-6376 USA

The usefulness of ALCHEMI (atom location by channeling enhanced microanalysis) in the development of intermetallic alloys is best seen in the context of systematic studies, where trends in site occupancies can be correlated with other alloy properties. For example, an ALCHEMI study of 3d-transition elements (Me) from Ti to Cu Fe₃₀Al₄₅Me₅ yields site distributions that cannot be understood only in terms of site preferences but must also take into account the site-equilibration mechanism, thus demonstrating the relative importance of thermodynamic and kinetic factors in alloy processing. Another ALCHEMI study of alloys with compositions across the entire B2 phase field in the system Ni-Fe-Al suggests the role of Fe as a buffer element, while helping to explain the variations in mechanical properties with the composition of these ternary alloys. Other applications of ALCHEMI to L1₂-ordered gamma prime particles, L1₀-ordered TiAl, and C15-ordered Laves phase intermetallics will also be presented.

10:00 AM

SOLUTE EFFECTS ON DEFORMATION AND FRACTURE OF Ni₃Si: T. Takasugi¹; ¹Tohoku University, Institute for Materials Research, Katahira 2-1-1, Aoba-ku, Sendai, 980-77 Japan

Nickel silicide based on L12-type Ni₃Si is considered to be utilized as high temperature structural materials because of low density, high strength at elevated temperatures, and superior corrosion and oxidation resistance. In this talk, the effects of Ti element as substitutional, and boron, carbon and hydrogen as interstitial on the deformation and fracture behavior of Ni₃Si are reviewed, based on the results of single crystals and polycrystals. It is mentioned that Ti addition (at atomic pct) to Ni₃Si has the effect of strengthening the matrix, shifting the positive temperature dependence of the strength toward higher temperature and also improving the ambient tensile ductility via enhancing intergranular cohesion. Absorbed or residual hydrogen significantly reduces the intergranular cohesion, resulting in the intergranular fracture. On the other hand, the doping of boron and carbon (in ppm level) to Ni₃Si has the effects of strengthening the matrix, and dramatically improving the ambient tensile ductility via preventing the intergranular fracture due to hydrogen.

10:30 AM

PROGRESS IN THE MODELING OF ORDERED INTERMETALLIC ALLOY MICROSTRUCTURES: *Guillermo Bozzolo*¹; Ronald D. Noebe²; Frank S. Honey²; ¹Ohio Aerospace Institute, 22800 Cedar Point Road, Cleveland, OH 44142 USA; ²NASA Lewis Research Center, Materials Division, 21000 Brookpark Road, Cleveland, OH 44135 USA

A detailed understanding of the structure of multicomponent systems relies heavily on the behavior of the individual component elements and their interaction with each other. Basic problems, like the precise site substitution scheme of specific alloying elements and their dependence with concentration and the number of other additions are necessary in order to predict and understand the effect of various alloying schemes on the physical properties of a material, its response to various temperature treatments, and the resulting mechanical properties. It is only recently that theoretical methods can provide useful insight in this area, as most current techniques suffer from strong limitations mainly in the type of elements considered as well as the crystallographic structure of the phases that form. The Bozzolo-Ferrante-Smith (BFS) method for alloys was designed to overcome these limitations, with the intent of providing a useful tool for the theoretical prediction of fundamental properties and structure of such complex systems. After a brief description of the method and its range of applications, we concentrate on the use of BFS for the determination of site substitution schemes for individual as well as collective alloying additions to intermetallic systems, mainly NiAl, as well as the resulting behavior with respect to solubility limits and second phase formation, and the concentration dependence of the lattice parameter. In addition, we show the results of large scale numerical simulations based on BFS for calculation of the energetics of these systems, providing useful insight on the temperature dependence of these properties. Attention is also given to the AB₂A₃BC two phase alloys and the influence of alloying additions on the crystallographic misfit between these phases, including results on their partitioning behavior.

11:00 AM

STRAIN RATE SENSITIVITY OF Cr-STABILIZED CUBIC TITANIUM TRIALUMINIDES: *Steven J. Miller*¹; Thomas D. Wood¹; Donald E. Mikkola¹; ¹Michigan Technological University, Department of Metallurgical and Materials Engineering, Houghton, MI 49931-1295 USA

Negative strain rate sensitivities and serrated flow have been commonly observed with the various titanium trialuminides in the range 400-700K. These effects have usually been attributed to dynamic strain aging, but no specific solutes have been identified. In this study, the effects of selected site substitutions of solutes on the strain rate sensitivity of a Cr-stabilized trialuminide have cast doubt on the dynamic strain aging mechanism. Detailed studies of the mechanical properties of several trialuminides as a function of temperature and strain rate will be presented, and then compared to the behavior of other phases with the same structure that have been tested in the same manner. Alternate means for understanding the occurrence of negative strain rate sensitivities with only certain of these alloys will be discussed. The support of NSF (DMR-9400507) is gratefully acknowledged.

11:30 AM

EFFECTS OF TERNARY ALLOYING ON THE STRENGTH AND DUCTILITY OF Al₂Ti INTERMETALLIC COMPOUND: *J. C. Ma*¹; M. J. Lukitsch¹; J. E. Benci¹; ¹Wayne State University, Materials Science and Engineering, Detroit, MI 48202 USA

As-cast ternary-alloyed Al₂Ti + X compositions were produced by repeated arc melting of the appropriate masses of high purity Ti, Al and one of the following ternary elements: Si, V, Cr, Mn, Fe, Ni, Cu, Mo or W. Each alloy nominally contains 2 at.% of the ternary element while the Al:Ti ratio was maintained at 2 to 1. The microstructures were characterized by SEM/EDS. The compressive yield strength of each alloy was between 300YC and 1000YC. The compressive plastic strain-to-failure was also measured for each composition

at room temperature and 500YC. All ternary alloying elements studied except V increase the 300YC yield strength of Al₂Ti. Si has the greatest strengthening effect at this temperature. All ternary alloys with the exception of the Si-containing alloy have lower strengths at 1000YC than binary Al₂Ti. The transition from higher strength at lower temperatures to lower strength at higher temperatures generally occurs around 800YC. Binary Al₂Ti exhibits 0.2% plastic strain-to-failure at room temperature. Cr-, Si- and W-alloyed Al₂Ti exhibit 0.4 to 0.6% plastic strains-to-failure while Fe-alloyed Al₂Ti fails after accumulating 1.0% plastic strain. Three of the other ternary alloys exhibit no measurable plastic strain-to-failure at room temperature while two show only marginally improved or the same strain-to-failure as the binary alloy. At 500YC, binary Al₂Ti exhibits 1.5% plastic strain-to-failure. With the exception of Mn, all of the ternary additions increase the plastic strain-to-failure at this temperature compared to the binary alloy. The Fe-containing alloy exhibits 10% plastic strain-to-failure at 500YC, the most of any of the ternary alloys studied.

MECHANICAL PERFORMANCE OF LAMINATED COMPOSITES: Structure-Property Correlations

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

Tuesday AM

Room: Paris C

October 13, 1998

Location: O'Hare Hilton Hotel

Session Chairs: Eric Taleff, University of Texas at Austin, Dept. of Aerospace Engineering and Engineering Mechanics, Austin, TX 78712 USA; Desiderio Kovar, University of Texas at Austin, Department of Mechanical Engineering, Austin, TX 78712 USA

8:30 AM INVITED PAPER

INFLUENCE OF INTERFACIAL FRACTURE RESISTANCE ON THE MECHANICAL BEHAVIOUR OF LAYERED CERAMICS:

*Desiderio Kovar*¹; M. D. Thouless²; ¹University of Texas at Austin, Department of Mechanical Engineering, Texas Materials Institute, Austin, TX 78712 USA; ²University of Michigan, Mechanical Engineering and Applied Mechanics Department, 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.

9:00 AM

IMPACT OF INTERFACIAL ELECTRONIC BAND STRUCTURE EFFECTS ON MULTILAYER STABILITY: *T. W. Barbee*¹; ¹Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

TUESDAY AM

It has been experimentally observed that copper wets many transition metals and transition metal compounds. This wetting is unexpected as the copper - transition metal binary alloys exhibit terminal solid solubilities and miscibility gaps in most cases. It has recently been experimentally observed that there is an electron transfer at the interfaces in copper - transition metal multilayer nano-laminates that results in a decrease in the d band occupancy of the copper at the interfaces and an increase in the d band occupancy of the transition metals at the interfaces. Calculations indicate that the first two monolayers in each material at an interface are involved in the electron transfer. This electron transfer results in the copper being more strongly bonded to the transition metal than to itself, an effect that may represent an increase in the bonding energy of 25 % of that characteristic of copper on copper without alloying. In this study the thermal stability of copper transition metal multilayers has been studied. Specific results for multilayers of copper-niobium, copper-vanadium, copper-vanadium carbide and copper-chromium carbide are presented and discussed in terms of the interfacial electron transfer and its effect on interface stability.

9:20 AM

MECHANICAL PROPERTIES/MICROSTRUCTURE RELATIONSHIP IN ELECTRODEPOSITED Cu/Ag LAMINATED NANOCOMPOSITES: *Qing Zhai*¹; Fereshteh Ebrahimi¹; ¹University of Florida, Dept. of Materials Science & Engineering, Gainesville, FL 32611-6400 USA

It has been widely reported that the laminated composites made up of two "soft" metals may display very high strength when the microstructural scale is in the nanometer range. The Cu-Ag laminated nano-composites studied in this paper are produced by electrodeposition using a single-bath cyanide solution. The effects of the copper layer thickness and low temperature annealing on the strengthening and the fracture behavior of these composites were investigated. Tensile testing was conducted to investigate the mechanical properties of the specimens. TEM and X-ray diffraction were employed to analyze the microstructure. SEM and AFM were used to study the fracture surfaces. The mechanical property-microstructure correlation and the explanation of the fracture behavior of these composites are presented.

9:40 AM

POTENTIAL OF MULTILAYER NANO-LAMINATES FOR TRIBOLOGICAL APPLICATIONS: *Tai D. Nguyen*¹; Troy W. Barbee¹; ¹Lawrence Livermore National Laboratory, Chemistry and Materials Science Department, 1L-350, Livermore, CA 94551 USA

Nano-laminate multilayers exhibit enhanced mechanical properties and thus are potential candidates for wear resistant coating applications. In this experiment, we study the mechanical and tribological properties of multilayer nano-laminates having bilayer thickness ranging from two to hundreds of nanometers prepared by magnetron sputtering. The microstructure was characterized using x-ray diffraction and transmission electron microscopy techniques. The effects of thermal annealing on the structure stability and performance are reported. Tribological results obtained from a pin-on-disc geometry apparatus, and residual stress and hardness are presented for both as-deposited and annealed samples. Correlations between the microstructure and mechanical and tribological performance of the multilayer laminates are discussed.

10:00 AM BREAK

10:20 AM

Nb/Nb₅Si₃ MICROLAMINATES FOR HIGH-TEMPERATURE STRUCTURAL APPLICATIONS: A. J. Gavens¹; D. Van Heerden¹; C. H. Shang¹; T. Foecke²; *T. P. Weihs*¹; ¹The Johns Hopkins University, Materials Science & Engineering Department, Baltimore, MD 21218 USA; ²National Institute of Standards and Technology, Gaithersburg, MD 20899 USA

Microlaminate foils with alternating layers of Nb and Nb₅Si₃ hold considerable promise as a new structural material for high-temperature aerospace applications. The Nb layers enhance fracture toughness at low temperatures, the Nb₅Si₃ layers provide creep strength at elevated temperatures, and both phases are chemically stable to temperatures above 1600°C. In this paper we describe mechanical characterizations of Nb/Nb₅Si₃ microlaminate foils that were sputter deposited near room temperature and then thermally processed at 1200°C. The free-standing foils are 50µm thick and contain many Nb and Nb₅Si₃ layers that individually measure 0.2µm, 1.0µm, or 5.0µm in thickness. The Nb phase is polycrystalline and equiaxed with a strong {110} texture while the Nb₅Si₃ phase is polycrystalline and equiaxed but random in orientation. The room temperature mechanical properties of the microlaminates were examined by performing tension tests on free-standing specimens. The measured fracture strengths range from 500 to 1000 MPa, and the fracture surfaces show clear signs of ductility within the Nb layers and brittle fracture within the silicide layers. Residual thermal stresses were also quantified for the specimens using asymmetric X-ray diffraction experiments. The results demonstrate that large (>100 MPa) residual stresses exist in the as-processed foils with the silicide layers being in residual compression. Variations in strength and ductility will be reported as a function of residual stress, layer thickness, and volume fraction of Nb.

10:40 AM

INFLUENCE OF PROCESSING VARIABLES ON MECHANICAL PROPERTIES OF 6090/SiC/25P//6013 LAMINATES: *C. K. Syn*¹; D. R. Lesuer¹; J. D. Rigney²; R. W. Bush³; ¹Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; ²General Electric Aircraft Engines, M/D M85, 1 Newman Way, Cincinnati, OH 45215-6301 USA; ³Alcoa Technical Center, Aluminum Co. of America, 100 Technical Drive, Alcoa Center, PA 15069 USA

The influence of bonding process and subsequent heat treatment on the mechanical properties of Al 6090/SiC/25p // Al 6013 laminates has been studied. A key processing variable in this study was the bonding time which was varied from 2 seconds to 2 hours. The variations in processing had significant influence on the interfaces between layers and the tendency for local delamination during subsequent testing. The mechanical properties were characterized by measuring fracture toughness in the crack arrester orientation (using straight notched three-point bend bar specimens); fracture toughness in the direction of the interfaces (using chevron notched short bar specimens); tensile properties in the parallel and normal directions to the constituent layers; and interface shear strength using lap-shear type of tests. It was shown that the bonding time was less significant than the heat treatment in affecting the tensile strength, toughness, and interfacial characteristics. Processing details and inter-relationships between the measured properties and failure behavior will be presented.

11:00 AM

PROCESSING AND CHARACTERIZATION OF MICROSCALE Ni-Nb MULTILAYER COMPOSITES PRODUCED BY COLD ROLLING: *F. H. Kavaranan*¹; K. S. Ravichandran¹; ¹University of Utah, Department of Metallurgical Engineering, 412 WBB, Salt Lake City, UT 84112 USA

An attempt was made to fabricate microscale Ni-Nb multilayer composites by diffusion bonding of alternately stacked high purity sheets followed by cold rolling. The general deformation behavior of these multilayers and the evolution of microstructure and hardness were studied. Hardness measurements were made as a function of nominal reduction during rolling. X-ray diffraction was used to study the possibility of formation of intermetallic compounds after heavy reduction. The development of {110} Ni preferred orientation and {100} Nb preferred orientation was observed in the multilayers. As the thickness of the laminate decreased, the microhardness of the laminate increased. For average thicknesses below 2 mm, a strong increase in microhardness was observed. Microstructural features and strengthening due to bilayer refinement were analyzed.

11:20 AM

PROCESSING, STRUCTURE AND PROPERTIES OF ALUMINUM-ALUMINIDE LAYERED COMPOSITES: *D. E. Alman*¹; ¹Albany Research Center, U.S. Department of Energy, 1450 Queen Avenue, S. W. Albany, OR 97321 USA

Laminate composites comprising of alternating aluminum and aluminide layers were produced by reactive bonding Al foils with either Ni, or Mg foils. Al foils (0.250 mm thick) were bonded to the thinner Ni foils (e.g., 0.025mm thick) by hot-pressing at 600°C and 20 MPa for 120 minutes. This produced a composite microstructure consisting of alternating Al and NiAl layers, with a variety of phases at the interface. This structure could be driven to equilibrium (e.g., alternating Al and Al₃Ni layers) by subsequent vacuum heat-treatment. For Mg-Al laminates, hot-pressing was performed at 400°C for 120 minutes. This produced a composite consisting of alternating Al, Mg₂Al₃, Mg₁₇Al₁₂, and Mg layers. Presented will be the microstructure, as well as, the tensile properties of these composite.

11:40 AM

DAMPING IN LAMINATED METAL COMPOSITES: *Donald R. Lesuer*¹; *Brian Bonner*¹; *Chol K. Syn*¹; *Albert E. Brown*¹; ¹Lawrence Livermore National Laboratory, Livermore, CA 94551 USA

Laminated metal composites (LMCs) can improve the damping response of structural materials through the activation of damping mechanisms associated with planar interfaces and abrupt changes in elastic constants on going from one layer to another. These damping mechanisms are additive to the ones active in the individual layers from which the LMC is composed. In many cases, the LMC can have higher damping capacity than the component materials. In this presentation we will describe damping studies conducted at low frequencies (2-40 Hz) and ultrasonic frequencies (2-30 MHz). Results at low frequency have been obtained on ultrahigh carbon steel (UHCS) / brass and Ti-6Al-4V / Al-Si-Fe and results at high frequency have been obtained on Al / Al-SiC, UHCS / brass, Mg-9%Li / Al-SiC and UHCS / Ti-6Al-4V. The influence of layer thickness and frequency have been studied. Possible mechanisms of damping in LMCs will be discussed.

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

Tuesday AM Room: Paris A
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: *Daniel B. Miracle*, Air Force Research Laboratories, Wright Patterson Air Force Base, OH USA

8:30 AM INVITED PAPER

HIGH ENERGY ABSORPTION COMPOSITES AND THE LOCALIZATION TO DELOCALIZATION TRANSITION: *N. Sridhar*¹; *B. N. Cox*¹; *J. B. Davis*¹; *F. W. Zok*²; ¹Rockwell Science Center, Thousand Oaks, CA 91358 USA; ²University of California, Materials Department, Santa Barbara, CA 93106 USA

We have developed novel composites possessing an exceptional capacity for high energy absorption. The high energy absorption is achieved by designing a reinforcement topology that has a large den-

sity of sites for damage initiation. This promotes "lock-up" leading to a change in damage from a localized to a delocalized mode of failure. The composite hardens over large strains (~100%) following the onset of damage. We demonstrate this concept in model composite systems consisting of an epoxy matrix and steel chains. Models for the deformation response as a function of the geometrical and material parameters are developed and with the aid of these results, the composite architecture is optimized.

8:55 AM

ON THE DETERMINATION OF FIBER FAILURE IN CONTINUOUSLY REINFORCED TITANIUM MATRIX COMPOSITES DURING ELEVATED TEMPERATURE TESTING EMPLOYING MODAL ACOUSTIC EMISSION: *A. H. Rosenberger*¹; *D. A. Stubbs*²; *N. E. Ashbaugh*¹; *P. M. Schubel*²; *D. J. Buchanan*²; ¹Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLN, Wright-Patterson AFB, OH 45433-7817 USA; ²University of Dayton Research Institute, 1031 Irving Avenue, Dayton, OH 45419-0218 USA

Fiber failure is the dominant damage mechanism during high temperature application of titanium matrix composites that incorporates periods of sustained loading. The accurate determination of the number of fiber failures is necessary for accurate modeling of the damage progression and determining the burst margin remaining after creep exposure. A new acoustic emission technique, incorporating broad band monitoring and modal analysis, has been applied to tests of composite samples in order to more accurately quantify fiber failures during mechanical testing at elevated temperatures. Alumina wave guides have been incorporated to allow monitoring at the elevated temperatures while maintaining the fidelity of the acoustic emission event. This technique has been applied to a unidirectional continuously reinforced titanium matrix composite, SCS-6/Ti-6Al-4V. Characteristics of the fiber break signature are discussed and the method is verified using an interrupted test in which the actual number of fiber failures is compared to the modal acoustic emission results.

9:20 AM

IN SITU OBSERVATIONS AND ACOUSTIC EMISSION ANALYSIS FOR SIGMA 1140+/Ti=6-4 SiC FIBRE REINFORCED TITANIUM COMPOSITES UNDER TENSILE TRANSVERSE LOADING: *X. Wu*¹; *H. Mori*²; *P. Bowen*¹; ¹The University of Birmingham, School of Metallurgy and Materials, Birmingham B15 2TT UK; ²The University of Tokyo, Research Centre for Advanced Science and Technology, 4-6-1 Komaba, Meguro-ku, Tokyo 153 Japan

The transverse response of 8 and 21% volume fraction of Sigma 1140+ SiC fibre reinforced Ti-6-4 matrix composites has been investigated in situ in a FEG-SEM using direct observation. Damage has also been monitored in air using conventional transverse tensile tests and Acoustic Emission (AE). An advanced acoustic emission system has been used to locate the position of damage and to collect AE events during such conventional tests. In situ observations have found that debonding of matrix/fibre interfaces in such a weakly debonded type of composite starts at a very low stress and does not produce a distinct knee on a conventional stress-strain curve. The first knee observed on the stress-strain curve actually corresponds to cracking of carbon-coating layers, rather than fibre/matrix interface debonding which has been previously assumed, and the second knee corresponds to local micro-cracking of the matrix near interfaces. Many AE events were recorded during cracking of the carbon-coating and were of amplitude of up to 90 dB and of energy of up to 160 mJ. Near to the end of the test, AE signals were again received in large numbers but now with an amplitude up to 70 dB and with an energy of less than 20 mJ. This combination of in situ observations and acoustic emission analysis has allowed the development of damage, as a function of volume fraction of fibres, to be quantified.

9:45 AM INVITED PAPER

STRENGTH AND FAILURE BEHAVIOR OF STITCHED CARBON/EPOXY COMPOSITES: *Ryuta Kamiya*¹; *Tsu-Wei Chou*¹; ¹Uni-

versity of Delaware, Center for Composite Materials and Department of Mechanical Engineering, Newark, DE 19716 USA

This paper presents a study of the effect of through-the-thickness stitching fibers upon the strength and failure behavior of multi-directionally reinforced composites. The woven fabric laminae were placed in four direction, (1, +45, 90), to form a quasi-isotropic preform. The fabrics have open space between adjacent tows. These inter-yarn spaces allow easy insertion of the through-the-thickness stitching fibers without seriously damaging the in-phase fibers. Fiber volume fractions over 58% were obtained by this method. The through-the-thickness yarn sizes used in this project were 2k, 4k, and 6k. Non-stitched preforms were also designed and manufactured with the same fiber and by the same procedure for the control experiments. All preforms were infiltrated with epoxy resin by the RTM technique. In-plane tensile and compressive strength, interlaminar shear strength, and Mode I fracture toughness have been measured at three through-the-thickness fiber contents for the carbon/epoxy composites. Although the through-the-thickness stitching fibers significantly enhance the Mode I fracture toughness, they tend to degrade the in-plane tensile and compressive strength. The failure process under interlaminar shear loading the DNS tests show two distinct stages: the fiber-matrix interfacial failure followed by the breakage/debonding of the through-the-thickness fibers. The through-the-thickness fibers cause a reduction of the initial failure load in the first stage, but can enhance the final failure load in the second stage. In composites with 6k through-the-thickness fibers, the final failure load can exceed the initial failure load. SEM and optical microscopic examinations have also been conducted for observing the failure mechanisms and fracture surfaces.

10:10 AM INVITED PAPER

NOTCH-SENSITIVITY OF FIBER-REINFORCED CERAMIC MATRIX COMPOSITES: EFFECTS OF INELASTIC STRAINING AND VOLUME-DEPENDENT STRENGTH: *F. W. Zok*¹; J. C. McNulty¹; G. M. Genin²; A. G. Evans³; ¹University of California, Materials Department, Santa Barbara, CA 93106 USA; ²Harvard University, Division of Applied Science, Cambridge, MA 02138 USA; ³Cambridge University, Currently with Engineering Department, Cambridge, England CB2 1PZ USA

The effects of circular holes and sharp notches on the tensile strength of several fiber-reinforced ceramic composites have been investigated. The role of inelastic straining in the local re-distribution of stress has been elucidated through measurements of the local strains in the regions of high stress concentration, coupled with finite element simulations of the test geometries, using a nonlinear constitutive law appropriate to CMCs. The scale-dependence of strength has been inferred from tests performed on specimens of varying size. The utility of two failure models that incorporate both the inelastic straining and the scale-dependence have been assessed: one based on the point stress failure criterion and the other on weakest link fracture statistics. Both approaches provide a reasonably consistent description of the experimental measurements. Some of the implications and limitations associated with the failure models will be discussed.

10:35 AM BREAK

10:50 AM INVITED PAPER

INITIATION OF TRAVERSE FRACTURE IN COMPOSITES FROM NONUNIFORM MICROSTRUCTURES: *Ramesh Talreja*¹; ¹Georgia Institute of Technology, School of Aerospace Engineering, Atlanta, GA 30332 USA

In fiber reinforced composites subjected to mechanical loads the first event of failure at the microscopic level is commonly believed to be fiber/matrix debonding. However, in polymer matrix composites with a glassy polymer, e.g., epoxy, as a matrix, the transverse fracture may actually initiate in the matrix before the fiber/matrix bond fails. Recently, Asp et al. (1996) investigated this and proposed cavitation-induced brittle cracking of the matrix as a critical mechanism governed by the dilatation energy density. We have recently conducted a

study of damage initiation in composites by computationally simulating nonuniform fiber distributions in an actual (as-processed) ceramic matrix composite (Bulsara, et al, 1998). The present study investigates transverse failure in polymer matrix composites under remotely imposed uniaxial and biaxial mechanical loads in addition to the residual stresses caused by thermal cooldown. Several interesting features of the failure process are revealed, noteworthy being the fact that the transverse failure tends to occur in a narrow range of the overall strain.

11:15 AM

TRANSVERSE CREEP RESPONSE OF Ti-22Al-23Nb/SiC REINFORCED COMPOSITES: *S. Woodard*¹; T. M. Pollock¹; ¹Carnegie Mellon University, Carnegie, PA USA

SiC-reinforced intermetallic matrix composites potentially offer high stiffness and specific strength at elevated temperatures. In some applications for which these materials are targeted, the transverse creep strength could be performance limiting. Both the matrix creep deformation and the fiber / matrix interface play a significant role in the transverse response of these materials. The research presented will focus on the interdependence of inhomogeneous local matrix plasticity and fiber/matrix interface damage during transverse creep of SiC-unidirectionally reinforced Ti-22Al-23Nb intermetallic matrix composites. Correlations between the push-out response of individual fibers, their fiber spacing environment and the macroscopic response of two Ti-22Al-23Nb intermetallic matrix composites reinforced with different SiC fibers will be discussed.

11:40 AM

TEXTURE AND RESIDUAL STRAIN IN SiC/Ti-6-2-4-2 TITANIUM MATRIX COMPOSITES: *Partha Rangaswamy*¹; M. A.M. Bourke¹; R. Von Dreele¹; R. Von Dreele¹; K. Bennett¹; J. A. Roberts²; N. Jayaraman¹; ¹Los Alamos National Laboratory, Lujan Scattering Center, H 805, LANSCE 12, Los Alamos, NM 87545 USA; ²University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Residual strain and texture variations were measured in two Titanium matrix composites reinforced with Silicon Carbide fibers (Ti/SiC) of similar composition but fabricated by different processing routes. Each composite comprised a Ti-6242 a/b matrix alloy containing 35% by volume continuous SiC fibers. In one case, the matrix was produced by a plasma spray (PS) route, and the other by a wire drawing (WD) process. The PS and WD composites were reinforced with SCS-6 (SiC) and Trimarc (SiC) fibers, respectively. The texture in the monolithic and composite Ti matrices differed significantly, from approximately 1.1x random for the monolithic and composite PS matrices to 17x random in the monolithic and 6x random in the composite WD matrices. No significant differences in matrix residual strains between the composites prepared by the two procedures were noted. The Trimarc (WD) fibers recorded higher (1.3x) compressive strains than the SCS-6 (PS) fibers in all the measured directions. The plane-specific elastic moduli measured in load tests on the unreinforced matrices, showed little difference.

12:05 PM

MODELLING OF TRANSVERSE TENSILE BEHAVIOUR OF Ti MMCS: *W. Ding*¹; J. Liu¹; P. Bowen¹; ¹The University of Birmingham, School of Metallurgy and Materials/IRC in Materials for High Performance Applications, Edgbaston, Birmingham B15 2TT UK

Ti MMCs unidirectionally reinforced by SiC fibres are anisotropic, and are often proposed to sustain load only in the direction of fibre alignment. However, even under such loading conditions, complex stress states may exist in the composite once a crack is introduced. Thus the aim of this paper is to quantify and model the transverse tensile behaviour of Ti MMCs. Both finite element (FE) modelling and analytical modelling based on a modified Eshelby method have been used. Attention is focused not only on the effects of matrix modulus and fibre volume fraction (V_f) but also on the effects of volume fraction of fibres with debonded interfaces (V_{f,B}), fibre pack-

ing distance, and fibre packing architecture (rectangular and hexagonal distributions). The modelling results have also been compared with experimental measurements carried out on Ti MMCs with different Vf and different fibre/matrix combinations and good agreement has been found. This gives the opportunity to design the distribution of SiC fibres so that an optimised combination of longitudinal and transverse properties can be achieved.

PAUL A. BECK MEMORIAL SYMPOSIUM: Paul A. Beck Memorial Symposium - I General, Texture & Heat Capacity

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Program Organizers: Karl A. Gschneidner, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Michael V. Nevitt, Clemson University, Dept of Physics & Astronomy, Clemson, SC 29634 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899

Tuesday AM Room: Rome
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Karl A. Gschneidner, Iowa State University, Ames Laboratory, Ames, Iowa 50011-3020 USA

8:30 AM OPENING REMARKS

8:40 AM INVITED PAPER

PAUL BECK: THE YEARS AT ILLINOIS: *Charles A. Wert¹*; ¹University of Illinois, Materials Science and Engineering, 1304 West Green St., Urbana, IL 61801 USA

Study of recrystallization, grain growth and textures occupied Paul Beck in his "first career". That work was in full bloom when he came to Illinois in 1951 but continued for 20 more years, through some 100 papers. Gradually his attention shifted to electronic and magnetic properties of alloys. He and his students carefully measured the electronic density of states of metallic alloys and the ferromagnetic and paramagnetic properties of solid solutions and compounds. Defining features of this work were careful preparation of alloys, precise measurements and excellent interpretation remarkably aided by close interaction with the condensed-matter-physicists. The work evolved to study of spin glasses and the magnetic character of thin layer alloys. He formally retired in 1976, but continued working with enthusiasm for some 14 more years. For years, he organized the colloquia of the department which became popularly known as the 3d-4s series. He also led TMS into regular inclusion of symposia in alloy theory. His work won for him many awards from both TMS and ASM. He was elected to the National Academy of Engineering and received an Honorary Degree from the University of Illinois, an honor rarely given to a faculty member.

8:55 AM INVITED PAPER

A BRIEF RECAP OF TMS COMMITTEE ON ALLOY PHASES: *Michael V. Nevitt¹*; ¹Clemson University, Dept. of Physics and Astronomy, Clemson, SC 29634 USA

Paul Beck took the initiative with TMS/AIME leadership in the early 1960's to establish a locus of interest in the physics and chemistry of alloy phases, their occurrence, structures and properties. As a result, the Committee on Alloy Phases (CAP) was formed. The cross-cutting interests of CAP have continued to emphasize the theoretical and experimental properties governing the occurrence and stability of solid solutions and intermetallics. A major role of CAP

has been in sponsoring relevant TMS symposia, most notably the annual Hume-Rothery Symposium and Award. A few highlights in CAP's thirty-year history will be reviewed.

9:10 AM INVITED PAPER

RECRYSTALLIZATION TEXTURES IN METALS: *Paul Shewmon¹*; ¹Ohio State University, Materials Science and Engineering, 2477 Lytham Rd., Columbus, OH 43220 USA

Prof. Beck advocated the 'oriented growth' theory of texture and published the results of several classic experiments demonstrating the importance of the rapid growth of certain orientations in texture development. The contesting theory was 'oriented nucleation', put forth strongly by Buerger, among others. Developments in this field since Beck's last publications on the topic 40 years ago will be summarized. It will be concluded that there is still a controversy about the relative importance of oriented growth and oriented nucleation in texture development.

9:45 AM BREAK

10:00 AM INVITED PAPER

ON THE CONTRIBUTION OF ORIENTED NUCLEATION AND GROWTH SELECTION ON THE FORMATION OF RECRYSTALLIZATION TEXTURES: *Olaf Engler¹*; ¹Los Alamos National Laboratory, Center for Materials Science, Mail Stop K765, Los Alamos, NM 87545 USA

For several decades the changes in crystallographic textures during recrystallization have been explained in terms of one out of two rivaling theories, oriented nucleation and growth selection. Whereas in the case of oriented nucleation, it is assumed that the preferred formation of special orientations determines the final recrystallization texture, the theory of growth selection - which goes back to Beck et al. - assumes that, starting from a broad spectrum of nucleus orientations, those with the best growth conditions with respect to the deformed matrix grow fastest and, therefore, dominate the recrystallization texture. The present paper reviews the two theories of oriented nucleation and growth selection in the light of recent experimental investigations - in particular with the help of electron back scattering diffraction to determine the crystallographic orientations of small regions down to sub-micrometer size in an SEM - which have provided new insight into the mechanisms of nucleation and growth of recrystallization. From these results it can be concluded that the recrystallization textures of Al-alloys evolve by a preferred formation of some orientations at characteristic nucleation sites and a subsequent growth selection of distinct orientations out of this spectrum of nucleus orientations.

10:35 AM

TEXTURE EVOLUTION IN RECRYSTALLIZED HAFNIUM AND TITANIUM: *Rong Bai¹*; Clyde L. Briant¹; ¹Brown University, Division of Engineering, 182 Hope Street, Providence, RI 02912 USA

This paper presents a study of the evolution of annealing texture in hafnium and titanium. The texture was measured by the analysis of electron back scattering diffraction patterns in the scanning electron microscope. It was found that in hafnium the texture changed both as a function of annealing temperature and cold work. After low temperature recrystallization, the deformation axis was parallel to [11-20] or [-12-10]. After annealing at high temperatures the axis parallel to the deformation axis as [01-10]. When the samples were rolled and recrystallized a texture was developed in which the rolling plane was parallel to the basal planes of the grains. These results on hafnium will be compared with those on titanium. This work was supported by the NSF MRG No. DMR-9223683 and the NSF-Sponsored MRSEC at Brown University.

11:10 AM INVITED PAPER

LOW TEMPERATURE HEAT CAPACITY IN MATERIALS RESEARCH: *James C. Ho*¹; ¹Wichita State University, Department of Physics and National Institute for Aviation Research, 1845 Fairmount, Wichita, KS 67260 USA

Based on statistical thermodynamics, heat capacity is a most fundamental property of materials. Low temperature calorimetry has played significant roles in the development of condensed matter physics. The results can be used to help characterize and analyze a given material in terms of lattice vibration, electronic density of states, magnetic behavior and phase transitions, if any. Examples of actual applications of low temperature heat capacity to materials research will be given here. Among others, they include the systematics in transition metal alloys, microstructural effect in titanium-base superconductors, magnetic clusters in paramagnetic matrix, magnetic ordering in nano-sized ferrites, and conducting polymers.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VII: Ceramics and Ceramic Matrix Composites

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

Tuesday AM Room: Chicago
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Dr. G. R. Edwards, Colorado School of Mines, Department of Metallurgical Engineering, Golden, CO USA; Dr. Mohan Edirisinghe, Loughborough University, Institute of Polymer Technology and Materials Engineering, Loughborough, Leicestershire LE 11 3TU United Kingdom

8:30 AM INVITED PAPER

SPONTANEOUS COPPER INFILTRATION OF ALUMINA BY VAPOR-STATE OXYGEN ALLOYING: *Daniel A. Javernick*¹; Glen R. Edwards¹; ¹Colorado School of Mines, Department of Metallurgical Engineering, Golden, CO USA

Additions of oxygen to copper have been shown to promote spontaneous infiltration of porous alumina bodies. This processing technique, termed the "carrier oxide" approach, is dependent upon the formation of an immiscible liquid oxide which reacts aggressively with alumina to form CuAlO_2 at the metal-ceramic interface. The relative importance of the interfacial reaction product, CuAlO_2 , and the oxygen activity in the molten copper on the processing of these materials have been investigated by analyzing the initial contact of molten copper droplets of varying oxygen content on CuAlO_2 and Al_2O_3 surfaces. These sessile drop measurements will help determine the optimal vapor-state oxygen concentration required to infiltrate porous alumina performs without depositing brittle copper oxides into the metal matrix.

9:00 AM INVITED PAPER

MULLITE-ZIRCONIA COMPOSITES PREPARED BY PLASMA SPRAYING OF ZIRCON AND ALUMINA MIXTURES: *K. A. Khor*¹; *Y. Li*¹; ¹Nanyang Technological University, School of Mechanical & Production Engineering, Nanyang Avenue 639798 Singapore

Reaction sintering of Zircon and Alumina is an easy and inexpensive route to obtain homogeneous mullite-zirconia ceramics with enhanced mechanical properties. This paper presents the preparation of ZrO_2 reinforced mullite by plasma spraying a mixture of zircon and alumina. The dissociation of zircon into zirconia and silica in a plasma flame is well-known phenomenon. Pre-mixed powders of zircon and alumina are injected into a dc plasma jet. The plasma sprayed particles are collected in distilled water and analyzed. The results indicate that the plasma sprayed powders consist of zirconia, zircon and alumina. It was found that fine grained, even amorphous and chemically homogeneous composite powders can be obtained by ball milling and plasma spraying. Recrystallization of amorphous phases and formation of mullite occurred at about 1000°C in plasma sprayed powders. This value is more than 500°C lower than the formation of mullite in as-milled powders. The effect of power and secondary gas pressure on the spheroidization of zircon and alumina mixtures was investigated. The results showed the both of them were of importance in controlling the morphology and phase composition of the spheroidized powders.

9:30 AM INVITED PAPER

EFFECT OF HEAT TREATMENT ON THERMAL CONDUCTIVITY AND MICROSTRUCTURE OF ALUMINUM NITRIDE: *Y. Baik*¹; *M. Entezaran*¹; *H. Vali*¹; *R. A. L. Drew*¹; ¹McGill University, 3610 University Street, Montreal H3A 2B2 Canada

Aluminum nitride (AlN) ceramics are in high demand for applications involving substrate materials for high speed, high capacity, and complex integrated circuits requiring high thermal conductivity. However, there is a challenge in obtaining high thermal conductivity values for polycrystalline AlN substrates compared to that of single crystal AlN which has a thermal conductivity of 320 W/m K. A study of the post-sintering heat-treatment of AlN in various reducing atmospheres was performed to determine the influence on thermal conductivity. It was found that the composition of the secondary phase and temperature of the heat treatment are the most influential factors controlling the thermal conductivity values. Two different mechanisms for the reducing reactions were proposed. The results indicate that such a heat treatment can improve the thermal conductivity of AlN up to nearly 200 W/mK. This is due further oxygen removal from both the AlN lattice and secondary grain boundary phase, simultaneously.

10:10 AM INVITED PAPER

BORON SUBOXIDE SYNTHESIS, CONSOLIDATION AND SUBSEQUENT METAL INFILTRATION: *Oguz Kayhan*¹; *Osman T. Inal*¹; ¹New Mexico Tech, Materials and Metallurgical Engineering Department, Socorro, NM 87801 USA

The intent of the program was to produce metal (aluminum) infiltrated boron suboxide ceramic drag cutters and drill bits that have improved hardness and fracture toughness values over those of commercially available ceramic Cutters. Synthesis of boron suboxide was performed by reactive sintering of crystalline boron and zinc oxide powders at 1450°C for 12 hours in an argon atmosphere. After sintering, Vickers microhardness testing was performed on randomly selected, synthesized samples and an average hardness value of 37 GPa was obtained. For further characterization, optical microscopy, SEM, and XRD analyses were performed on the powder material synthesized. After characterization, consolidation of the powder was performed. Two different routes of consolidation were carried out, explosive consolidation and hot pressing. Through both routes, satisfactory levels of compaction were obtained. Following consolidation, the compacted bodies were given a high temperature sintering treatment for full densification, at 1800°C in vacuum with no pressure applied. Aluminum infiltration was performed into these sintered bodies, in an argon atmosphere, for 10 hours, in the temperature range 1100-1250°C, again with no pressure applied. During infiltration, optimization of the infiltration temperature was also achieved. After every step (consolidation, densification sintering and infiltration steps), the samples were characterized by XRD, SEM, Vickers microhardness and density tests. Three-point bend tests were

also performed on all samples, in order to determine the subsequent fracture toughness and flexural strength values of boron suboxide after each step. The characterized infiltrated samples were finally tested at Sandia National laboratory for penetration and drag force evaluation. The presentation will detail our efforts.

10:40 AM INVITED PAPER

SOLID FREEFORM FABRICATION OF CERAMICS: *M. J. Edirisinghe*¹; ¹Loughborough University, Institute of Polymer Technology and Materials Engineering, Loughborough, Leicestershire LE11 3TU United Kingdom

Solid freeform fabrication (SFF) of ceramics has generated a great deal of interest as it offers highly automated, rapid desktop forming of small ceramic parts without molds. In SFF, 3-D computer images of parts generated with CAD software is sliced to produce 2D images and then used to control a fabrication unit which manufacturers it layer by layer. SFF methods applicable to ceramics will be briefly reviewed. The presentation will focus on continuous ink-jet printing which is a novel SFF method where a ceramic suspension is pressurized and delivered through a fine nozzle and deposited on a substrate according to a pattern generated by a computer. Research into this technique will be elucidated. The development of ceramic suspension for this process, i.e., materials and properties required will be analyzed. The use of suspensions to print monolayers and multilayers of ceramic will be illustrated with several examples. The future potential of the process to micro-engineer components while tailoring the microstructure will be elucidated.

11:10 AM INVITED PAPER

FABRICATION AND PROPERTIES OF DENSELY SINTERED TITANIUM DIBORIDE CERAMICS: *Jun-ichi Matsushita*¹; ¹Tokai University, 1117 Kitakaname, Hiratsuka 259-1292 Japan

Fabrication and properties of pressureless sintered TiB₂ containing metal and carbon for sintering aids were investigated. The powders were blended in the required ratio and wet-mixed in ethyl alcohol for 24 h using a plastic container and nylon balls. After drying, the mixture was formed with a press under 30MPa, and finally formed under CIP at 300 MPa. Pressureless sintering was conducted at 1673 to 2173K for 1 h in an argon atmosphere. Cr-C and Ni-C play an important role in the densification of TiB₂. Sintered TiB₂ containing about 7 wt% Cr(Ni) and 1 wt% C exhibited a relative density of 99(99)%, bending strength of 400(450) MPa and Vickers hardness of 22(17) GPa. The microstructure of TiB₂ sintered containing Cr(Ni) and C was found to retard the grain growth and the fracture surface of sintered body showed both transgranular and intergranular fracture modes. XRD analysis detected CrB(Ni₃B) and TiC phases besides in addition to TiB₂ in the sintered body.

11:40 AM

CERAMIC-INTERMETALLIC COMPOSITES PRODUCED BY MECHANICAL ALLOYING AND SPARK PLASMA SINTERING: *J. G. Cabanas-Moreno*¹; *G. Garcia-Pacheco*¹; *O. Delgado-Gutierrez*¹; *R. Marrtinez-Sanchez*¹; *M. Umemoto*²; ¹Instituto Politecnico Nacional, ESIQIE, Apdo. Postal 75-373, Mexico D.F. 07300 Mexico; ²Toyohashi University of Technology, Tempakucho, Toyohashi 441 Japan

Nano- and micro-composites of intermetallic (Co₃Ti, AlCo₂Ti) and ceramic (Ti(C₁N), Al₂O₃) phases have been produced by spark plasma sintering (SPS) of powders resulting from the mechanical alloying of Al-Co-Ti elemental powder mixtures under argon/air atmospheres. The mechanically alloyed powders consisted of mixtures of nanocrystalline and amorphous phases which, during sintering, transformed into complex dispersions of the above mentioned phases. For Al contents lower than about 30 at % in the original powder mixtures, the use of SPS to densify the alloyed powders resulted in compacts of low porosity (1 - 2 %) and nano- or microcrystalline structures, showing microhardness values as high as ~1700kg/mm²; in these cases, the matrix of the composites was mainly formed by the Co₃Ti intermetallic phase, although the largest volume fraction cor-

responded to the Ti(C₁N) phase. The AlCo₂Ti and Al₂O₃ phases were both present in small volume fractions. Details of the processing and microstructural characterization of sintered compacts, as well as the results of attempts to characterize their fracture behavior, will be also presented. Work supported by CONACYT and IPN in Mexico and through an AIEJ scholarship in Japan.

12:00 PM INVITED PAPER

A NEW UNIDIRECTIONALLY SOLIDIFIED CERAMIC EUTECTIC WITH HIGH STRENGTH AT HIGH TEMPERATURES: *Yoshiharu Waku*¹; ¹UBE Industries, Ltd., Ube Research Laboratory, 1978-5 Kogushi, Ube City, Yamaguchi Prefecture Japan

New unidirectionally solidified eutectic composites such as Al₂O₃/Er₃Al₅O₁₂ or GdAlO₃ has recently been fabricated by controlling accurately the unidirectional solidification. The eutectic composite has a new microstructure, in which continuous networks of single-crystal Al₂O₃ and single crystal oxide compounds (Er₃Al₅O₁₂ or GdAlO₃) interpenetrate without grain boundaries. The eutectic composite fabricated is thermally stable and has the following properties: 1) the flexural strength at room temperature can be maintained up to just below the melting point of about 2040-2150 K, 2) the compression creep strength at 1873°K and a strain rate of 10⁻⁴/sec is about 7 times higher than that of sintered composites of the same composition (in case of Al₂O₃/Er₃Al₅O₁₂), 3) it shows neither weight gain or grain growth even upto heating at 1973 K in air atmosphere for 500 hours (case of Al₂O₃/Er₃Al₅O₁₂), 4) it shows substantial plastic deformation at 1873°K with a flexural yield stress of about 690 MPa and it is found that the plastic deformation occurred by dislocation motion in each phase (in case of Al₂O₃/GdAlO₃).

12:30 PM

CONTROLLED COMBUSTION SYNTHESIS IN THE TiH₂-B SYSTEM FOR PRODUCTION OF TiB-TiB₂-Ti CMC'S: *Sedat Ozbilen*¹; ¹Gazi University, Metallurgy Education Department, Education Department, Teknik Okullar, Ankara Turkey

Controlled Combustion Synthesis (CS) was carried out in the TiH₂-B system. Samples with different TiH₂ levels (sample #1: 82wt%TiH₂-18wt%B, sample #2:41wt%Ti-18wt%B, sample #3: 82wt%Ti-18wt%B) were studied to investigate the influence of the surface condition of the diluents (Ti in this study) on the level of exothermicity of the self-propagating reactions. Homogeneously mixed and then compressed pellets of samples #1-#3 (green compacts) were combustion synthesized in controlled fashion and in thermo explosion mode under vacuum. Thermal analysis under vacuum by DTA was carried out on the green compacts. SRD and SEM investigation were used for the examination of pressed and as-reacted pellets. It was observed that the exothermicity of the CS reactions can be increased when the amount of the activated, fresh surfaces of diluent Ti created by TiH₂ decomposition during heating of the samples under vacuum is increased as in sample #1 [having the highest TiH₂ content (82wt%) among the samples studied] thus promoting better kinetics conditions (i.e., faster CS reactions) for chemical reactions.

12:50 PM

MODIFIED OXALATE METHOD FOR THE SYNTHESIS OF PURE BARIUM TITANATE CERAMICS: *R. Ramanathan*¹; *V. Kamaraj*¹; ¹Anna University, Division of Ceramic Technology, Department of Chemical Engineering, Chennai 600 025 India

Barium titanate is an important electroceramic material with wide industrial applications such as capacitors and positive thermal coefficient thermistors. Since its discovery in 1943, processing of barium titanate has been an active field of research. In this paper a simple modified oxalate method is adopted for the preparation of barium titanate. Barium chloride dihydrate and titanium tetrachloride are used as starting materials. The solutions of the starting materials are mixed with the oxalic acid solution. After continuous stirring the solution is allowed to precipitate. The precipitate is filtered and then dried at 70°C. The dried powders are calcined at 1000°C and sintered

at 1300°C. The powder characterization studies like TGA, DTA, and SRD are done.

PROCESSING & PROPERTIES OF ADVANCED STRUCTURAL CERAMICS: Advanced Structural Ceramics: Processing

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Rajiv S. Mishra, University of California, Dept of Chemical Engineering & Materials Science; Amiya K. Mukherjee, University of California, Dept of Chemical Engineering & Materials Science

Tuesday AM Room: London
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: T. E. Mitchell, Los Alamos National Laboratory, Center for Materials Science, Los Alamos, NM 87545 USA; Rajiv Mishra, University of California, Department of Chemical Engineering and Materials Science, Davis, CA 95616 USA

8:30 AM OPENING REMARKS

8:35 AM KEYNOTE

A TECHNICAL COST FRAMEWORK FOR HIGH TEMPERATURE MANUFACTURING OF SMALL COMPONENTS AND DEVICES: *A. G. Evans*¹; *J. W. Hutchinson*¹; *T. J. Lu*²; ¹Harvard University, Applied Sciences Department, Cambridge, MA 02138 USA; ²University of Cambridge, Cambridge CB2 1PZ UK

One goal of manufacturing process development is the discovery of a method for processing a material into components or devices that attain explicit property and performance specifications at minimum cost. These specifications are usually established by the system level design strategy. Establishing a methodology that links these aspects in a systematic manner is elusive. One approach that provides focus and may lead to a tractable methodology embraces a technical cost framework (TCF). This framework interrelates parameters from design and manufacturing, through a structure that accepts output from both process simulations and from design calculations. It is simple and direct. The challenge is to establish a modeling environment that either calculates or measures the input parameters with acceptable certainty. Cost simulations are used to illustrate the approach and the potential for its further development. When a high temperature step is involved, it often dominates the non-material contributions to the cost. Various methods commonly used for heat treatment are examined, in order to facilitate a logical decision about the preferred approach in any new manufacturing initiative. The philosophy evolves around the expectation that the market for the products is substantial enough to allow the furnace to operate at maximum capacity, with allowance for down-time upon maintenance and repair. Moreover, the mix of product geometries and the properties are considered to be sufficiently similar that property development occurs in essentially the same way for all products, such that yields can be controlled and characterized.

9:15 AM INVITED PAPER

DUCTILE MACHINABLE TERNARY CARBIDES AND NITRIDES FOR HIGH TEMPERATURE APPLICATIONS: *M. W. Barsoum*¹; ¹Drexel University, Department of Materials Engineering, Philadelphia, PA 19104 USA

Recently we identified two classes of layered hexagonal ternary carbides and nitrides; the M_3BX_2 and M_2BX phases, where M is a transition metal, B is a B-group element and X is either C or N. Three interrelated characteristics distinguish these phases from other layered materials: i) the metallic nature of the bonding; ii) the presence of an operative slip system at room temperature and its confinement to the basal planes; and, iii) absence of "strong" primary bonds between layers. This results in compounds that are machinable, relatively soft ($H_v \sim 3-6$ GPa), and good thermal and electrical conductors ($2-15 \times 10^6 \Omega^{-1}m^{-1}$). They also possess excellent ambient and high temperature mechanical properties that are decoupled from machinability, room temperature plasticity in oriented samples, excellent damage tolerance, thermal shock, oxidation and creep resistance, among others. The technological implications of having these naturally layered materials will be discussed. Work funded by DMR division of NSF and AFOSR.

9:45 AM

JOINING OF STRUCTURAL CERAMICS USING THIN SPIN-ON LAYERS VIA BOTH MICROWAVE AND CONVENTIONAL HEATING: *E. D. Case*¹; *K. Y. Lee*¹; *J. G. Lee*¹; ¹Michigan State University, Materials Science and Mechanics Department, East Lansing, MI 48824 USA

Structural ceramics having complex geometries can be difficult to process and difficult to check for processing-induced flaws. However, both the processing and quality control of such components could potentially benefit from an efficient and reliable methodology to build up components of complex shape by joining subcomponents of simpler geometry. Using spin-on layers of 150 nm to 600 nm thick, the authors have joined alumina and alumina/zirconia composites specimens using zero or small (20 - 60 gram) dead-weight loads. (This is in contrast to other relatively high loads used in joining structural ceramics, especially in terms of the microwave joining work). This paper will discuss joining work on alumina, alumina/zirconia composites, and partially stabilized zirconia specimens. Microstructural and mechanical characterization of the join integrity will also be discussed.

10:10 AM

FUNCTIONAL CERAMIC-REFRACTORY METAL LAYERED COMPOSITES BY PLASMA SPRAY FORMING: *Daniel J. Sorelet*¹; *M. F. Besser*¹; *R. L. Terpstra*¹; *I. E. Anderson*¹; ¹Ceramist, Ames Laboratory, 107 Metals Development, Ames, IA 50011 USA

Melt processing of extremely reactive, high temperature alloys, e.g., Ti-based, has created the need for advanced refractory materials. Processes such as gas atomization require melting components, e.g., tubes and crucibles, to resist attack from superheated alloys much better than conventional graphite, zirconia, or alumina. We have found that rare earth oxides resist attack by molten Ti-based alloys. However, conventional sintered microstructures of these ceramics are too sensitive to thermal shock during the transient conditions of gas atomization. Tougher, more thermal shock resistant rare earth oxide components may be fabricated by thermal spraying. The lamellar microstructure and finely distributed porosity developed during thermal spraying appears to mitigate the cracking that is typical of dense, fine grained rare earth oxide. In addition, the incorporation of refractory metal sublayers can further enhance the toughness of the composite system. Developments on spray formed melting components for atomization will be reviewed. Department of Energy-BES support (W-7405-Eng-82) is acknowledged.

10:35 AM BREAK

10:45 AM INVITED PAPER

NOVEL PROCESSING ROUTES FOR ADVANCED FUNCTIONAL CERAMICS: *S. C. Danforth*¹; *A. Safari*¹; ¹Rutgers, The State University, Department of Ceramic and Materials Engineering, Center for Ceramic Research, Piscataway, NJ 08855-0909 USA

Advanced structural ceramics require a high degree of processing perfection in order to insure functional properties such as high fracture strength, high fracture toughness and a high Weibull modulus. It is still very costly and time consuming to develop advanced structural ceramic components for new applications, due to the costs and time associated with each design iteration. To solve this difficulty, solid freeform fabrication (SFF) processes are now under development for processing ceramics such as silicon nitride, Si_3N_4 . Solid freeform fabrication processes offer significant potential for manufacturing functional quality components, free from the constraints imposed by the external geometric complexity of the part. This paper gives a detailed description of the Fused Deposition of Ceramics (FDC) process, a SFF technique developed at Rutgers University, specifically for structural ceramic applications. In the FDC process, a high solids loading of ceramic powders are mixed with a multi-component thermoplastic binder and subsequently extruded into 1.778 mm filaments, which are then driven, via computer controlled counter rotating rollers, into a hot extruder and deposited onto a z-motion platform. The extruder moves in the x-y plane, depositing material only in locations specified by the original CAD file. This paper will detail the processing and quality control steps needed to fabricate high quality FDC filament feedstock using 55 vol. % of AlliedSignal's GS-44 insitu-reinforced (ISR) Si_3N_4 . The major process steps include ball milling, surfactant coating, compounding with the binder, filament extrusion, part building by FDC, thermal binder removal and finally sintering. Issues critical to manufacturing of advanced structural ceramics by the FDC process which have high strength and high Weibull modulus will be detailed. The authors wish to acknowledge sponsorship of this research under DARPA ONR Contract Number N00014-94-C-0115.

11:15 AM

SELECTIVE AREA LASER DEPOSITION VAPOR INFILTRATION, SALDVI, OF POWDERS AS A SOLID FREEFORM FABRICATION, SFF, PROCESS: James E. Crocker¹; Shay Harrison¹; L. Sun¹; H. L. Marcus¹; L. Shaw¹; ¹Institute of Materials Science, University of Connecticut, Storrs, CT 06269-3136 USA

SALDVI is a SFF approach that uses thermal or photo decomposition of precursor gases to infiltrate layer by layer of powders to create a shape. The process will be defined in its application to ceramics and results of processing variables on the microstructures will be described. The range of possible materials and combinations of materials will be presented with the majority of the effort reported on SiC and Si_3N_4 and their composites.

11:40 AM

AMORPHOUS FIBERS BY MELT EXTRACTION: *Ena A. Aguilar*¹; Robin A.L. Drew¹; Claudia Milz²; Bilge Saruhan-Brings²; Bernd Hildmann²; ¹Department of Mining and Metallurgical Engineering, McGill University, 3610 University St., Montreal, Quebec H3A 2B2 Canada; ²DLR, Deutsche Forschungsanstalt für Luft- und Raumfahrt e.V., Porz-Wahnheide Linder Höhe, D-51147 Köln Germany

Alumina-yttria fibers were fabricated using the melt extraction technique. These fibers were completely amorphous in their as-extracted state. Two types of fibers are generally obtained depending on the extraction speed. At very low speed (<3 m/s), fine and uniform fibers of high quality are produced. However, when the wheel speed exceeds a critical velocity, Rayleigh waves are formed on the free surface of the fiber due to instability and the effects of surface tension. The average tensile strength of the extracted fibers was 1 GPa for thin, uniform diameter fibers. Differential Scanning Calorimetry (DSC) on the as-extracted fiber gave one exothermic peak for YAG and YAG/YAP eutectic compositions, and three exothermic peaks for the $\text{Al}_2\text{O}_3/\text{YAG}$ eutectic composition. In order to characterize each phase transformation, x-ray diffraction (XRD) analysis was performed on the $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3$ fibers heat-treated to each phase transformation completion temperature. For YAG and YAG/YAP eutectic compositions, the exotherm corresponds to the crystallization of amorphous phase into YAG. However, for $\text{Al}_2\text{O}_3/\text{YAG}$ eutectic composition, first peak corresponds to the crystallization of the amorphous phase into YAG, the second peak is due to the crystallization of

the remaining amorphous phase to $\delta\text{-Al}_2\text{O}_3$, finally, the third peak corresponds to the polymorphic phase transformation from $\delta\text{-Al}_2\text{O}_3$ to $\alpha\text{-Al}_2\text{O}_3$.

12:05 PM

CONTROLLED COMBUSTION SYNTHESIS IN THE $\text{TiH}_2\text{-B}$ SYSTEM: *S. Ozbilen*¹; ¹Gazi University, Faculty of Technical Education, Metal Education Dept., Teknikokullar, Ankara Turkey

Controlled combustion synthesis was carried out in the $\text{TiH}_2\text{-B}$ system. Compositions of different TiH_2 levels were studied to investigate the effect of diluents on the level of exothermicity of the self-propagating reactions. Compressed pellets of $\text{TiH}_2\text{-B}$ mixtures were degassed and ignited in a reaction chamber. Thermal analysis by DTA was carried out on the samples. XRD and SEM investigation were used for the examination of as-reacted pellets of TiB-Ti ceramic matrix composites. Resulting microstructures and properties are discussed.

TEXTURE IN FILMS AND COATINGS: Session III

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

Tuesday AM

October 13, 1998

Room: Zurich/Tokyo

Location: O'Hare Hilton Hotel

Session Chair: J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

8:30 AM INVITED PAPER

THE DEVELOPMENT OF CRYSTALLOGRAPHIC TEXTURE IN FILMS USED IN MAGNETIC RECORDING: *David E. Laughlin*¹; Yu-Nu Hsu¹; Bin Lu¹; ¹Carnegie Mellon University, Department of Materials Science and Engineering, 3307 Weon Hall, Pittsburgh, PA 15213-3890 USA

Thin magnetic films used for magnetic recording are sputter-deposited on another film usually called the underlayer. The underlayer has important functions which include the control of the magnetic layer grain size, the control of the magnetic layer crystallographic texture and the production of a solute enriched grain boundary in the magnetic film. In this talk we will review these important functions of the underlayer and discuss the way that current state of the art magnetic recording films have evolved over the past two decades. Our emphasis will be on the microstructural development of the films and how Materials Science principles have been applied to aid in the development of today's Co based alloys used in high density thin film storage media. This work has been supported over the years by the Data Storage Systems Center of CMU through NSF ECD 89-07068 and the DOE (DE-FG02-90ER45423). B. L. has been supported by a grant from INTEVAC.

9:05 AM

CHARACTERIZATION OF CRYSTALLOGRAPHIC TEXTURE IN Co-BASED MAGNETIC MEDIA : *Gaurav Khanna*¹; James Freitag¹; Bruce M. Clemens¹; ¹Stanford University, Materials Science and Engineering, 416 Escondido Mall, Bldg. 550, Stanford, CA 94035 USA

In polycrystalline Co-based magnetic media, the magnetic and recording properties are strongly influenced by the orientation tendencies of the c-axis of the hcp media grains. For example, many manufacturers use a (100)-oriented Cr underlayer to induce a nominal (11-20) fiber texture in the media layer. This places the easy c-axis in

the plane of the disk, favoring in-plane magnetization. For smooth disks, the in-plane orientation of the c-axis is random and the magnetic properties are also isotropic in the plane. However on grooved disks, the magnetic properties are anisotropic, and the orientation ratio (the ratio of circumferential to radial coercivity) can be greater than one, leading to improved recording performance. Various possible explanations for this effect have been presented, including anisotropic in-plane distribution of Co c-axis and anisotropic strain. The structural origins of the orientation ratio were explored using synchrotron-based x-ray diffraction and atomic force microscopy. Asymmetric diffraction geometry was used to avoid the differential absorption and shadowing which plagues grazing incidence scattering experiments on grooved samples. X-ray wavelengths which are strongly absorbed by the NiP underlayer were used to eliminate scattering from the Al substrate. By comparing the surface topography, as measured by AFM, to the texture x-ray rocking curves parallel and perpendicular to the grooves, it was observed that both the Cr underlayer and the Co media layer exhibit conformal texture with the Cr (100) and Co (1120) planes following the local surface normal. This produces a fanning effect which reduces the intensity of diffraction features along a direction perpendicular to the grooves. However, after accounting for this fanning effect, variations in the two-fold Co diffraction peak intensities parallel and perpendicular to the grooves seem to offer strong evidence of an anisotropic in-plane distribution of the Co c-axes, with a preference for the c-axes to lie along the groove direction. Current experiments with epitaxial Cr/Co-alloy films grown on stepped MgO (001) substrates will give quantitative values for the contribution of anisotropic strain and c-axis orientation on the magnetic orientation ratio.

9:25 AM

CRYSTALLOGRAPHIC TEXTURE OF THIN FILM MATERIALS EXHIBITING GIANT MAGNETORESISTANCE: *L. P.M. Brandao*¹; Xin Dong¹; H. Garmestani¹; K. H. Dahmen²; ¹Florida State University, Department of Mechanical Engineering and MARTECH, Tallahassee, FL, USA 32310-2175, and Departamento de Engenharia Mecânica e de Materiais, IME, Rio de Janeiro, Brazil; ²Florida State University, Department of Chemistry and MARTECH, Tallahassee, FL 32306-3006 USA

Thin films of $\text{La}_{1-x}\text{M}_x\text{MnO}_3$ (M=Ca, Sr) with different thickness have been produced on (100) oriented LaAlO_3 , yttrium stabilized zirconia (YSZ) and Si substrates by liquid delivery metal organic chemical vapor deposition. The effects of the substrate type and film thickness on the crystallographic texture of these films were investigated using x-ray technique. XRD analyses showed that for thickness of ≤ 500 Å, the films were monocrystalline and epitaxially grown on LaAlO_3 substrates and polycrystalline on YSZ. At thickness ≥ 500 Å, all the films were polycrystalline and highly textured. The magnetoresistance resistance of the films on YSZ was the highest and it was explained in terms of their texture.

9:45 AM

GROWTH PROCESSES, TEXTURE AND INTERGRANULAR MAGNETIC INTERACTION IN HARD MAGNETIC Co-W AND Co-P BASED FILMS: *Vladimir Grigor'evich Shadrow*¹; Anatoly Vasil'evich Boltushkin¹; Lyudmila Vasil'evna Nemtsevich¹; ¹Inst.Solid State Physics, Acad.Sci.of Belarus, P.Brovki,17, Minsk 220072 Belarus

Texture, microstructure and magnetic properties of hard magnetic electrodeposited Co-W, Co-P based films have been investigated by means of EM, XRD, AFM and AGFM. It is shown that the character of the microstructure formation and that of the texture growth are interconnected and accounted for by electrode reactions products adsorption at the cathode. A comparison is made with structure formation peculiarities in hard magnetic Co-P based films crystallized from the initial amorphous state and with those in Co deposited alumite films. The texture and microstructure influence on magnetic properties, in particular, intergranular magnetic interaction, evaluated from the remanence and delta M curves, is discussed as well as the irreversible susceptibility curves shape dependence on magnetic (and structural) films inhomogeneity.

10:05 AM

MICROSTRUCTURES OF PLASMA-SPRAYED AISI 304 STAINLESS STEEL COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim²; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 730-701 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Textures and microstructures of Plasma-sprayed AISI 304 stainless steel coatings were studied by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. Parameters of plasma-spraying for AISI 304 stainless steel are gun power, arraying distance, surface speed, voltage and powder feed rate. The Plasma-sprayed layer shows a lamella structure and is built up particle by particle. Diffraction pattern of plasma-sprayed layer is changed by plasma-spraying parameter Pole figure of plasma-sprayed layer is similar to AISI 304 stainless steel polycrystalline and is affected by plasma-spraying parameter.

10:25 AM BREAK

10:35 AM

A STUDY ON THE TEXTURE OF PLASMA-SPRAYED AISI 316 STAINLESS STEEL COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim¹; Young Woo Rheem¹; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 730-701 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Surface morphology and texture of the plasma-sprayed AISI 316 stainless steel coatings were investigated by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. The corrosion and erosion resistance of the various coatings are investigated by using potentiostat. The corrosion and erosion resistance of the coatings are greatly affected by the gun power. Pole figure of coating layer is similar to MSI 316 stainless steel polycrystalline, and is changed by plasma-spraying parameter. The thickness of the splats and disturbance of the unmelted particles were increased with a decreasing the gun power.

10:55 AM

SURFACE MORPHOLOGY AND TEXTURE OF PLASMA-SPRAYED Ni COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim²; Sung Jun Hong²; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 305-764 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Surface morphology and texture of the plasma-sprayed AISI 316 stainless steel coatings were investigated by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. The corrosion and erosion behaviour of the various coatings are studied by using potentiostat. Diffraction pattern of plasma-spraying coatings layer is changed by plasma-spraying parameter. Texture of coating layer is similar to Nickel polycrystalline, and is affected by plasma-spraying parameter.

11:15 AM

TEXTURES AND MICROSTRUCTURES OF AS PLASMA-SPRAYED AND RECRYSTALLIZED ALUMINIUM AND COPPER COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim²; Sung Jun Hong²; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 305-764 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Texture and microstructures of as plasma-sprayed and recrystallized Al and Cu coatings were studied by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. The corrosion and erosion behavior of the various types of coatings layer are investigated by using potentiostat. Texture of a plasma-sprayed and

recrystallized Al and Cu coatings are similar to each polycrystalline, and is affected by plasma-spraying parameter. Plasma-sprayed coatings layer shows the dendrite morphology.

11:35 AM

TEXTURE MEASUREMENT OF PLASMA-SPRAYED NiCr AND NiCrAl ALLOY COATINGS: *Insoo Kim*¹; Dong Soo Suhr²; Byoung Hee Kim²; Sung Jun Hong²; ¹Kumoh National University of Technology, School of Materials and Metallurgical Engineering, Kumi, Kyung Buk 730-701 Korea; ²Chung-Nam National University, Department of Materials Science and Engineering, Taejon 305-764 Korea

Texture and surface morphology of plasma-sprayed NiCr and NiCrAl coatings were investigated by using x-ray diffractometer, pole figure goniometer, SEM and optical microscopy. The corrosion and erosion behavior of the various types of coatings layer are investigated by using potentiostat. The diffraction pattern and texture of as plasma-sprayed NiCr and NiCrAl alloy coatings is changed by plasma-spraying parameters. Also, texture change and corrosion and erosion behavior of plasma-spray coatings layer are affected by plasma-spraying parameters.

Technical Program

TUESDAY PM

DISCONTINUOUSLY REINFORCED ALUMINUM-COMPOSITES: PRESENT & FUTURE: Synthesis of Discontinuously Reinforced Aluminum

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

Tuesday PM Room: Dublin
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Daniel B. Miracle, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433

2:00 PM INVITED PAPER

CAST DISCONTINUOUSLY REINFORCED ALUMINUM FOR OPTICAL MECHANICAL ASSEMBLIES: *Joseph M. Kunze*¹; ¹Triton Systems, Inc., 200 Turnpike Road, Chelmsford, MA 01824 USA

High volume fraction (55-75%) discontinuously reinforced aluminum (DRA) produced by pressure infiltration casting techniques have been accepted by the electronic packaging industry due to their attractive combination of improved heat dissipation and tailored coefficient of thermal expansion. With their greatly increased stiffness, these DRA's also offer a lightweight alternative for use in optical mechanical assemblies (OMA's). In the past, several barriers have existed to their acceptance in this area including prototype delivery time, unit cost, and ability to process thin sections. Under SBIR funding from the Army, Triton Systems is prototyping an OMA component to demonstrate the potential of Triton's Enhanced Pressure Infiltration Casting technology in fabricating DRA OMA's. The benefits of casting a DRA OMA with this technology include increased performance, lower cost and reduced part count obtained by integrating several parts of a complex, lightweight OMA into a single casting. This paper reviews the development of the DRA component and materials.

2:30 PM

TiB₂ - REINFORCED ALUMINUM COMPOSITES: *J. D. Ellis*¹; ¹London & Scandinavian Metallurgical Co. (LSM), Fullerton Rd., Fotherham, South Yorkshire S60 1DL England

For the past 3 years LSM has been developing TiB₂ reinforced aluminum composites in a European Community funded project. This collaborative project involved 10 other organizations from Europe comprising users of aluminum composites, universities and parts fabricators. At the outset of the project a maximum of 8 wt % TiB₂ could be incorporated into the aluminum. Now over 20 wt % can be incorporated. Strength and modulus values have increased in line with this to the level at which they are comparable with SiC and Al₂O₃ reinforced aluminum. The TiB₂ is inert in aluminum and being 1 mm in size means that the MMC is easily machined and mechanically formed. Initial applications for the Al-TiB₂ MMC are for wear resistant applications requiring mechanical processing in their manufacture and sales of tens of tonnes a year are expected by 1999. The presentation will give a detailed account of the scientific, engineering

and marketing effort that LSM has undertaken, presenting an optimistic view of the future.

3:00 PM

PROCESSING AND EVALUATION OF SiC-REINFORCED ALUMINUM ALLOY COMPOSITES : *A. B. Pandley*¹; R. Wheeler¹; B. S. Majumdar¹; D. B. Miracle²; ¹UES, Inc., 4401 Dayton-Xenia Road, Dayton, OH 45432 USA; ²Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLM, Wright Patterson AFB, OH 45432 USA

Discontinuously reinforced aluminum (DRA) composites usually possess superior specific stiffness and strength, wear resistance, creep resistance, and thermal resistance as compared to the unreinforced aluminum alloys. Powder metallurgy processing offers better distribution of SiC particles than the conventional casting technique. Vacuum degassing of powders constitute an important step in the processing of these materials. Although higher degassing temperature may be preferred to avoid blistering during post-processing heat treatment, it may be detrimental from an oxidation point of view. In this study, the effect of degassing temperature/time on the microstructure and tensile properties of DRAs will be evaluated. The materials based on 2009/15 vol.% SiC and 7091/15 vol.% SiC will be considered for this purpose. The role of oxides on the strength and ductility of these composites will also be discussed.

3:30 PM

IN-SITU FORMATION OF SILICON CARBIDE IN ALUMINUM ALLOYS: *R. G. Reddy*¹; D. M. Kocherginsky¹; ¹The University of Alabama, Department of Metallurgical and Materials Engineering, Tuscaloosa, AL 35487 USA

Discontinuously reinforced aluminum composites are rapidly emerging as new commercial materials for aerospace and automotive industrial applications. In this study, the effect of temperature and alloy composition on the equilibrium species distribution in the liquid Al-Si/SiC composite was calculated using Gibbs energy minimization method. The alloy (Al-30%Si) and two types of carbon sources were used for in-situ formation of a silicon carbide. The experiments were carried out for 1 to 20 hours at 1273 to 1573K using alumina crucibles. The experimental results showed that when natural gas was used as a carbon source the efficiency of SiC formation was very low and also a small amount of SiC formed was floated to the surface. When the activated carbon particles were used as a carbon source most of the carbon completely reacted and formed a silicon carbide. The SiC was formed by movement of the reaction zone from the surface to the center of the carbon particles. The silicon carbide formed in the melt retained the size and the shape of initial carbon.

4:00 PM

PROCESSING AND CHARACTERIZATION OF 6061Al-TiN PARTICULATE REINFORCED METAL MATRIX COMPOSITES PRODUCED BY MELT STIR CASTING: *J. Vijay Kumar*¹; ¹Indian Institute of Science, Department of Metallurgy, Bangalore 560 012

The present paper describes the processing and characterization of a novel metal matrix composite system, 6061 Al-10TiN. There are no significant reports existing in literature on this system. The composite has been prepared using the meltstir casting route and subsequently cast billet is extruded. The present study aims at mechanical property evaluation, age hardening behaviour, sliding wear behaviour and thermal characterization. Three types of composites with varying volume fraction of reinforcement have been prepared, viz: 6061Al-5vol%TiN(78micron), 6061Al-10vol%(78micron) and 6061Al-10vol%(38micron). The unreinforced alloy also has been

processed under identical conditions to compare the results with the composites. The initial microstructural characterization has showed some interesting results. In the 38micron TiN containing composite, the microstructure reveals the extensive formation of a second phase around the TiN particles. However it is not the case with the 78micron particulate reinforced composite. Detailed SEM and XRD results indicate that the formation of the second phase is due to the conversion of the TiN particles into TiO₂ and other off stoichiometric nitrides. We are in the process of correlating the microstructure with the mechanical properties. The composites are also being tested for their room temperature and high temperature tensile and compressive strengths. We are also studying the wear behaviour of the composite under dry sliding condition. The results of all these will be presented.

4:30 PM

LOW COST ALUMINUM DROSS REINFORCED METAL MATRIX COMPOSITES: *Kevorkijan M. Varuzan*¹; ¹Head Of The Project Consortium, MMCs, Lackova 139, Limbus, Slovenia 2341

In the casting-house, aluminum dross represents a permanent source of waste generation, especially on the end of secondary dross processing, when a fine, low metallic dust is generated by tumbling. In the present work, this finely powdered by-product was employed as a low cost reinforcement in discontinuously reinforced aluminum matrix composites. By using the fine (less than 50mm) fraction of non-metallic particles generated during aluminum dross treatment and the part of in-house recovered aluminum alloy (A 380), low cost aluminum dross metal matrix composites (AIDMMCs) were routinely produced by the conventional squeeze casting and pressure infiltration techniques. The tensile strength and hardness properties of up to 20 vol. % fine dross particles in AIDMMC were examined. Some preliminary dry sliding wear testing was also carried out using a pin-on-ring wear testing machine. The initial results suggest that the fine fraction of aluminum dross particles is promising filler for wear resistant MMCs.

DYNAMIC BEHAVIOR OF MATERIALS: Dynamic Behavior II

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 Mats Sci & Engrg, Atlanta, GA 30332-0245; Ken S. Vecchio, University of California, Dept of Ames, San Diego, CA 92093 USA

Tuesday PM Room: Sydney
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Kenneth S. Vecchio, University of CA, Dept. of AMES, La Jolla, CA 92093-0411 USA; Marc A. Meyers, University of CA, Dept. of AMES, La Jolla, CA 92093-0411 USA

2:00 PM

PLASMONS AT SHOCK FRONTS: *John J. Gilman*¹; ¹University of California at Los Angeles, Materials Science and Engineering, 6532 Boelter Hall, UCLA, Los Angeles, CA 90095-1595 USA

In dense solids, plasmons are collective analogs of molecular polarization oscillations. Thus they occur in all solids. The oscillation frequency, ω_p , is given by: $(4\pi nq_e/m)$ where n = electron density,

q_e = electron's charge, and m = its mass. Experimental frequencies (from EELS) equal: (cB) where B = bulk modulus, and c is a constant given by simple theory. Thus EELS can be used to determine local elastic stiffnesses. In addition, for the simple metals, $\omega_p = \mu S_{4/5}$ where S is the specific surface energy and μ is a constant. In semiconductors, the plasmon energy equals about 10X the band gap. Plasmas are transparent to optical wavelengths shorter than λ_p (1550 Å for Li, e.g.), so plasmon diagnosis could be useful for studying in situ shock propagation. Since the damping in the electron gas is very small, and a shock front is an intense collective excitation, it can readily cause resonant plasmon excitation.

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FLOW STRESS OF COMMERCIAL PURE TITANIUM OVER A BROAD RANGE OF TEMPERATURE AND STRAIN RATE: *Sia Nemat-Nasser*¹; W. G. Guo¹; J. B. Isaacs¹; ¹Center of Excellence for Advanced Materials, Department of Applied Mechanics and Engineering Sciences, University of California, San Diego, La Jolla, CA 92093-0416 USA

The flow stress of a commercially pure titanium (CP-Ti) is evaluated experimentally over the temperature range of 77K to 1000K and the strain-rate range of 0.001/s to 8,000/s. Experiments show that the material exhibits a three-stage hardening behavior in the temperature range of 400K to 800K, depending on the strain rate. Interrupted tests are performed to examine the resulting microstructural features by optical microscopy. Deformation twinning is seen to play an important role in the plastic response of CP-Ti. The density of the deformation twins increase with decreasing temperature and increasing strain rate and strain. Although the deformation mechanisms in CP-Ti include slip by dislocation movements and deformation twinning, the dislocation is the main mode of plastic flow for CP-Ti. To develop a reasonable constitutive model for the flow stress of CP-Ti, the effect of deformation twinning must be included

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DYNAMIC RESPONSE OF XD Ti-46Al: *William Frederick Cost*¹; Mustafa Guden²; I.W. Rick Hall²; Ernest S.C. Chin¹; Justin Combs²; ¹U.S. Army Research Laboratory, Weapons Materials Division, ATTN: AMSRL-WM-ME, Aberdeen Proving Ground, MD 21005 USA; ²University of Delaware, Materials Science Department, Spencer Laboratory, Newark, DE 19716 USA

An earlier ballistic study of 5%, 10%, and 15% TiB₂ reinforced XD TiAl composites showed promising dynamic behavior due to high strain-rate sensitivity. Subsequent investigation was initiated to characterize the quasi-static and high strain-rate deformation of 1 vol.% TiB₂ XD Ti-46Al in a variety of heat treated conditions to better understand the relationship between microstructure and rate-dependent phenomena. Quasi-static and Split Hopkinson Pressure Bar compression experiments were performed to characterize and compare the stress-strain response of this material. Subsequent microscopic analyses were performed to identify the dynamic flow mechanisms exhibited in this material. Details of this study will be presented along with comparisons to earlier high strain-rate results from higher reinforcement loaded XD materials tested at ambient and elevated temperatures.

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TESTING WITH THE TORSIONAL SPLIT HOPKINSON BAR AT STRAIN RATES ABOVE 10,000 1/S: *Amos Gilat*¹; Ching-Shan Cheng¹; ¹Ohio State University, Applied Mechanics, 155 W. Woodruff Ave, Columbus, OH 43210 USA

The torsional split Hopkinson bar technique is used for testing materials at strain rates above 10,000 1/s. This strain rate, which is an order of magnitude higher than the typical with this technique, is obtained by using a specimen having a very short gage length. Results from tests on steel, aluminum, and titanium show that there is a significant increase in rate sensitivity as the strain rate increases from the range of 1,000's to the range of 10,000's 1/s. The observed increase in strain rate sensitivity is in line with results obtained from

testing with the pressure-shear plate impact technique at strain rates of the order of 100,000 1/s. In addition, the results show that the formation of adiabatic shear bands is sensitive to the strain rate. Microscopic examination of tested specimens shows that the deforming specimen gage is typical of the microstructure of the bulk material. A numerical analysis of the stresses in the specimen, and a comparison of results from tests with specimens of various lengths at the same strain rate, shows that even though some error is introduced when a very short specimen is used, the increase in strain rate sensitivity represents the true material response.

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DYNAMIC RESPONSE OF NATURAL AND ENGINEERED SYNTHETIC BRITTLE/DUCTILE LAMINATE COMPOSITES: *Kenneth S. Vecchio*¹; David Harach¹; Andrew Strutt¹; ¹University of CA, San Diego, Dept. of AMES, Materials Science Group, La Jolla, CA 92093-0411 USA

The quasi-static and dynamic response of brittle/ductile laminate composites are investigated over a range of strain rates, orientations, and loading conditions. The natural brittle/ductile composites, in the form of seashells, were studied to form the basis for mimicking these microstructures and developing engineered synthetic laminate composites (biomimetics). The synthetic composites are metallic (Ti, Ni, or Fe) -intermetallic (Ti-aluminide, Ni-aluminide, or Fe-aluminide) laminate composites containing a range of layer thicknesses, phase volume fractions, layer sequencing, and numbers of layers. The mechanical responses of these synthetic composites were evaluated in order to optimize the microstructure for improved dynamic performance. Deformation behavior and damage evolution will be discussed as a means of supporting the optimization process. Ballistic performance of these natural and synthetic laminate composites will also be presented.

3:40 PM BREAK

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A SHOCK-INDUCED OMEGA TRANSFORMATION IN TANTALUM AND TANTALUM-TUNGSTEN ALLOYS: *Luke M. Hsiung*¹; ¹Lawrence Livermore National Laboratory, Materials Science and Technology, L-370, P.O. Box 808, Livermore, CA 94551-9900 USA

A shock-induced omega transformation has been observed within shock-recovered pure Ta and Ta-10W samples. Plate- or lath-like ω phase is found to coexist with the $\{112\}<111>$ type deformation twins. Both deformation twins and shock-induced ω phase are formed along the $\{211\}$ slip planes, and have a common $\{211\}$ habit plane with the parent β matrix. It is suggested that the $\{211\}<111>$ shear has played a major role in the occurrence of the omega transformation in shocked pure Ta and Ta-10W. The result of higher volume fraction of shock-induced ω phase in Ta-10W than in pure Ta suggests that the shock-induced omega transformation can be promoted by solid-solution alloying. A dislocation mechanism for the shock-induced omega transformation is proposed and discussed. This work was supported by the Joint DOD/DOE Munitions Development Program under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

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A COMPARISON OF MECHANICAL RESPONSE BETWEEN A WROUGHT AND AN INVESTMENT CAST GAMMA-TiAl ALLOYS AS A FUNCTION OF STRAIN RATE AND TEMPERATURE: *Zhe Jin*¹; George T. Gray¹; ¹Los Alamos National Laboratory, Materials Science and Technology Division, MST-8, MS G755, Los Alamos, NM 87545 USA

Mechanical responses of a wrought and an investment cast gamma-TiAl alloys at strain rates ranging from 0.001/s to 3000/s and temperatures from -196°C to 1000°C were studied in compression. Both alloys exhibited similar work hardening rates and strain rate sensitivi-

ties within the strain rates and temperatures studied. Although the temperature dependence of yield and flow stresses was similar in these two alloys, the wrought TiAl alloy showed high yield and flow stresses compared to the cast TiAl alloy. The difference in their yield and flow stresses is ascribed to the smaller grain size in the wrought TiAl alloy and the difference in texture between them. The yield and flow stresses were found to be sensitive to texture in both alloys. Mechanisms controlling mechanical responses at different strain rates and temperatures are analyzed.

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SPONTANEOUS AND FORCED SHEAR LOCALIZATION IN HIGH-STRAIN-RATE DEFORMATION OF TANTALUM: *Yuejian Chen*¹; Marc A. Meyers¹; Vitali F. Nesterenko¹; ¹University of California, San Diego, Department of Applied Mechanics and Engineering Sciences, 9500 Gilman Drive, La Jolla, CA 92093 USA

High-strain-rate shear localization was induced in tantalum by (a) lowering the deformation temperature or (b) subjecting it to high strains by dynamic deformation (up to $\epsilon_t = -0.8$) or (c) pre-shocking (at $\epsilon_{eff} = 0.22$) and then deforming it. Although at ambient temperature the deformation of tantalum is macroscopically uniform to high strains ($\epsilon_t @ -0.8$), at 77 K shear localization under the same loading condition was developed with a critical strain of $-0.2, -0.3$. This higher propensity to shear localization at low temperatures is a direct consequence of the combination of lower heat capacity and higher rate of thermal softening. At the three temperatures investigated (77, 190, and 298 K), localization occurs at strains significantly higher than the instability strains (the maxima of the adiabatic stress-strain curves for these three temperatures). The thicknesses of the forced localization regions and shear bands were found to be a function of temperature, and decreased with decreasing temperature (at the same strain) in accord with the equation proposed by Y. Bai et al. (Acta Mechanica Sinica, 2(1986), 1). Shock deformation of tantalum enhances its predisposition to subsequent shear localization, and this was demonstrated by subjecting shocked and unshocked specimens to high strain, high strain rate deformation through the collapse of a thick-walled cylinder assembly.

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DYNAMIC BEHAVIOUR OF SOME STRUCTURAL MATERIALS AT STRAIN RATES 10^2 - 10^4 S⁻¹: *Anatoly M. Bragov*¹; Andrey K. Lomunov¹; Vladimir B. Korobov¹; Andrey G. Ugodchikov¹; ¹Research Institute of Mechanics, State University of Nizhny Novgorod, 23, Gagarin Ave., Korp.6, Nizhny Novgorod, Nizhegorodskaya obl. 603600 Russia

In this lecture we suggest the results of the study of the effect of the strain rate and its history of change on the physico-mechanical properties of some metals and alloys. With the help of Kolsky method and using the Split Hopkinson Pressure Bar dynamic diagrams have been obtained for deformation under tension, compression and shear as well as limit characteristics of strength and plasticity. Along with macroscopic properties, microstructural features of the high strain rate processes have been investigated. To this end an analysis of microhardness, of residual microstresses of the second kind and of the block-type structure parameters has been accomplished. Based on the results obtained a mechanism is suggested which explains the strain-rate hardening of the materials tested. The complex of physico-mechanical properties obtained testifies to a complicated rheological behaviour of the materials tested.

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ON THE TIME-TEMPERATURE CORRESPONDENCE FOR YIELD STRESS DURING THE DYNAMIC DEFORMATION OF METALS: Alexey A. Gruzdokov¹; *Nikita F. Morozov*¹; Yuri V. Petrov¹; ¹St.Petersburg State University, Mathematics & Mechanics Faculty, Petrodvorets, Bibliotechnaya pl., 2, St.Petersburg 198904 Russia

The dynamic yield criterion applicable for both high-strain-rate and quasistatic deformation of metals is proposed. The criterion is based on the notion of incubation time previously proposed by au-

thors for use in short pulse fracture mechanics. In special cases the approach coincides with the classical critical stress and the Campbell time integral of stress criteria. The incubation time is associated with the mean dislocation move velocity. On the basis of this criterion the estimation of the time-temperature correspondence for mild steel is carried out. Good agreement between theoretical calculations and well-known experiments is observed.

EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Diffuse Interface Approaches to Microstructure Evolution

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; Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Dept.; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Tuesday PM Room: International Ballroom
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Paul Fife, University of Utah

2:00 PM INVITED PAPER

PHASE FIELD MODELS OF SOLIDIFICATION: *James A. Warren*¹; ¹NIST, Metallurgy Division & CTCMS, Bldg 223/B164, Gaithersburg, MD 20899 USA

Phase field models are now a popular technique for simulating the dynamics of solidifying materials. These models employ the concept of a diffuse liquid-solid interface and energy penalties for gradients in place of the more traditional interface tracking techniques. Many of the fundamental ideas embodied in current phase field models were first elucidated by John W. Cahn in 1960 (nearly twenty years before the development of current models). This talk will address the enormous variety of phenomena which are now being modeled with this method, with emphasis on directional solidification, grain boundary formation and grain growth.

2:45 PM INVITED PAPER

MODELING PRECIPITATION AND COARSENING OF ORDERED INTERMETALLICS USING SYSTEMS OF CAHN-HILLIARD AND ALLEN-CAHN EQUATIONS: *Long-Qing Chen*¹; ¹Penn State University, Materials Science and Engineering, 118 Steidle Building, University Park, PA 16802 USA

Precipitation of an ordered intermetallic phase or phases from a disordered matrix is a basic process that underlies the processing of many advanced alloys such as high-temperature superalloys and ultralight aluminum alloys. It involves a number of different processes, including atomic ordering to produce the ordered structure, compositional clustering to reach the equilibrium precipitate composition, and coarsening to reduce the total interfacial energy. The

precipitate morphology and coarsening kinetics depend on many factors such as volume fraction, temperature, and the lattice mismatch between precipitates and matrix. In this presentation, a phase-field model using systems of Cahn-Hilliard and Allen-Cahn equations will be presented for modeling the microstructural evolution during precipitation of ordered intermetallics from a disordered matrix. The dependencies of the two-phase morphology and the corresponding coarsening kinetics on the volume fraction, the lattice mismatch, and an externally applied stress will be discussed. The results will be compared with existing theories and experimental measurements. This work is supported by the Office of Naval Research and the National Science Foundation.

3:30 PM BREAK

3:50 PM INVITED PAPER

THE ROLE OF INTERFACES AND DOMAIN BOUNDARIES IN ORDER-DISORDER TRANSITIONS: *Annick Loiseau*¹; ¹Laboratoire d'Etudes des Microstructures, Unite Mixte Onera-Cnrs, O.N.E.R.A., B.P. 72, 29 av. de la Division Leclerc, 92322, CH=E2tillon France

In recent years substantial experimental and theoretical progress has been made in our knowledge of the thermodynamics and kinetic behaviour of domain boundaries and interfaces in order-disorder transitions. Recent advances include quantitative studies of this behaviour at equilibrium of domain walls and surfaces close to a phase transition and numerical simulations of the kinetics of ordering reactions and microstructural evolutions. The scope of the talk is to present the state of the art in this field.

FATIGUE BEHAVIOR OF TITANIUM ALLOYS: Fatigue in Titanium Intermetallics

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

Tuesday PM Room: Athens/Berlin
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: G. Lutjering; R. O. Ritchie

2:00 PM KEYNOTE

EFFECTS OF MICROSTRUCTURE ON THE FATIGUE CRACK GROWTH RESISTANCE AND TOTAL LIFE OF GAMMA TiAl BASED ALUMINIDES: *P. Bowen*¹; *Z. W. Huang*¹; *M. Salmani*¹; ¹The University of Birmingham, School of Metallurgy and Materials, Edgbaston, Birmingham B15 2TT UK

The influence of microstructure, lamellar colony size, and lamellar colony orientation on fatigue crack growth resistance and total life at room and elevated temperatures will be highlighted. Particular emphasis throughout the presentation will be placed on the fully lamellar and/or near (fully) lamellar microstructures. In this context detailed TEM observations of dislocation and twinning behaviour local to a fatigue crack-tip will be introduced, and micromechanisms of crack advance will be discussed with particular reference to translamellar and intralamellar failure. The importance of both aligned lamellar colonies and randomised lamellar colonies on fatigue crack growth resistance and total life will be addressed. In particular, the

problem of premature interlamellar failure under cyclic loading, and its engineering significance, will be explored. Here, the surface condition of the testpiece must also be taken into account and the influence of as-machined conditions and residual stresses produced by shot peening will be considered. The concept of stress-sampling volume correlations will then be used to rationalise the total life of gamma based aluminides under cyclic loading observed for both plane sided and notched testpieces. Such correlations will be essential and must be quantified if the performance of large scale components is to be predicted (and approved) on the basis of tests carried out on relatively small scale testpieces.

2:30 PM KEYNOTE

OPPORTUNITIES AND CHALLENGES FOR THE USE OF GAMMA TiAl ALLOYS IN HIGH PERFORMANCE TURBINE ENGINES: *James M. Larsen*¹; A. H. Rosenberger¹; K. Li¹; R. John¹; D. C. Maxwell¹; W. J. Porter¹; S. K. Plank¹; N. E. Ashbaugh¹; ¹U.S. Air Force, WL/MLLN, Bldg. 655, 22230 Tenth St., Ste 1, Wright Patterson Air Force Base, OH 45433-7817 USA

Gamma TiAl alloys are highly promising candidates for replacement of more dense nickel-base superalloys currently used in a variety of components in high performance gas turbine engines. The applications under consideration include both static and rotating components, many of which will be subjected to complex loading spectra containing combinations of low and high-cycle fatigue over a range of temperatures. To quantify opportunities and challenges to the widespread use of gamma alloys in engines, a variety of anticipated service conditions were examined, and the effects of a range of loading variables on component fatigue life were evaluated. Key life-limiting conditions were identified for a variety of microstructures. Factors addressed include effects of intrinsic material defects, service-induced damage, loading spectra, temperature, and environment. Emphasis is given to damage tolerance requirements for major, fracture critical components such as engine disks. For components that are exposed to significant levels of high cycle fatigue, such as airfoils, resistance to crack formation and threshold crack growth are crucial to achieving reliability. As an aid to interpretation of the findings, structural capabilities of gamma alloys are contrasted with properties of more conventional materials. The results of this study suggest avenues for materials development and component design that maximize the potential for the use of these materials for long-term applications under realistic engine operating conditions.

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EFFECTS OF MACHINING ON FATIGUE STRENGTH AND CRACK INITIATION IN Ti-47Al-2Nb-2Cr: *P. E. Jones*¹; D. Eylon²; ¹General Motors Powertrain Division, Saginaw, MI 48605-5073 USA; ²University of Dayton, Graduate Materials Engr., 300 College Park, Dayton, OH 45469-0240 USA

Conventional machining processes, such as turning and grinding, will be used to fabricate fatigue loaded components from gamma titanium aluminide alloys. These metal removal processes created a severely work hardened surface layer on Ti-47Al-2Nb-2Cr (at%) samples produced by permanent mold casting and heat treated to a near-gamma microstructure. Effects of this layer on the high cycle fatigue strength (10⁶ cycles) and fatigue initiation sites were examined above and below the ductile-to-brittle transition temperature. All samples were tested to failure under the same step loading profile. Comparisons were made between samples having the same load history. At room temperature, fatigue strength and initiation sites were equivalent for turned and electropolished surface conditions. At the anticipated service temperature, 760C, the work hardened layer quickly recrystallized. This fine recrystallized surface enhanced the fatigue crack initiation resistance of turned specimens when compared to electropolished samples which did not recrystallize during the test. Surprisingly, the severe surface deformation resulting from conventional machining did not impair the high cycle fatigue behavior of this intermetallic alloy under the conditions evaluated.

3:20 PM BREAK

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FATIGUE STRENGTH OF GAMMA TITANIUM ALUMINIDE AFTER IMPACT DAMAGE: *C. M. Austin*¹; P. K. Wright¹; S. L. Draper²; J. M. Perriera²; B. A. Lerch²; M. V. Nathal²; M. P. Rubal³; P. S. Steif³; T. S. Harding⁴; J. W. Jones⁴; ¹G.E. Aircraft Engines, Cincinnati, OH 45140 USA; ²NASA-Lewis Research Center, 21000 Brookpark Road, Cleveland, OH 44135 USA; ³Carnegie Mellon University, Department of Mechanical Engineering, Pittsburgh, PA 15213-3890 USA; ⁴University of Michigan, College of Engineering, 2300 Hayward Street, Ann Arbor, MI 48109 USA

Gamma titanium aluminide alloys have properties that are well-matched to the requirements of low pressure turbine blades, offering significant weight reduction potential. A possible exception is impact resistance: gamma blades will be much more vulnerable than superalloy blades. Since impact damage is rare in the latter parts of the turbine where gamma is to be used (barring major upstream malfunction), the actual requirement for impact resistance is difficult to define. Nevertheless, extensive work is being done to characterize the nature of impact damage and its ramifications. A variety of objectives and approaches is being pursued. One group is studying the relationships between impact events, specimen geometries, and observed deformation and cracking. Another group is studying the features controlling crack formation and subsequent growth in fatigue. Another group is directly measuring the fatigue resistance of simulated airfoils after ballistic impacts done at temperature and under tensile load. This paper will provide an overview of these programs and the engineering context in which they are being performed. The authors are grateful for support from NASA and AFOSR.

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FATIGUE LIFE PREDICTION IN IMPACT-DAMAGED GAMMA TiAl: *Trevor S. Harding*¹; J. Wayne Jones¹; Tresa M. Pollock²; Matt P. Rubal³; Paul S. Steif³; ¹University of Michigan, Materials Science and Engineering, 2105 H.H.Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA; ²Carnegie Mellon University, Materials Science and Engineering, Wean Hall, Pittsburgh, PA 15213 USA; ³Carnegie Mellon University, Mechanical Engineering, Scaife Hall, Pittsburgh, PA 15213 USA

The room temperature and elevated temperature growth of fatigue cracks induced by impact damage in gamma titanium aluminides has been investigated in terms of application to damage in low pressure turbine blades. The responses of two gamma titanium aluminide alloys, a duplex Ti-47.9Al-2.0Cr-1.9Nb alloy and nearly fully lamellar Ti-47.3Al-2.2Nb-0.5Mn-0.4W-0.4Mo-0.23Si alloy, were examined in this study. Damage was induced by low speed impacts and characterized in terms of critical parameters for predicting fatigue failure stress. An experimental method for determining this stress was developed. Results for tests conducted at both room temperature and 600°C will be discussed. Modeling of near-threshold growth of damage induced cracks and its effect on fatigue failure stress prediction will be described, along with an explanation of the possible effects of small crack behavior in impacted specimens.

4:20 PM

FATIGUE DEFORMATION AND FAILURE IN WROUGHT Ti-46Al-2Cr-3Nb-0.2W: *Y-W Kim*¹; D. M. Dimiduk¹; ¹UES, Inc., 4401 Dayton-Xenia Road, Dayton, OH 45432 USA

The fatigue properties of a wrought gamma TiAl alloy in air were investigated at temperatures between 600 and 870°C in a duplex (GS~12 micron) and a fully-lamellar (FL) (GS~300 micron) microstructural forms. Tension-tension (R=0.1) fatigue experiments conducted on hourglass specimens show that both microstructures exhibit fairly flat S-N curves at 600°C, with runout fatigue strengths (FS) as high as 0.96UTS at 10⁷ cycles. The duplex material shows higher FS's, which is consistent with their tensile strengths. At and above 800°C, both materials exhibit a two-stage behavior, a gradual decrease of FS at the low cycle regime and a more rapid drop at higher cycle

regime, and the FL material exhibits higher fatigue resistance than the duplex material. The dependency of fatigue behavior on microstructure and temperature is explained using the corresponding tensile behavior with respect to respective brittle-ductile transition temperatures (BDTT). At all temperatures tested, crack-initiation appears to be related to soft grains, microstructural nonuniformity, or flaws. The rapid crack growth behavior under cyclic loading conditions and the small but apparent effect of microstructure on the propagation are discussed in connection with the da/dN curves measured on the same materials. The fracture modes at low temperatures are predominantly transgranular for both materials, but as temperature approaches BDTT, intergranular fracture and lamellar splitting become more important. This boundary weakening is similar to that observed under tensile loading conditions, and its implications will be discussed.

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FATIGUE LIFE PREDICTION FOR TiAl-BASED ALLOY: *Kwai S. Chan*¹; ¹Southwest Research Institute, Materials Engineering Department, 6220 Culebra Road, P.O. Drawer 28510, San Antonio, TX 78228-0510 USA

The prediction of fatigue life in TiAl-based alloys is considered in this paper. The fatigue characteristics of TiAl-based alloys are first summarized. Particular attention is focused on the mechanisms of fatigue crack initiation, growth, and coalescence in these alloys, followed by appropriate treatments of these individual processes. The use of conventional life prediction approaches to predict the fatigue life of TiAl-based alloys is then examined through parametric calculations. Fatigue life and crack growth approaches are both considered, either with or without using the concept of a fatigue limit or a large crack growth threshold. The roles of small fatigue cracks and current crack nucleation and coalescence in fatigue life are also addressed. These results are used to identify possible stress regimes where adequate damage tolerance characteristics can be achieved in TiAl-based alloys.

5:00 PM

ELEVATED TEMPERATURE FATIGUE CRACK GROWTH BEHAVIOUR OF A TiAl-BASED ALLOY: *Gilbert Henaff*¹; *C. Mabru*¹; *J. Petit*¹; ¹ENSMA, Laboratoire de Mécanique et Physique des Matériaux, UMR CNRS 6617, Téléport 2, BP 109, Futuroscope Cedex 86960 France

TiAl-based alloys are currently holding high promises as potential elevated-temperature structural materials. Indeed thanks to their superior combination of specific strength as well as their good oxidation and burn resistance, they outperform conventional Ti alloys and may also replace superalloys in the temperature range 550-850°C. With this prospect their damage tolerance must be assessed. However their fatigue crack growth behaviour is still ill-known. In particular a literature survey indicates the influence of temperature on the fatigue crack growth resistance is extremely controversial. The reason is that the observed behaviour actually results from a complex balance of different factors which may act in opposite ways. The present investigations is addressing this issue. The material considered is a quaternary alloy Ti-48Al-2Mn-2Nb tested in the as-cast condition. The methodology used aims in uncoupling the respective influence of temperature on the various factors involved in the growth process: intrinsic resistance, environmental deleterious influence, crack closure shielding, Testings have been carried at room and elevated temperature, in vacuum and ambient air. Some ancillary testings on intermediate atmospheres have also been performed at room temperature in order to gain deeper insights in the embrittling mechanisms. Thus the intrinsic fatigue crack growth resistance, i. e. the resistance exhibited in an inert environment and after crack closure correction, has been investigated at room and elevated temperatures (up to 850°C). It comes out that increasing the temperature induces an enhanced fatigue crack growth resistance in the mid to high growth rate regime in relationship with an improvement in fracture toughness. However the near-threshold behaviour is nearly unaffected when the temperature varies. The same remark applies for the fatigue crack

propagation in ambient air. Besides it has been shown that the magnitude of crack closure effects is nearly similar whatever the temperature and unaffected by environment. In particular, even at elevated temperature, the oxide-induced closure mechanism is not relevant in these materials. In-situ observations support a prevailing roughness-induced mechanism. As a consequence it appears that the environmental contribution in the slow growth rate regime is nearly the same at room temperature as in the range 650- 850°C. This result is discussed in terms of active species, surface reaction kinetics and possible embrittlement by hydrogen released by the dissociation of adsorbed water vapour molecules.

HOT DEFORMATION OF ALUMINUM ALLOYS: Constitutive Modeling, Texture Evolution

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

Tuesday PM

October 13, 1998

Room: Orchard

Location: O'Hare Hilton Hotel

Session Chairs: Paul Dawson, Cornell University, Sibley School of Mechanical and Aerospace Engineering, Ithaca, NY 14853 USA; Dr. Armeand J. Beaudoin, University of Illinois, Urbana-Champaign, IL USA

2:00 PM INVITED PAPER

RECRYSTALLIZATION PROCESSING OF HOT DEFORMED ALUMINUM: "WHAT IS KNOWN AND WHAT NEEDS TO BE UNDERSTOOD": *R. D. Doherty*¹; ¹Drexel University, Department of Material Engineering, Philadelphia, PA 19104 USA

Recent work on rolled or plane strain compressed commercial purity aluminum by different groups has largely solved the long standing problems of the origin of recrystallization texture and its dependence on deformation temperature, strain and initial grain structure. The key features are the apparent stability of cube oriented grains and the ability of such grains to develop low stored energies so giving a very high frequency of cube grain nucleation especially after warm deformation. A further critical feature has been identification of the importance of "orientation pinning - the strong inhibition of growth of recrystallized grains with an orientation that occurs very frequently in the deformed structure. The critical feature of this pinning is when the spacing, in the normal direction, of the texture component in the rolled material being significantly less than the final grain thickness in the fully recrystallized state. However, at least two major problems need to be understood before these initial successes can be repeated in other cases of aluminum alloy processing. The first is a means of predicting the stability of different orientations during deformation and the second task is to understand the precipitation of supersaturated solute during warm deformation of high solute, age-hardening alloys and the influence of such precipitation on recrystallization processing.

2:30 PM INVITED PAPER

MECHANISMS RESPONSIBLE FOR TEXTURE DEVELOPMENT IN A 5182 ALUMINUM ALLOY DEFORMED AT ELEVATED TEMPERATURE: *M. G. Stout*¹; *S. R. Chen*¹; *U. Fred Kocks*¹; *A. Schwartz*²;

S. R. MacEwen³; A. J. Beaudoin⁴; ¹Los Alamos National Laboratory, MST-8: Structure/Property Relations, Materials Science & Technology Division, Mail Stop G755, Los Alamos, NM 87544 USA; ²Lawrence Livermore National Laboratory, Los Alamos, NM 94550 USA; ³Alcan International Ltd., Kingston, Ontario K7L 5L9 Canada; ⁴University of Illinois at Urbana-Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA

The textures that develop in a 5182 aluminum alloy as a result of monotonic high-temperature compression have been investigated. We found that the deformation texture was a function of temperature. For compressive deformation at 200°C and below the material formed the classic (101) deformation texture, while the material develops a texture that is a combination of the classical uniaxial compression deformation texture, (101), and static recrystallization texture, (001), as a result of the deformation alone when the deformation temperature was above 400°C. Our investigation has focused on determining the mechanism responsible for the development of this unusual progression of deformation textures. In order to understand the mechanism(s) responsible for this particular development, we have performed interrupted compression experiments as well as microstructural analyses. The deformation experiments consisted of deforming to a given strain, holding at temperature for various times at zero strain rate, followed by either quenching or further deformation. In addition we have performed orientation imaging microscopy (OIM) to identify the shapes of grains with particular orientations and grain to grain orientation relationships. Finally, crystal plasticity theory was used to model whether or not deformation via different slip systems and diffusion mechanisms could produce the (101) and (001) texture components. Our conclusions are summarized as follows: The texture development is not a result of static processes, i.e. static recrystallization at the various hold times altered neither the textures nor the constitutive behavior. Using the crystal plasticity analysis, we could not predict the (001) texture component, as a result of deformation, with reasonable assumptions of active slip systems. Finally, the microscopic OIM results are consistent with these observations. Sponsored by: Basic Energy Sciences, Division of Materials Sciences, US Department of Energy.

3:00 PM INVITED PAPER

EXPERIMENTAL VALIDATION OF MICROMECHANICAL MODEL PREDICTIONS FOR COLUMNAR GRAIN POLYCRYSTALS: *R. Becker*¹; *H. Weiland*¹; ¹Alcoa Technical Center, Alcoa Center, PA 15609 USA

Polycrystal plasticity models for predicting the evolution of crystallographic texture during deformation have been available for several decades. They have been successful in explaining why certain crystallographic orientations are preferred and have provided reasonable predictions of crystallographic orientation distributions. However, quantitative comparisons of the evolution of lattice orientations in individual grains with experimental results have been less than satisfactory. This discrepancy has been attributed to essential simplifications in the models whereby each orientation is assumed to deform homogeneously and that each grain deforms independently of the influence of the specific surrounding grains. In more recent years, several research groups have incorporated the kinematics of crystal plasticity as the constitutive model in finite element codes. Simulations have been successful in predicting the development of shear bands in single crystals as well as slip patterns and lattice rotations near bicrystal boundaries. By representing the combined effects of several orientations at each integration point, process models have provided predictions of earing in cup drawing and the evolution of crystallographic texture during rolling, forging and extrusion processes. Despite these successes, finite element micromechanical models of the grain structure in a polycrystal have not been successful in simulating the details of the grain fragmentation and local lattice rotations. The reason given is that the two dimensional models are not capable of capturing the true three dimensional interactions of grains in a polycrystal. Thus, validation of micromechanical models for polycrystal deformation would require either three dimensional polycrystal models or two dimensional polycrystals. The former necessitates not only significant computational resources but full three

dimensional characterization of the polycrystal before and after deformation. The latter approach is simpler as it requires only creation of polycrystals with a columnar grain structure. Columnar grain structures have been cast for commercial purity aluminum and three binary aluminum alloys to assess the ability of micromechanical models to simulate details of the deformation of a polycrystal. The columnar grain samples have been deformed at room and elevated temperature in a channel die with the columnar direction of the grains aligned with the zero strain direction of the channel. Microstructural measurements at the surface of the specimens were obtained at several stages of deformation by orientation imaging microscopy. At the final strain, the specimens were sectioned along their mid-plane to obtain lattice orientation measurements devoid of the surface effects. The measured initial grain structure was used to define the initial state of the micromechanical models. The full specimen cross section was modeled to alleviate concerns about the effects of boundary conditions on the deformation. The slip system constitutive behavior and slip mode were altered to account for changes in mechanisms at the various temperatures. Results from both the experiments and analysis are given as well as a detailed comparison.

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COUPLED FINITE ELEMENT-MONTE CARLO SIMULATION OF MICROSTRUCTURE AND TEXTURE EVOLUTION DURING HOT DEFORMATION OF ALUMINUM ALLOYS: *B. Radhakrishnan*¹; *G. Sarma*¹; *T. Zacharia*¹; ¹Oak Ridge National Laboratory, Computer Science and Mathematics Division, Oak Ridge, TN 37831-6140 USA

A novel simulation technique for capturing the microstructure and texture evolution during hot working of fcc polycrystals is presented. The technique is based on coupling a microstructural deformation model based on crystal plasticity with a Monte Carlo simulation of recovery and recrystallization. The deformation model captures the stored energy and the crystallographic orientation distributions in the microstructure for a given incremental deformation. The Monte Carlo simulation captures the microstructural evolution by thermally assisted phenomena such as recovery and recrystallization in the corresponding time increment. The recovery model captures the evolution of the dislocation substructure and also the formation of dynamically recrystallized nuclei. A unique feature of the simulation technique is its ability to predict the orientations of the dynamically recovered/recrystallized grains, so that it is possible to follow the texture evolution as a function of deformation. The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. DE-AC05-96OR22464. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes. *This research was sponsored by the Division of Materials Science, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation. The authors acknowledge the use of the Intel Paragon XP/S 35 located in the Oak Ridge National Laboratory Center for Computational Sciences (CCS), funded by the Department of Energy's Office of Scientific Computing.

4:00 PM INVITED PAPER

POLYCRYSTAL SIMULATIONS OF TEXTURE EVOLUTION DURING DEFORMATION PROCESSING: *G. Sarma*¹; *B. Radhakrishnan*¹; *T. Zacharia*¹; ¹Oak Ridge National Laboratory, Oak Ridge, TN 37831-6140 USA

Some recent research on the hot deformation of aluminum crystals has led to the theory that at elevated temperatures, slip occurs on {110}<110> systems in addition to the usual {111}<110> systems active at lower temperatures. The effect of these additional slip systems on the texture evolution of aluminum single and polycrystals is studied using finite element simulations. The crystals are deformed in plane strain compression, and the constitutive response is modeled using crystal plasticity to track the reorientation and work hardening of the crystals. By discretizing each crystal with a large number of elements, the non-uniform deformations due to local inhomogene-

ities and interactions with neighboring crystals are modeled. The resulting textures and microstructures at different strains are examined with regard to effect of including the additional systems, initial orientation of the single crystals, and stability of the cube orientation in a polycrystal. The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. DE-AC05-96OR22464. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes. *This research was sponsored by the Division of Materials Science, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation. The research was supported in part by an appointment to the Oak Ridge National Laboratory Postdoctoral Research Associates Program administered jointly by the Oak Ridge National Laboratory and the Oak Ridge Institute for Science and Education. The authors acknowledge the use of the Intel Paragon XP/S 35 located in the Oak Ridge National Laboratory Center for Computational Sciences (CCS), funded by the Department of Energy's Office of Scientific Computing.

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MODELING OF THE RECRYSTALLIZATION TEXTURES OF AL-ALLOYS AFTER HOT DEFORMATION: *O. Engler*¹; *H. E. Vatne*²;

¹Los Alamos National Laboratory, Center for Materials Science, K765, Los Alamos, NM 87545 USA; ²Hydro Aluminum a.s. R & D Materials Technology, P.O. Box 219, Sunndalsorm N-6601 Norway

The recrystallization textures of most Al-alloys can be traced down to a growth selection of grains with an approximate 40% $\langle 111 \rangle$ orientation relationship out of a limited spectrum of preferentially formed nucleus orientations. To model this behavior, a function f^{nuc} describing the probability of nucleation and a function f^{grow} representing the growth probability must be derived. The resulting recrystallization textures are then simulated by multiplying these two probability functions. The probability of nucleation is given by the distribution of potential nucleus orientations, which have been determined by local texture analysis for the most important nucleation sites in commercially deformed Al-alloys, i.e. cube-bands, grain boundaries and second phase particles. If several nucleation sites are active simultaneously, the nucleation probabilities are weighted according to their respective contributions. For that purpose, the number of nuclei forming at each nucleation site is calculated according to an approach to derive the number of nuclei forming at the respective nucleation sites, which is based on experimental investigations on the nature and particularly on the evolution of the various nucleation sites. The probability of growth of the respective grains is associated with a 40% $\langle 111 \rangle$ transformation of the rolling texture. The paper describes the model approach of recrystallization texture simulation in Al-alloys and gives examples of recrystallization textures simulated regarding a variation of different metallurgical parameters to demonstrate the predictive power of the model.

INTERSTITIAL AND SUBSTITUTIONAL SOLUTE EFFECTS IN INTERMETALLICS II: Titanium 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

Tuesday PM Room: Madrid
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Y. Kim, Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, Ohio 45433 USA; P. J. Maziasz, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

2:00 PM

EFFECTS OF W, Mo AND B ADDITIONS ON THE STRUCTURE AND PROPERTIES OF LAMELLAR TiAl ALLOYS: *C. T. Liu*¹; *P. J. Maziasz*¹; *D. J. Larson*¹; ¹Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

This paper summarizes our recent work on the effects of alloying additions of W, Mo, and B on the microstructure and mechanical properties of lamellar TiAl alloys based on Ti-47Al-2Cr-2Nb (at. %). The dual-phase alloys were prepared by arc melting and drop casting, followed by hot extrusion at temperatures above T_α . The mechanical properties of the TiAl alloys were determined by tensile testing at temperatures to 1000°C, and their microstructure and solute distribution were characterized by TEM and atom probe field ion microscopy. The mechanical properties of the lamellar TiAl alloys are sensitive to their microstructural features, which are strongly influenced by the alloy additions at levels $< 2\%$. Boron is effective in refining lamellar structures, in addition to pin the colony boundaries by borides. Tungsten tends to segregate to α_2 / γ interfaces and stabilize fine lamellar structures. Mo promotes the formation of β phases, which harden the alloys at ambient temperatures but lower the strength at elevated temperatures. The correlation between the microstructure and mechanical properties will be discussed. Research was sponsored by the Division of Materials Science, US Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

2:30 PM

ALLOY DESIGN OF TiAl-BASED ALLOYS: *Y.-W. Kim*¹; ¹Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433 USA

Significant improvements in tensile properties in wrought gamma TiAl alloys have recently been realized possible by refining lamellar grains and spacing. Grain refinement was achieved by various means, including: adding small amounts of boron, employing novel processing routes, and conducting heat treatments in two-phase fields. Lamellar spacing was refined mainly by innovative heat-treatment cycles involving rapid cooling through, or before, and after the lamellar formation process. These refined microstructures were also found to enhance creep resistance, fatigue strength and damage tolerance at temperatures below BDTT. The improvements, however, were made often at the expense of higher-temperature deformation/fracture resistance. The investigations at Wright Laboratory have indicated that the deficiencies can be significantly alleviated by alloy modifica-

tion and/or microalloying. This talk briefs the fundamentals for designing TiAl alloys, and then discusses the importance, as well as limitations, of combining microstructure control and chemistry modification with highlights from specific examples. *UES, Inc., Dayton, OH 45432, USA

3:00 PM

THE INFLUENCE OF SOLID SOLUTIONS ON FLOW BEHAVIOR IN GAMMA-TiAl: *C. Woodward*¹; S. A. Kajihara²; A. Kajihara²; S. I. Rao²; D. M. Dimiduk²; ¹Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright Patterson AFB, OH 45433-6533 USA; ²UES Inc., Materials Research Division, Dayton, OH 45432

Intermetallic alloys, such as TiAl and NiAl, are an important class of structural material due to their low density and excellent strength retention at high temperature. Modifications of alloy chemistry are often used to tailor the intrinsic flow behavior of these structural materials. Relaxed structures and energies, for intrinsic and substitutional point defects are calculated using a first principles plane-wave-pseudopotential method. Calculated defect energies are used to predict the density and site preferences of solid solutions (Si, Nb, Mo, Ta and W) in gamma-TiAl. Size and modulus misfit parameters are calculated and the interaction of these defects with active deformation modes are evaluated within anisotropic elasticity theory. The derived interaction strength is then related to solid solution strengthening for these defect centers. Solid solution effects for the binary alloy are in good agreement with experimental observations and several ternary compositions are predicted to have improved flow properties at intermediate temperatures.

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YIELD POINT PHENOMENA OF TiAl-BASED ALLOYS CONTAINING BORON: *T. T. Cheng*¹; R. Botten¹; P. Bate¹; H. A. Lipsitt¹; ¹The University of Birmingham, School of Metallurgy and Materials, Edgbaston, Birmingham, B15 2TT United Kingdom

It is becoming standard practice to add boron to TiAl-based alloys for refining the grain size in the as-cast microstructure. In addition to the grain refinement. Kampe et al. (S.L. Kampe, P. Sadler, L. Christodoulou and D.E. Larsen, *Met & Mat. Trans. A*, 25 (10), 2181-2197 (1994).) have reported a strengthening effect greater than that could be accounted for by particulate strengthening due to borides. In this presentation the issue has been addressed by comparing tensile data obtained from a number of paired TiAl-based alloys whose compositions are identical except for the addition of boron. A yield plateau and strain aging phenomena have been observed in all those alloys which have a duplex microstructure and contain boron. It is proposed that boron additions result in solute locking of the dislocations and the results above the interpreted in that light.

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THE EFFECT OF CARBON CONCENTRATION ON PRIMARY CREEP OF AN INVESTMENT CAST Ti-47Al-2Mn-2Nb-0.8 WT.% TiB₂ ALLOY: *D. Y. Seo*¹; T. Everard¹; T. R. Bieler¹; P. A. McQuay²; ¹Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; ²Howmet Research Corp., 1500 S. Warner St., Whitehall, MI 49461-1895 USA

Creep experiments on an investment cast XDTM alloy in the as-HIPed duplex microstructure with carbon additions exhibit significantly improved primary creep resistance compared to the same alloy without carbon additions. The carbon levels examined are 0.006, 0.04, 0.07 and 0.11 wt%. The microstructure is investigated in selected tensile creep specimens deformed to 0.5% strain at 650°C and 276 MPa, 760°C and 815°C at 138 MPa. A ten-fold increase in time to 0.5% was observed under all conditions tested with a carbon concentration between 0.04 and 0.07 wt%, with little or no additional benefit from higher concentrations. Prior studies with high levels (0.1 wt%) of interstitial oxygen and nitrogen indicated only a two-fold increase in primary creep times to 0.5% strain (MRS symp. proc. vol. 460). The higher amounts of oxygen and nitrogen interstitials

also caused a higher volume fraction of the alpha-2 phase, and increased the contribution of mechanical twinning in lamellar refinement. Primary creep investigations on low carbon alloys indicate that primary creep is divided into two stages. The initial rapid process refines lamellar spacing up to strains between 0.2 and 0.5%, and it has a low activation energy. The second primary creep process has a higher activation energy, and no lamellar refinement is associated with this process (*Metal. Mater. Trans* 29A, p. 89). The effect of carbon on these two primary creep processes will be examined using measurements of initial lamellar spacing, and the changes in lamellar spacing after 0.5% strain.

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DISLOCATION-SOLUTE EFFECTS IN Al-RICH TiAl INTERMETALLICS: *J.M.K. Wiezorek*¹; M J. Mills¹; H. L. Fraser¹; ¹The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43221 USA

Single-phase TiAl alloys with slightly Al-rich compositions in the range of up to 56at.%Al exhibit both anomalous strengthening and serrated flow at intermediate temperatures. Traditionally and in analogy to intermetallics with the L1₂-structure the anomalous strengthening has been associated with an intrinsic effect, i.e. cross-slip locking. The operation of such a mechanism requires the activity of superdislocations with dissociated core configurations. However, for TiAl a yield strength anomaly is also observed when the predominantly activated deformation systems consist of ordinary dislocations with compact cores which are not prone to undergo Kear-Wilsdorf like locking. Furthermore, serrated flow has been observed for single-phase TiAl alloys. The latter constitutes an extrinsic effect, an interaction between mobile dislocations and thus far unidentified obstacles. The present transmission electron microscopy based study elucidates the micro-mechanisms underlying the observed mechanical behavior of single-phase Ti-(52-56at.%)Al alloys. The microscopic observations are discussed in relation to the mechanical behavior of the alloys.

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CREEP DEFORMATION OF TWO-PHASE TiAl ALLOYS CONTROLLED BY THE VISCOUS GLIDE OF INTERFACIAL DISLOCATIONS: *L. M. Hsiung*¹; T. G. Nieh¹; ¹Lawrence Livermore National Laboratory, Materials Science and Technology Division, P.O. Box 808, L-370, Livermore, CA 94551-9900 USA

A linear creep behavior, i.e. creep rate is linearly proportional to the applied stress, was observed when two-phase TiAl alloys with a refined lamella microstructure (thickness of γ' lamella: 100-300 nm) were creep deformed at stresses < 400 MPa and at temperatures above 650°C. TEM examinations of the corresponding deformation substructures revealed that interface sliding by a cooperative motion of interfacial dislocations is the predominant deformation mechanism. The measured activation energy (145-160 kJ/mol) of creep was found to be close to the activation enthalpy of oxygen diffusion in TiAl. This supports the idea that the viscous glide of interfacial dislocations dragged by interstitial atoms controls creep in this regime (<400 MPa, T > 650°C). A constitutive equation for the linear creep behavior is accordingly derived.

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, UES, Inc., Dayton, OH 45432-1894 USA; Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

Tuesday PM Room: Paris A
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Andy Rosenberger, Air Force Research Laboratories, Materials and Manufacturing Directorate, Wright Patterson Air Force Base, OH USA USA

2:00 PM INVITED PAPER

MODELLING AND CHARACTERISATION OF FATIGUE CRACK INITIATION AND GROWTH IN SiC FIBRE REINFORCED Ti MMCs UNDER CYLOADING: P. Bowen¹; J. Liu¹; W. Ding¹; D. Parrott¹; N. Wang¹; ¹The University of Birmingham, School of Metallurgy and Materials/IRC in Materials for High Performance Applications, Edgbaston, Birmingham B15 2TT UK

The initiation and growth of fatigue cracks in SiC fibre reinforced Ti MMCs differs from that in monolithic alloys. Minor defects and damage can arise from a local fibre failure and/or defects at the ends of fibres. Thus, fatigue crack initiation in the matrix of the composites can be facilitated by such defects and damage. Observations have been made both for selectively reinforced Ti MMCs containing artificially embedded fibre ends and modelling based on a 3-D finite element method shows that the local stress at the ends of fibres is critical. For fatigue crack growth, in selectively reinforced (clad) testpieces, fatigue cracks can either grow into the composite so that fibre bridging can occur or along the fibre-matrix interface thus preventing fibre bridging. To characterise this latter mode of failure has necessitated extensive experimental and modelling studies. In uniformly reinforced Ti MMCs, fibre bridging can always occur and to model the bridging effect, in-situ SEM tensile tests have been conducted to provide insight into the physical nature of bridging. A combination of modelling and in-situ SEM measurements has been used to predict accurately the transition from crack arrest to catastrophic specimen failure.

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STRESS RATIO AND TEMPERATURE EFFECTS ON CRACK PROPAGATION IN A UNIDIRECTIONALLY REINFORCED ORTHORHOMBIC TITANIUM MATRIX COMPOSITE: Reji John¹; Jay R. Jira²; ¹Air Force Research Laboratory (AFRL/MLLN), Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433 USA; ²University of Dayton Research Institute (UDRI), Dayton, OH 45469 USA

Titanium matrix composites (TMC) based upon orthorhombic "O" phase titanium aluminide and high strength Ultra SCS™ fibers are being considered for applications in rotating engine components operating at temperatures up to 650C. This presentation discusses the results of the experimental and analytical investigation of fatigue crack propagation in [0]8 Ultra SCS™ /Ti-22Al-26Nb. The tests for crack growth perpendicular and parallel to fibers were conducted at 21, 482 and 649C with stress ratios of 0.05, 0.5 and 0.8. Baseline tests were also conducted on fiberless Ti-22Al-26Nb under similar loading conditions. During some of the tests on the composite, crack

opening displacement profiles were measured to verify and/or calibrate the fiber bridging models. The ability of the shear lag models to predict bridged crack growth perpendicular to fibers in Ultra SCS™ /Ti-22Al-26Nb will be discussed. The correlation between crack growth parallel to fibers in Ultra SCS™ /Ti-22Al-26Nb and that in fiberless Ti-22Al-26Nb will also be discussed.

2:50 PM INVITED PAPER

ANALYSIS OF BRIDGED SURFACE CRACKS IN FIBER REINFORCED METAL MATRIX COMPOSITES: G. Bao¹; Z. Lin¹; ¹The Johns Hopkins University, Baltimore, MD 21218 USA

In this work, a systematic study of fracture of fully bridged 3D surface cracks is performed using a finite element method. The surface cracks under study include semi-circular, semi-elliptical, quarter-circular and quarter-elliptical; both finite and infinite body problems are considered. A non-dimensional loading parameter is identified which combines the applied load, the material properties of the composite, and the interfacial sliding shear stress. The mode I stress intensity factors at the crack tip and the maximum stress in the bridging fibers are calculated for surface crack specimens with different crack aspect ratio, crack depth, specimen width, and the effective applied load. Numerical curve fit solutions are developed for the crack tip stress intensity and the maximum fiber stress of fully bridged 3D surface and corner cracks based on the finite element solutions. An engineering model is further developed to predict the crack-tip stress intensity factors of partially bridged surface cracks based on the curve fit functions for fully bridged and unbridged surface cracks. The model is subsequently validated by comparing the model predictions with experimental results for fatigue crack growth of surface cracks with an initial unbridged notch.

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DAMAGE AND DEFORMATION PREDICATION OF [0] TITANIUM MATRIX COMPOSITES UNDER FATIGUE-DWELL LOADING: D. J. Buchanan¹; N. E. Ashbaugh¹; A. H. Rosenberger²; ¹Air Force Research Laboratory (AFRL/MLLN), Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA; ²University of Dayton Research Institute, 1031 Irving Ave., Dayton, OH 45419-0218 USA

A one-dimensional deformation and rupture model for a unidirectional continuously reinforced titanium matrix composite, SCS-6/Ti-6Al-4V, has been extended for application to thermomechanical fatigue spectrum loading. A refined constitutive model for the Ti-6Al-4V matrix has been shown to be more accurate than the Bodner-Partom model for predicting creep and relaxation behaviors. The deformation model also incorporates a two parameter Weibull distribution to characterize the SCS-6 fiber strengths. The Weibull parameters, m and so, were deduced for an entire panel from a single test specimen. The deformation, accumulation of fiber failures and rupture life for other specimens in the panel were predicted with the deduced values of m and so. Acoustic emission data were collected during the tests and provided the history of the damage accumulation to compare with the predicted fiber failures from the model. The predictions accurately captured the creep and relaxation behavior and predicted time to failure within a factor of two.

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S/N BEHAVIOUR AND FIBRE STRENGTH DEGRADATION FOR Ti-MMCs: S. Zamperini¹; P. McDonnell¹; H. Mori²; P. Bowen¹; ¹The University of Birmingham, School of Metallurgy and Materials, Edgbaston, Birmingham B15 2TT UK; ²The University of Tokyo, Research Centre for Advanced Science and Technology, 4-6-1 Komaba, Meguro-ku, Tokyo 153 Japan

The total life of TiMMCs have been studied to determine the extent of fibre degradation due to fatigue and elevated temperature exposure. In all cases fibre strength distributions have been com-

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EVALUATING DAMAGE MECHANISMS IN SCS-6/TI-15-3 CAUSED BY ELEVATED TEMPERATURE FATIGUE: *J. R. Calcaterra*¹; *S. Mall*²; ¹Department of the Air Force AFRL/VAVS, Building 63, 1901 Tenth Street, Wright-Patterson AFB, OH 45433-7605 USA; ²Department of the Air Force, AFIT/ENY Building 640, 2950 P. Street, Wright-Patterson AFB, OH 45433-7765 USA

The majority of experimental fatigue studies performed on Titanium Matrix Composite (TMC) systems have taken the specimens to complete failure. Unfortunately, this type of study provides little information as to the progression of damage mechanisms or the residual strength degradation of the material. To address this concern, an experimental test program has been conducted on SCS-6/Ti-15-3 composites with laminate orientations of [0]8, [0/90]2s, and [0/45/90]s. The main goal of this research was to determine the effect of strain-controlled, fully-reversed, elevated-temperature fatigue on the degradation of residual strength in TMCs. Specimens with varying fiber volume fractions were subjected to isothermal fatigue and residual tensile strength tests, all at 427°C. Specimens were cycled until they had reached either 50 or 75 percent of their fatigue life. After cycling, the specimens were statically tested until failure. Extensive post-test microscopic examination of the specimens provided information as to the progression of damage mechanisms during fatigue. Results indicate the laminate orientation is the primary factor controlling damage progression morphology. The results also show that fiber volume fraction has an effect on both strain-controlled fatigue life and residual strength degradation.

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EXTRINSIC AND INTRINSIC FACTORS INFLUENCING FATIGUE CRACK GROWTH BEHAVIOUR OF SiC FIBRE REINFORCED Ti MMCs: *J. Liu*¹; *P. Bowen*¹; ¹The University of Birmingham, School of Metallurgy and Materials/IRC in Materials for High Performance Applications, Edgbaston, Birmingham B15 2TT UK

The improved fatigue crack growth resistance of Ti MMCs derives from fibre bridging, and the magnitude of this resistance is strongly affected by extrinsic and intrinsic factors. The aim of this paper is to examine the effects of extrinsic factors, and the intrinsic factor of fibre strength. The results show that under the same initial K_{max} values, small initial notches under high stress can be more damaging than large initial notches under low stress. In addition a wider specimen can always sustain higher initial K_{max} values without failure, and this can be explained by a modified bridging model. Single fibre tensile tests have been conducted on fibres extracted from composite specimens to establish the degradation of fibre strength under fatigue. The influence of fibre failure on fatigue crack growth has been monitored by a combination of acoustic emission (AE) and direct current potential drop (DCPD) techniques. The influence of reduced fibre strength in producing poorer fatigue crack growth resistance has thus been quantified in this study.

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MICROSTRUCTURAL ANALYSIS OF CVD-SiC MONOFILAMENTS FOR METAL MATRIX COMPOSITES: *K. L. Dyos*; *R. A. Shatwell*

A comparative study of commercially available and experimental monofilaments has been carried out using fractography techniques, micro-indentation, X-ray diffraction, scanning electron microscopy, wavelength dispersive analysis and transmission electron microscopy. The fracture surfaces have been examined, and their behavior correlated with their microstructures. Initiation of fracture has been identified at various interfaces. The strength has been linked to variations in the SiC microstructure, which affect the fracture toughness.

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DAMAGE MECHANISMS AND FIBER FABRIC ORIENTATION EFFECTS ON MECHANICAL BEHAVIOR OF A NEXTEL|312(BN)/BLACKGLAS| LOW COST CERAMIC COMPOSITE: *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; ¹University of Tennessee at Knoxville, Dept. of Materials Science and Engineering, Dougherty Engr. Bldg., 1512 Middle Dr., Knoxville, TN 37996-2200 USA; ²Northrop Grumman, 9314 W. Jefferson Blvd., Dallas, TX 75211 USA; ³University of Tennessee, Dept. of Biochemistry, Cellular and Molecular Biology, 232 Sci. & Engr. Facilities, Knoxville, TN 37996 USA; ⁴University of Tennessee, Dept. of Mechanical and Aerospace Engineering and Engineering Sciences, 323 Perkins Hall, Knoxville, TN 37996 USA

In this paper, the damage mechanisms and fiber fabric orientation effects on the mechanical behavior of a Nextel 312(BN)/Blackglas composite are investigated. The laminated composite is a newly developed advanced low cost ceramic composite. The Blackglas matrix is pyrolyzed with a crowfoot satin Nextel 312 ceramic fiber (3M product) as reinforcements. A four-point-bend testing is performed using a Material Testing System (MTS) at room and elevated temperatures. Samples are cut with fiber fabric at angles of 30, 45, 60 to the fiber longitudinal direction. A scanning electron microscopy (SEM) is employed to examine microstructures, fiber/matrix interface, and fracture surfaces. Chemical variations after oxidation are detected by a SEM energy dispersive X-ray spectroscopy (EDXS) line-scan technique. A quantification model based on a classical laminated plate theory is also provided to rationalize the experimental results. The present work is supported by National Science Foundation under a contract number, EEC-9527527, with Mary Poats as the program manager.

PAUL A. BECK MEMORIAL SYMPOSIUM: Paul A. Beck Memorial Symposium - II Heat Capacity & Alloy & Structure

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee

Program Organizers: Karl A. Gschneidner, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Michael V. Nevitt, Clemson University, Dept of Physics & Astronomy, Clemson, SC 29634 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899

Tuesday PM

Room: Rome

October 13, 1998

Location: O'Hare Hilton Hotel

Session Chair: Michael V. Nevitt, Clemson University, Dept. of Physics & Astronomy, Clemson, South Carolina 29634 USA

2:00 PM INVITED PAPER

LOW-TEMPERATURE HEAT CAPACITY AS RELATED TO MAGNETIC PHENOMENA: *Vitalij K. Pecharsky*¹; ¹Iowa State University, Ames Laboratory, 242 Spedding, Ames, IA 50011-3020 USA

The heat capacity is a fundamental thermodynamic property of solids that allows experimental measurements of various contributions to the total entropy. The experimental magnetic heat capacity and the magnetic entropy yield important information about magnetic phenomena. A study of the heat capacity in magnetic fields allows one to clarify the nature of magnetic phase transitions, even though the behavior of the magnetic heat capacity does not disclose details of the magnetic structure. Low-temperature (1.5 to 350 K) heat capacity data measured in zero magnetic fields and in fields up to 10 T for a variety of magnetic systems including ferromagnets, antiferromagnets and spin glasses show distinct differences in the observed heat capacity. The discussion will include the use of the heat capacity as a valuable tool to: 1) determine the magnetic ordering temperature; 2) to refine the magnetic phase diagram; 3) to establish whether the magnetic phase transition is of the first or the second order; and 4) to estimate whether the observed magnetic phenomenon occurs in the matrix phase or in the impurity phase. This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences under Contract No. W-7405-ENG-82.

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CONCEPTS IN THE STRUCTURAL CHEMISTRY OF STABLE AND METASTABLE INTERMETALLICS: *Bill C. Giessen*¹; ¹North-eastern University, Barnett Institute and Dept. of Chemistry, 360 Huntington Ave., 341 MU, Boston, MA 02115 USA

Through his and his students' encyclopedic studies of alloy phase diagrams, especially those of ternary transition metal systems, Paul Beck contributed early to the present immense body of knowledge on alloy phases, especially their occurrence and structural classification. We trace the path of increasing understanding from phenomenological beginnings of alloy phase classification through the systematic structural compilations, the emphasis on distance and size relationships (Pearson), "electron lattice" concepts (Schubert), the pseudopotential approach (Heine and Weaire), quasichemical considerations and structure maps (Miedema; Phillips and Rabe) to first principles calculations (Pettifor), considering metastable alloy phases as well as equilibrium ones and extrapolating to future progress in the field.

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TRANSITION ELEMENT ALLOYS AS IMPLANTABLE BIOMATERIALS: *Denes I. Bardos*¹; ¹Smith & Nephew, Inc., Scientific Affairs, 1450 Brooks Rd., Memphis, TN 38116 USA

All current high quality implants are fabricated from transition element alloys. Phase relationships, re crystallization, sigma phase and other intermetallics as well as other metallurgical advances pioneered by Dr. Beck enabled the author to contribute to the development of new and improved biomaterials for orthopaedic surgical implant applications. The high strength thermo-mechanically formed Co-Cr-Mo alloy is now the choice for total hip replacement (arthroplasty), Ti-Zr-Hf alloys offer far superior biocompatibility than any other alloy, other Ti alloys are the safest hypo allergenic materials. A special medical grade Fe-Cr-Ni-Mo type stainless steel has been improved for trauma applications with unprecedented clinical results. Professor Beck's legacy lives on to reduce pain and restore mobility to millions of orthopaedic patients.

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4:05 PM INVITED PAPER

"METALLIC" ZINTL PHASES: ARE THEY POSSIBLE?: *Gordon J. Miller*¹; ¹Iowa State University, Department of Chemistry, 3659 Gilman, Ames, IA 50011 USA

Zintl phases are semiconducting compounds that bridge intermetallics and valence compounds: they are formed from elements with (semi)metallic character yet build structures according to the octet rule. Recent experimental and theoretical efforts with ternary aluminides show that intermetallic structures exist which have no energy gap and yet show a distinct separation between bonding and antibonding states. Examples from alkaline earth/Li/Al (CaLi(x)Al(2-x)), alkaline earth/Cu/Al (AECu(5-x)Al(8+x)), and rare earth/Au/Al (LnAu(x)Al(4-x)) demonstrate how the observed compositions, structures, atomic arrangements, and properties are influenced by the electronic structure. Results from synthetic, structural, and theoretical investigations will be presented. In fact, for these types of compounds, theoretical calculations are imperative as no simple chemical bonding picture exists for their description. This work is supported by the National Science Foundation under DMR-96-27161.

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PHASE STABILITY, POINT DEFECTS, AND FRACTURE TOUGHNESS IN Nb-Cr-Fe TERNARY SYSTEMS: *J. H. Zhu*¹; *P. K. Liaw*¹; *L. M. Pike*²; *C. T. Liu*²; ¹University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996 USA; ²Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

The phase stability, point defects, and fracture resistance in ternary Nb-Cr-Fe Laves phase system are studied in this paper. Our previous work indicates that when the atomic size ratios are kept identical, the average electron concentration factor, e/a , is the dominating factor in controlling the phase stability of NbCr₂-based transition-metal Laves phases. In order to verify the e/a correlation with phase stability experimentally, addition of Fe was selected to replace Cr in the NbCr₂ Laves phase. The boundaries of the C15/C14 transitions were precisely determined and the e/a ratios corresponding to the different Laves polytypes were calculated and compared to the previous prediction. It was found that the electron concentration and phase stability correlation is obeyed in the Nb-Cr-Fe system. The changes in lattice constant, hardness, and fracture toughness were also determined as a function of the Fe content, which were discussed in light of the phase stability difference of the alloys. The point defects of the binary NbFe₂ and NbCr₂ and ternary Nb(Fe,Cr)₂ were also determined and anti-site substitution was found to be the major defect mechanism in these Laves phases.

PROCESSING OF HIGH TEMPERATURE ALLOYS: Session I

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: Thomas Zogas, Carpenter Technology Corp., Reading, PA 19612-4662 USA; Ronald D. Noebe, NASA Lewis Research Center, Cleveland, OH 44135 USA

Tuesday PM Room: Paris C
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Thomas Zogas, Carpenter Technology Corp., Reading, PA 19612-4662 USA; Ronald D. Noebe, NASA Lewis Research Center, Cleveland, OH 44135 USA

2:00 PM

THE GROWTH-PATH METHOD FOR PREDICTION OF SPURIOUS GRAIN NUCLEATION: *Ralph E. Napolitano*¹; ¹National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD USA

Predictive techniques for the assessment of potential yield are essential for the cost-effective design and production of superalloy single-crystal castings. Such castings can be rendered defective by many types of crystal imperfections, several of which are difficult to control during processing. The tendency for the formation of these grain defects must be evaluated and minimized during the development of a casting design/process. Currently, yield maximization often involves several design cycles where castings are produced and evaluated experimentally to provide a basis for design modification. The high cost of this approach is driving the development of predictive techniques which can be used by casting designers to assess defect likelihood without the need for test castings. The growth-path technique, presented here, is a method for the quantitative assessment of the potential for spurious grain nucleation in a single-crystal casting. Ledge and platform areas of single-crystal turbine blades are especially prone to such defects. For a casting of complex geometry, the model must predict whether the undercooling in any part of the casting becomes sufficiently large to induce nucleation, before the dendritic structure growing from the grain selector reaches that point. The growth-path method uses thermal simulation results, a kinetic model for dendrite tip and branch growth, and a specified dendrite crystal orientation to determine the path by which the dendritic structure reaches any point or region in the casting. Using thermal simulation results and a nucleation model which may be based on actual experimental data, the nucleation tendency at all points along the path can be computed. The method is demonstrated for a René N5 casting and compared with experiment.

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DEVELOPMENT OF A P/M SUPERALLOY CONSOLIDATION PROCESS FOR CONVENTIONAL FORGING: *X. Pierron*¹; T. Banik¹; G. E. Maurer¹; ¹Special Metals Corporation, 4317 Middle Settlement Road, New Hartford, NY 13413-9576 USA

Recent investigation has identified hot isostatic pressing (HIP) conditions of P/M Superalloys that significantly enhance hot workability of the consolidated materials. This process incorporates HIP'ing of the powder at temperatures close to the liquidus of the alloy. It has been tested on UDIMET Alloy 720 and René 95 and has resulted in superior hot workability compared to that of conventional Superalloy powder metallurgy. Use of this method could result in reducing the cost of P/M Superalloys with the ability to produce large components with uniform microstructures and properties by subsequently using conventional forging practices other than isothermal forging. Results of the study on the effect of process parameter and composition on the hot workability of UDIMET Alloy 720 and René 95 will be presented. In addition, mechanisms leading to the enhanced workability observed in the P/M alloys consolidated by this new process will be proposed.

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MANUFACTURING OF IRON ALUMINIDE SHEETS BY THERMOMECHANICAL PROCESSING: *S. C. Deevi*¹; M. R. Hajaligol¹; V. K. Sikka²; C. Scorey³; ¹Philip Morris, Research and Development Center, 4201 Commerce Road, Richmond, VA 23261 USA; ²Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, Oak Ridge, TN 37831-6083 USA; ³Ametek Specialty Metal Products Division, P.O. Box 5807, Wallingford, CT 06492 USA

An innovative combination of powder metallurgy and thermomechanical processing were used to manufacture FeAl alloy intermetallic sheets. Green sheets of FeAl were obtained by roll compaction of water-atomized FeAl powder with a polymeric binder. Roll-compacted green sheets were debindered and partially sintered prior to cold rolling through tungsten carbide rolls. Cold rolling lowered the thickness, reduced the level of porosity, and heavily work hardened the FeAl sheets. Several intermediate annealings at or above 1100 C were found to be necessary to relieve the stresses (from work hardening) prior to rolling the sheets to a final thickness of 0.20 mm. Thermomechanical processing of cold-rolled sheets allowed the commercial manufacture of FeAl intermetallic sheets without the neces-

sity of hot rolling of a cast FeAl ingot. Mechanical properties of FeAl sheets are comparable to the properties of hot-extruded FeAl alloys and far superior to those of cast iron aluminides.

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DESIGN OF CARPENTER'S NEW 20-TON TILT-CHAMBER VIM FURNACE FOR MELTING OF PREMIUM WROUGHT SUPER-ALLOYS: *W. E. Johnson*¹; D. D. Wegman¹; ¹Carpenter Technology Corporation, P.O. Box 14662, Reading, PA 19601 USA

A new ALD Vacuum Technologies 20-ton tilt-chamber VIM furnace was commissioned in February 1997. The addition of this largest and most advanced tilt-chamber furnace in the world has enabled Carpenter to reliably manufacture premium VIM product in excess of 100 tons/day in order to meet the demand of the superalloy industry. Significant design and operational aspects which enhance the productivity, reliability, and product quality of the furnace to be reviewed are: 1) the dual melting/casting chamber design which allows many tasks to be performed simultaneously, 2) the tilt melt chamber utilizing external cable connections for quick transfer of furnace vessels, 3) the long launder used for metal transfer and inclusion flotation/removal and complementary video camera system, 4) the 5 MW power supply with supplementary 3-phase inductive stirring capability, 5) the Saveway lining monitor system providing real time measurement of metal penetration through the refractory lining, 6) the vacuum filter system to capture dirt and extend vacuum pump life, 7) the material recharge bucket system allowing for maximization of recharge weight and minimization of required recharges within a heat. The furnace has already been qualified to supply premium wrought product to many superalloy customers with the remaining qualifications in process. Multi disciplinary teams continue to work on improving the furnace productivity, reliability, and product quality.

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ALUMINUM VAPORIZATION KINETICS DURING SKULL MELTING OF TITANIUM ALUMINIDES : *Linda L. Rishel*¹; Thomas Kelly²; Tresa M. Pollock¹; Alan Cramb¹; ¹Carnegie Mellon University, MSE Department, 5000 Forbes Ave., Pittsburgh, PA 15213 USA; ²General Electric Aircraft Engines, One Neumann Way, Cincinnati, OH 45215 USA

Gamma titanium aluminides are currently being considered for aerospace and automotive components. Presently, gamma titanium aluminide components are cast after vacuum arc remelting or vacuum induction skull remelting. Control of melt chemistry, especially aluminum and other volatile elements, during melting in vacuum is critical to the successful implementation of these materials. In this work a series of experiments were conducted to determine the vaporization kinetics of aluminum from gamma titanium aluminides in the vacuum induction skull remelting process. Aluminum loss will be presented for different melt durations and vacuum levels. Rate controlling steps for the vaporization of aluminum from the meltbath will be discussed and a kinetic model for the vaporization of aluminum from the meltbath will be presented.

3:40 PM
CHARACTERIZATION OF SPRAY FORMED ALLOY 718 BILLET: Lee Shaw²; Graham Oakes²; Larry Jackman¹; *Chris O'Brien*¹; Robin Forbes Jones¹; Dick Kennedy¹; ¹Allvac-SMP, P.O. Box 5030, 2020 Ashcraft Avenue, Monroe, NC 28110-0530 USA; ²Allvac-SMP

Spray forming of superalloy billets by the Osprey Process is attractive because it offers the possibility of cost savings relative to conventional cast/wrought and powder metallurgy processing routes. Also, benefits may be derived from the unique structure that is generated by spray forming. In this presentation spray formed alloy 18 billet, 203 mm (8 inches) in diameter is characterized and compared to cast/wrought billet of the same diameter. The spray formed billet was produced from a sprayed preform 419 mm (16.5 inches) in diameter which was also characterized. Structures were investigated by macro etching, optical microscopy, and scanning electron micros-

copy. Hot workability was determined by Gleeble testing and various mechanical properties were measured. Thermal stability was also evaluated and electron beam buttons were melted to evaluate cleanliness. Several unique features were observed for the spray formed product.

4:00 PM

THE IMPORTANCE OF FINISHING TEMPERATURE CONTROL DURING HOT WORKING OF SUPERALLOYS: *R. B. Frank*¹; ¹Carpenter Technology Corporation, P.O. Box 14662, Reading, PA 19612-4662 USA

Because superalloys are designed for high strength at elevated temperatures, these alloys are very resistant to deformation during hot working operations. Because of this stiffness and typically narrow hot working temperature ranges, the temptation is to hot roll or forge as hot as possible to avoid reheating, surface tears, and wear of hot working equipment. However, finishing at lower temperatures within the typical hot working range of 1750-2100 F is often necessary to avoid hot tearing or to meet grain size and mechanical property requirements. The importance of finishing temperature control during hot working of several Carpenter superalloys including Pyromet alloys 718, A-286, 41 and 751 as well as corrosion-resistant alloys MP159 and Custom Age625 PLUS will be discussed.

4:20 PM

MODELING SPRINGBACK IN SUPERALLOY SHEET FABRICATION: *R. A. Jaramillo*¹; *B. A. Baker*¹; ¹Inco Alloys International Inc., 3200 Riverside Drive, Huntington, WV 25705-1771 USA

Precision forming of aerospace and automotive components is becoming increasingly critical as performance criteria become more demanding. It was, therefore, deemed advantageous to develop a more precise means of determining springback in superalloy sheet, particularly, because of the differing cost and tensile properties of superalloys compared to ferritic and stainless steels. A model was developed which incorporates the work-hardening behavior of a material into the springback model of Queener and De Angelis. Several work-hardening models were evaluated from which the Kock's differential equation was selected for best fit. Experimental bend tests using INCONEL alloy 625LCF were performed and compared to calculated values. Strengths and limitations of the calculation are discussed. INCONEL and 625LCF are trademarks of the Inco group of companies.

4:40 PM

THE ELEVATION OF THE INCIPIENT MELTING TEMPERATURE OF STELLITE 21 ALLOY THROUGH NITROGEN ALLOYING: *Wade L. Karlsen*¹; ¹Helsinki University of Technology, Laboratory of Engineering Materials, Otakaari 4, P.O. Box 4100, Espoo, FIN 02015 HUT Finland

The Co-Cr-Mo alloys such as Stellite 21 were historically used in structural components for high-temperature environments such as jet-engines. However, now more well known for its application in demanding biomedical components, it has suffered from incipient melting during high-temperature sintering heat-treatments associated with porous coating of implants. As a part of a broader study, this paper discusses how alloying a Stellite 21 with nitrogen has elevated the incipient melting temperature of the alloy from below 1240°C, to above 1260°C. Findings indicate that as the nitrogen content is increased, a Cr sub 2 N second phase is favored over the traditional Cr sub 23 C sub 6 carbide, leading to the subsequent elevation of the incipient melting temperature. Besides its significance to the biomedical component fabrication industry, this finding may also be of renewing interest to high-temperature applications of this alloy.

5:00 PM

PROCESSING OF HIGH TEMPERATURE ALLOYS BY LASER-ENGINEERED NET-SHAPING|: *Richard J Grylls*¹; *T J Lienert*¹; *D M Keicher*²; *H L Fraser*²; ¹The Ohio State University, Department of

Materials Science and Engineering, 2041 College Road, Columbus, Ohio 43210 U.S.A.; ²Optomec Design Company, 2701-D Pan American Freeway, NE, Albuquerque, NM 87107 U.S.A.

Laser-Engineered Net-Shaping (LENS|) is a novel process for direct fabrication of metal powders to form net-shapes direct from a CAD model. A focussed laser provides localized melting of a stream of metal powder, and the beam is rastered such that a large part can be built-up progressively. A wide range of metals have been processed in this way, from aluminum to tungsten. One advantage of the process is that melting is containerless, with surface tension providing support for the molten metal. Also, the small heat input and rapid heat extraction can give cooling rates of up to 10⁵ Ks⁻¹, such that the benefits of rapid solidification may be achieved in a bulk structure. In this presentation the application of LENS| to a variety of high-temperature alloys, such as nickel, titanium and niobium-based alloys, will be discussed. Detailed microstructural characterization has been used to investigate the effect of the processing technique on the final product. The advantages and disadvantages of this technique will be addressed. LENS| is a trademark of Sandia National Laboratories.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VII: Metal-Matrix Composites - I

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

Tuesday PM Room: Chicago
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Dr. M. Manoharan, Nanyang Technological University, Division of Materials Engineering, Singapore 639798; Dr. G. S. Bhat, University of Tennessee, Knoxville, TN

2:00 PM INVITED PAPER

A COMPARATIVE STUDY ON THE WORK HARDENING BEHAVIOUR OF AN UNDERAGED AND AN OVERAGED SiC REINFORCED ALUMINUM METAL - MATRIX COMPOSITE: *M. Gupta*¹; *M. Manoharan*²; ¹National University of Singapore, Department of Mechanical and Production Engineering 639798 Singapore; ²Nanyang Technological University, Singapore, Division of Materials Engineering, School of Applied Science 639798 Singapore

An understanding of the work hardening behaviour of particulate reinforced metal matrix composites is crucial in optimizing the parameters for deformation processing of these materials. The microstructure and mechanical properties of these composites can be altered by a suitable aging treatment. The precipitation hardening mechanisms can be changed by moving from an underaged to an overaged microstructure. In particulate reinforced composites, however, the strength and ductility, as measured by strain to fracture are determined predominantly by the effects of the reinforcement. Thus, a model for predicting the mechanical properties of particulate reinforced composites should be able to do so for all aging conditions. In this paper a modified continuum model is used to relate the work hardening behaviour of the composite to microstructural parameters. The model is also used to predict the fracture strain of the composite for an underaged and an overaged condition. The model is shown to predict the fracture strain of the composite quite accurately for both aging conditions.

2:30 PM INVITED PAPER

MIXED-MODE TOUGHNESS OF A 6061 ALUMINUM ALLOY BASED COMPOSITE: M. Manoharan¹; L. M. Tham²; R. K. Oruganti¹; H. P. Seow¹; M. Gupta²; ¹Nanyang Technological University, Division of Materials Engineering, School of Applied Science, Singapore 119260; ²National University of Singapore, Department of Mechanical and Production Engineering 119260 Singapore

The advancement of technology has increased the need for materials which have exceptional performance characteristics. In many situations where the structural integrity is important, the fracture toughness of the material is crucial in determining the suitability of a particular material. Traditionally fracture toughness has been determined under tensile or Mode I conditions. However, cracks in real materials can be subjected not just to tensile stresses but to complex stress states so that the development of suitable parameters to characterize mixed-mode crack initiation and propagation is important in the evolution of suitable design criteria. Further, observations indicate that initially flat cracks in some tough materials tend to reorient themselves to oblique planes during growth. For these materials, crack propagation can be said to occur under combined-mode conditions. This paper will outline some recent work on the mixed mode behaviour of a 6061 aluminum alloy based composite.

3:00 PM INVITED PAPER

THE ROLE OF FABRICATION METHOD ON STRENGTHENING AND TOUGHNESS OF A PARTICULATE METAL MATRIX COMPOSITE: M. J. Hadianfard¹; ¹Shiraz University, Department of Materials Science, School of University, Shiraz Iran

Discontinuously reinforced metal matrix composites (Mmes) have considered as materials for demanding conditions because of their superior stiffness, higher specific mechanical properties and higher wear resistance. The properties of (Mmes) are largely influenced by factors such as; matrix and reinforcement properties, reinforcement shape, size and volume fraction, and fabrication methods. The effects of four different fabrication methods; powder metallurgy & hot extrusion, on strengthening and toughness of a 6061 Al reinforced with SiC particles were investigated by using mechanical sand electron microscopy testing. Results show extensive effect of fabrication method on properties of the MMC. The results are discussed according to microstructural and metal/particles interfacial factors.

3:40 PM INVITED PAPER

BLADE FABRICATION PROCESS OF TITANIUM MATRIX COMPOSITES (TMC): T. Yamada¹; T. Tsuzuku¹; Y. Kawachi¹; K. Yasuhira¹; ¹Research Institute of Advanced Material Gas-Generator, 4-2-6 Kohnata, Bunkyo-ky, Tokyo 112 Japan

Continuous fiber reinforced titanium matrix composites (TMC) are attractive as one of the potential structural materials for aerospace applications, because of their high specific strength and stiffness. However, TMC parts are not yet put into practical use due to an enormous production cost resulting from process limitation requiring preliminary forming and elaborate tooling for consolidation. In order to reduce the production cost, superplastic TMC sheets (SiC/Ti-4.5Al-3V-2Mo-2Fe) have been developed, and deformation properties, mechanical properties and cavitation behavior have been investigated. SiC/Ti-4.5Al-3V-2Mo-2Fe composites were demonstrated to be superplastic with m-value of 0.58 and to show elongation exceeding 69% in true strain, at the temperature of 1048°K and strain rate of 5x10⁻⁵ s⁻¹. The blade-shaped model was formed successfully out of the newly developed TMC sheet by means of Argon-gas-pressure forming technique. Diffusion bonding techniques of attaching root blocks to deformed TMC sheets are also discussed.

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SUPERPLASTIC FOAMING OF Ti AND Ti-6Al-4V: Naomi G. Davis¹; David C. Dunand¹; ¹Northwestern University, Materials Science and Engineering, 2225 N. Campus Drive, Room 2036, Evanston, IL 60208 USA

Pure titanium and the titanium alloy Ti-6Al-4V were foamed by expansion of small, high-pressure, argon-filled pores. To delay cell wall busting, the foaming was carried out under conditions where the matrix exhibited transformation superplasticity. This mechanism does not depend on the grain size as in fine grained superplasticity but, instead, relies on internal stresses due to the density mismatch between the a and the b phases that occurs during the allotropic phase transformation. Elastic constants (Young's modulus, shear modulus and Poisson's ratio) were measured as a function of porosity and correlated to initial and foamed pore morphology.

4:30 PM

OPTIMIZATION OF ARTIFICIAL AGEING RESPONSE OF AL-SiC(P), METAL MATRIX COMPOSITE SYSTEM: D. Sood¹; G. Grewal¹; H. R. Anand²; ¹Thapar Corporate Research and Development Centre, Patiala India; ²Thaspar Institute of Engineering and Technology, Patiala India

In the present study, a systematic investigation is undertaken to parametrically model the artificial ageing characteristics of a composite system based on 2024 type aluminum alloy matrix reinforced with particulate silicon carbide. The composite system, which comprises of a series of compositions with varying volume fraction and mean particle size of the reinforcing phase, has been fabricated using a novel two-stage technique comprising of mushy-zone hot pressing followed by extrusion in a heated die. The entire study has been conducted in the framework of an analytical methodology based on statistical experiment design and two way analysis of variance with interactions effects included. Based on a rigorous analysis of the data obtained, a response surface, for the various optimal ageing parameters, is derived in the two variable design space of the volume fraction and mean particle size of reinforcing phase. Further, the ageing response of the metal matrix composite system is correlated with the evolving microstructure using optical metallography and scanning electron microscopy.

4:50 PM

DEVELOPMENT OF ALUMINUM-CORUNDUM METAL-MATRIX COMPOSITES: K. V. Sharma¹; ¹UVCE, Department of Mechanical Engineering, K.R. Circle, Bangalore, Karnataka 560001 India

The main objective of the paper is to characterize the aluminum-corundum particulate metal matrix composites. Addition of aluminum is expected to yield properties better than that of alloy. The mechanical properties better than that of alloy. The mechanical properties viz, ultimate tensile strength, compressive strength, wear resistance, etc. are evaluated. Aluminum corundum material will be prepared through liquid metallurgy technique using the vortex method, in which, a vortex is created in the melt through an impeller rotated at 400 RPM. The uncoated but preheated corundum particulates will be added into vortex in the melt & stirred thoroughly before pouring into molds. The specimen is tested for above properties and results presented. Work is already in progress and results are expected soon.

5:10 PM

MACHINABILITY STUDIES ON ALUMINUM-CORUNDUM METAL-MATRIX COMPOSITES: K. V. Sharma¹; ¹UVCE, Department of Mechanical Engineering, K.R. Circle, Bangalore, Karnataka 560001 India

Aluminum-Corundum composites were prepared using liquid metallurgy technique. Apart from other tests, machinability test were conducted to ascertain condition under which these composites can be machined, specifically to check the effect of particles in chip length. Earlier reports showed chip length of composite samples decrease with dispersion of ceramic particles in aluminum matrix. Al-Corundum composites were subjected to machinability tests at different cutting conditions, such as cutting speed, cutting feed and depth of cut. Fx, Fy and Fz with chip thickness and length were measured under various cutting conditions. These values were used to calculate chip thickness ratio Yc and hence shear plane angle ϕ_1 . Tangible evidence were provided using SEM Techniques.

PROCESSING & PROPERTIES OF ADVANCED STRUCTURAL CERAMICS: Advanced Structural Ceramics: Processing

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Rajiv S. Mishra, University of California, Dept of Chemical Engineering & Materials Science; Amiya K. Mukherjee, University of California, Dept of Chemical Engineering & Materials Science

Tuesday PM Room: London
October 13, 1998 Location: O'Hare Hilton Hotel

Session Chair: Brian Derby, University of Oxford, Department of Materials, Oxford, OX1 3PH UK

2:00 PM KEYNOTE

STRONG CERAMIC MATERIALS THROUGH NANO TO MOLECULAR LEVEL DESIGN: Koichi Niihara¹; Masafumi Kusunose¹; Tohru Sekino¹; Yong-Ho Choa¹; ¹Osaka University, The Research Institute for Scientific and Industrial Research (ISIR), 8-1 Mihogaoka, Ibaraki, Osaka 567 Japan

It is now well-known that the nanocomposite concept has a great possibility to essentially improve the mechanical properties of ceramic materials even at high temperatures. For example, the room temperature strength could be increased two to five times by only 5vol% nanodispersion, and the high temperature creep resistance was also improved two to four orders by this technology. In addition to the structural ceramic based composites, strong improvement in mechanical properties was also observed in the ceramic/metal systems. Recently, we applied the nanocomposite technology to the electronic ceramics such as BaTiO₃, ZnO and PZT, intermetallic compounds and also polymers, and found that this technology will work well even in these materials field. The purpose of nanocomposite technology extent is to find new materials design concept for developing the multi-functional ceramic materials, and many fruitful results are now coming in. In the BaTiO₃/Ni nanocomposite system, for example, two to three times improvement was achieved not only in mechanical properties such as strength and toughness but also in dielectric characteristics. For the Al₂O₃/Ni, MgO/BaTiO₃ and MgO/PZT nanocomposite systems, we succeeded to incorporate the fracture and/or stress sensing function originated from ferromagnetic of Ni and ferroelectric of BaTiO₃ and PZT with improved mechanical properties. Striking finding during these researches is that the functionally graded structure control of interfaces between the nano-sized particle and matrix drastically decreases the volume fraction of second phases at which the mechanical properties are strongly improved: 5vol% for usual nanocomposites to below 0.5vol% for this new materials with the functionally graded interface structure. In the presentation, new processing for the nanocomposites and the property improvements by this technology is first reviewed and then their property improvement mechanisms will be discussed. In these discussions, special emphasis will be placed on understanding of the roles of the interface structure. Finally, the new materials design concept of ceramic materials will be proposed. These new concept was induced from the molecular level design of the matrix grain and the functionally graded interface structure between the nano-sized particle and matrix grain.

2:40 PM

MICROWAVE SINTERING OF CaO DOPED ZrO₂ NANOCERAMICS: S. Bhaduri¹; E. Zhou¹; ¹University of Idaho, Metallurgical and Mining Eng., Moscow, ID 83844-3024 USA

Nanocrystalline CaO doped ZrO₂ powders were synthesized using the Auto Ignition technique. The objective of the work was to densify nanocrystalline ZrO₂ using an activated sintering process such as microwave sintering so that nanocrystalline grain size is maintained while achieving densification. Zr-nitrate and Ca-nitrates were used as oxidizers and urea was used as the fuel. The as-synthesized powders were nanocrystalline in size. Compacts were pressed and sintered at 1200YC for up to half an hour. It was seen that microwave coupling was better with more CaO content. Development of phases and microstructures will be discussed.

3:05 PM

FABRICATION OF Ni-Co ALLOY DISPERSED ALUMINA COMPOSITE AND ITS PROPERTIES: Sung-Tag Oh¹; Masanobu Awano²; Mutsuo Sando²; Koichi Niihara³; ¹Fine Ceramics Research Association, Synergy Ceramics Laboratory, 1-1 Hirate-Cho, Kita-Ku, Nagoya, Aichi 462-8510 Japan; ²National Industrial Research Institute of Nagoya, 1-1 Hirate-Cho, Nagoya, Aichi 462-8510 Japan; ³Osaka University, ISIR, 8-1 Mihogaoka, Ibaraki, Osaka 567-0047 Japan

In purpose of introducing ferromagnetism into structural ceramics, ceramic based composites dispersed with nano-sized phases were studied. In this approach, Al₂O₃, which has been well examined as a structural ceramics, and Ni-4.5 wt% Co alloy as a material having ferromagnetic property were selected for the ceramic matrix and dispersions, respectively. The composites were fabricated by the hydrogen reduction and the hot-pressing of Al₂O₃ and NiO-CoO powder mixtures, in which the Ni- and Co-nitrate was used as the source of NiO and CoO. Microstructural observation revealed that the nano-sized Ni-Co alloy particles were homogeneously dispersed in the Al₂O₃ matrix. The composite with 10 wt% alloy showed enhanced fracture strength as 1070 MPa. The composite exhibited the ferromagnetic property, and the magnetic response such as coercive force and magnetostriction showed the strong dependence on the dispersoid size.

3:30 PM

PROCESSING OF CERAMIC-MATRIX NANOCOMPOSITES BY HIGH PRESSURE SINTERING: Rajiv S. Mishra¹; Amiya K. Mukherjee¹; ¹University of California, Dept. of Chemical Eng. and Materials Science, One Shields Avenue, Davis, CA 95616 USA

Ceramic-matrix composites with nanocrystalline (grain size less than ~100 nm) matrix provide new opportunities, both scientific and technological. The experimental results on creep and toughness do not follow the existing theoretical framework. The knowledge gap and opportunities to develop ceramic-matrix nanocomposites will be highlighted. We have been able to synthesize fully dense alumina nanocomposites with grain size as fine as 30-40 nm. The results show significantly higher hardness and toughness in nanocomposites.

3:55 PM BREAK

4:05 PM KEYNOTE

AMORPHOUS AND CRYSTALLINE POLYMER-DERIVED CERAMICS: Fritz Aldinger¹; ¹MPI fuer Metallforschung, PML, Heisenbergstrasse 5, 70569 Stuttgart, Germany

The condensation of preceramic compounds is done by solid state pyrolysis, chemical vapour deposition or chemical liquid deposition. The general idea of such process routes is that the precursor molecules contain already structural units of the residual inorganics, thus providing novel paths of controlling the composition, atomic array and microstructure of materials. For the manufacture of bulk components the production of covalent bonded inorganics on the basis of Si, B, C and N by solid state pyrolysis is of special interest. Using proper precursor polymers either amorphous or crystalline materials can be

produced. Due to the lack of grain boundaries and a low atomic diffusivity in covalent materials their amorphous states provide thermal stability, oxidation resistance and very attractive mechanical properties at rather high temperatures. The crystalline materials also create substantial interest. Depending on the type of precursor, devitrification of the amorphous state into the nanocrystalline state occurs at very high temperatures providing the basis for thermally quite stable microstructures. Another favorable feature of the process is that for the shaping of products as parts, coatings, fibers or other components economic techniques of standard polymer engineering can be applied.

4:45 PM

MICROSTRUCTURAL STABILITY AND PROCESSING IN SILICON NITRIDE COMPOSITES: *K. Rajan*¹; Pavol Sajgalik²; ¹Materials Science and Engineering Department, Rensselaer Polytechnic Institute, Troy, NY 12180-3590 USA; ²Institute of Inorganic Chemistry, Slovak Academy of Sciences, Bratislava Slovak Republic

The relationship between microstructural evolution in beta silicon nitride composites and processing parameters is explored in this presentation. Of particular interest is the coupling of microchemistry and interface structure on anisotropic grain growth. The implications on developing controlled microstructures during processing by controlling interfacial chemistry is emphasized.

5:10 PM

PREPARATION, MICROSTRUCTURE AND OXIDATIVE STABILITY OF SiC-C NANO-COMPOSITES BY A POLYMER PRECURSOR ROUTE: *K. V. Moraes*¹; *K. Rajan*¹; *L. V. Interrante*²; ¹Materials Science & Engineering Department, Rensselaer Polytechnic Institute, Troy, NY 12180-3590 USA; ²Rensselaer Polytechnic Institute, Department of Chemistry, Troy, NY 12180-3590 USA

SiC-C nano-composites having different proportions of carbon and SiC were prepared by co-pyrolysis of a mixed precursor system. Allyl-hydridopolycarbosilane (AHPSC) was used as the precursor for SiC and a pitch as a precursor for carbon. Microstructural development in these materials as a function of annealing temperature up to 1600°C was studied by transmission electron microscopy. The oxidation rates in air, of these SiC-C nano-composites for various combinations of SiC and carbon will also be presented and correlated to the microstructure. This system is being studied as a potential source of oxidation-resistant fiber coatings for SiCf/SiCm composites.

5:35 PM

COMBINED MECHANICAL ALLOYING AND COMBUSTION SYNTHESIS IN THE TiH₂-B SYSTEM: *S. Ozbilen*¹; ¹Gazi University, Faculty of Technical Education, Metal Education Dept., Teknikokullar, Ankara Turkey

Combined mechanical alloying and combustion synthesis was carried out in the TiH₂-B system to investigate the influence of particle size of reactants in the exothermicity of the self propagating reactions together with the other effects of mechanical alloying on CS technique. Mechanically alloyed and compressed pellets of TiH-B mixtures were degassed and ignited in a reaction chamber. A combination of DTA, XRD and SEM study of samples before and after MA and CS was resulted in fruitful data collection for TiB-Ti ceramic matrix composites which will be presented and discussed in detail.

Technical Program

WEDNESDAY AM

DAMAGE PROCESSES IN ADVANCED MATERIALS: Composites and Metallic Glasses

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee, Corrosion and Environmental Effects Committee, Physical Metallurgy Committee

Program Organizers: Russ H. Jones, Pacific Northwest Natl. Lab., Richland, WA 99352 USA; Richard Ricker, NIST, Materials Rm B254, Gaithersburg, MD 20899; Norman S. Stoloff, Rensselaer Polytechnic Inst., MS&E Dept., Troy, NY 12190-3590 USA

Wednesday AM Room: Paris C
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: R. H. Jones, Pacific Northwest National Laboratory, Richland, WA 99352 USA; N. S. Stoloff, Rensselaer Polytechnic Institute, Troy, NY 12180-3590 USA

8:30 AM INVITED PAPER

MECHANISMS OF FRACTURE AND FATIGUE IN ADVANCED MATERIALS: R. O. Ritchie¹; ¹University of California, Materials Sciences Division, Lawrence Berkeley National Laboratory and Department of Materials Science and Mineral Engineering, Berkeley, CA 94720 USA

Whereas damage-tolerant design and life-prediction methodologies have been widely practiced for metallic structures for years, particularly in the nuclear and aerospace industries, the development of advanced structural materials, such as metallic glasses, toughened ceramics, intermetallics and their respective composites, poses particular problems. These problems pertain to the fact that, in general, these advanced materials possess high strength yet invariably low ductility, meaning that they are highly sensitive to the presence of flaws. Moreover, it is now known that these materials are susceptible, as in metals, to premature failure by fatigue; this provides a prominent mechanism for the subcritical growth of cracks and further limits life. In this presentation, studies to discern the microstructural mechanisms associated with subcritical crack propagation in intermetallics, ceramics and metallic glasses are examined, and compared to behavior in traditional metals. Despite obvious differences in the processes of microstructural damage and crack advance in such ductile and brittle materials, the commonality of mechanisms both ahead and behind the crack tip are explored.

9:10 AM INVITED PAPER

OXIDATION EFFECTS ON THE TIME-DEPENDENT FAILURE OF FIBER-REINFORCED SiC AT ELEVATED TEMPERATURE: E. Lara-Curzio¹; Peter F. Tortorelli¹; Karen L. More¹; ¹Oak Ridge National Laboratory, Metals and Ceramics Division, P. O. Box 2008, Oak Ridge, TN 37831-6069 USA

Under constant tensile loading in air between 600 and 900°C, Nicalon-reinforced SiC composites can exhibit significant strains to failure. This behavior can be explained on the basis of a progressive fiber-failure model that incorporates removal of the fiber-matrix interphase by oxidation, formation of silica on the Nicalon and SiC, and subsequent fiber failure and stress redistribution. Using a straight-forward fiber-bundle analysis, measured oxidation rates, and reason-

able assumptions, the model can accurately predict the observed stress-rupture curves and measured times-to-failure over extended temperature and loading ranges. Research sponsored by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, U. S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

9:50 AM INVITED PAPER

OXIDATION EFFECTS ON MECHANICAL PROPERTIES OF BLACKGLASS| MATRIX NEXTEL|312 REINFORCED CFCC'S: P. J. Balin¹; Michael J. McNallan; ¹University of Illinois-Chicago, CME Dept., M/C 246, Rm 3083 ERF, 842 W. Taylor St., Chicago, IL 60607 USA

Blackglas™ is a silicon oxycarbide glass, produced by pyrolysis of a preceramic polymer. Composites based on Blackglas™ can be fabricated by resin transfer molding and are suitable for use at moderately elevated temperatures. Mechanical properties of these composites may degrade as a result of oxidation reactions in service, with the kinetics of degradation affected by the pyrolysis procedure and service conditions. Composite panels of Blackglas™ matrix- Nextel™ 312 fiber reinforced CFCC's have been fabricated. Coupons cut from the panels have been subjected to 3-point and 4-point bend flexure testing before and after oxidation exposures at 600°C in air, with and without humidification. The effects of exposure time, humidity, and test geometry on the measured mechanical properties of the CFCC's are discussed.

10:30 AM

HIGH-TEMPERATURE DAMAGE PROCESSES IN SiC/SiC COMPOSITES: R. H. Jones¹; C. A. Lewinsohn¹; C. H. Henager¹; ¹Pacific Northwest National Laboratory, MSIN P8-15, Richland, WA 99352 USA

There are a number of high-temperature processes that can produce damage in SiC/SiC composites. Time-dependent matrix cracking as a result of fiber relaxation mechanism (FRM), interphase removal mechanism (IRM) or oxidation embrittlement mechanism (OEM) are three such processes. FRM, IRM and OEM occur at different environmental conditions and exhibit different effects on the material. FRM occurs over a range of temperatures but at very low O₂ concentrations while both OEM and IRM require the presence of O₂. IRM occurs at T > T_g, the glass transition temperature for SiO₂, and pO₂ less than atmospheric pressure while OEM occurs at T < T_g and atmospheric pressure of O₂. Damage from matrix cracking includes changes in the compliance of the composite, hermetic performance and loss of protectiveness for the interior fibers and interphases. However, under some circumstances, the fracture strength of the composite is unaffected for matrix cracks up to a/W of 0.6. Comparison between the various crack growth processes and the type of composite damage will be summarized.

10:50 AM

EFFECTS OF TIME, STRESS, AND ENVIRONMENT EXPOSURE ON A GLASS POLYURETHANE AUTOMOTIVE COMPOSITE: Kelly L. Willson¹; John M. Henshaw¹; Dan Q. Houston²; ¹University of Tulsa, Mechanical Engineering, 600 S. College Ave., Tulsa, OK 74104 USA; ²Ford Motor Company, Manufacturing Systems Department, 20000 Rotunda, MD 3135 SRL, Dearborn, MI 48121 USA

A continuous-strand mat (CSM) glass reinforced polyurethane composite is exposed to different environments (distilled water and warm humid air) for times up to 2000 hours and at stresses up to 50% of ultimate strength. The effects of these conditions on the material are described and analyzed. Residual properties (ultimate strength and modulus of elasticity) are seen to degrade with increasing time and

stress level. Most of the effects of the environmental exposures on residual properties are shown to be reversible upon drying out of the material. Moisture absorption (weight gain) is more rapid at higher stresses, but the saturation level appears to be independent of stress level. The interactions among the material, stress, and environment are investigated and discussed.

11:10 AM

MECHANISMS FOR FRACTURE AND FATIGUE-CRACK PROPAGATION IN A BULK METALLIC GLASS: *C. J. Gilbert*¹; V. Schroeder¹; R. O. Ritchie¹; ¹University of California, Department of Materials Science and Mineral Engineering, Berkeley, CA 94720 USA

Novel measurements of the fracture and fatigue properties of a newly developed bulk metallic glass alloy, $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ (at.%) have been made with specific emphasis on determining microstructural mechanisms. Fatigue pre-cracked compact-tension specimens indicated that the amorphous alloy had a plane-strain fracture toughness of ~ 55 MPa m. Under monotonic loading, fracture surfaces exhibited a typical vein morphology, and evidence for local melting was observed. Attempts were therefore made to rationalize the measured toughness in terms of a previously developed micromechanical model based on the Taylor instability, as well as on the observation of extensive crack branching and deflection. Upon crystallization, the alloy was severely embrittled with toughnesses dropping to ~ 1 MPa m. Commensurate with this toughness decrease was a marginal increase in hardness and a reduction in ductility (as measured via depth-sensing indentation experiments). Under cyclic loading, crack-propagation behavior in the amorphous structure was similar to that observed in traditional polycrystalline alloys, in that the crack-advance mechanism was associated with alternating blunting and resharping of the crack tip. This was evidenced by striations on fatigue fracture surfaces. Conversely, the (unnotched) stress-life properties were markedly different. Specifically, crack initiation occurred quite readily due to the lack of microstructural barriers which normally provide crack-arrest points; this resulted in a low fatigue limit of ~ 0.04 of the tensile strength.

11:30 AM

EXPERIMENTAL OBSERVATION OF DISLOCATION-TYPE DEFECTS IN AMORPHOUS ALLOYS: *Mikhail V. Finkel*¹; L. B. Zuev²; V. I. Danilov²; ¹DAATH-Scientific Center, Materials Science, 9926 Haldeman Ave. #36A, Philadelphia, PA 19115-1609 USA; ²Institute for Metal Physics, Tomsk Russia

Metallography methods have been used to experimentally discover dislocation-type defects in amorphous structure. Pile-ups of defects with the density of distribution up to $10^{12}m^{-2}$ have been revealed in various rapidly solidified amorphous alloys. Defects are oriented perpendicularly to the ribbon surface, have length of several micrometers, and their estimated cross-section diameter is 10^{-9} - 10^{-8} m. Reordering of defect pile-ups due to structural relaxation, plastic deformation, and fracture of material is studied. Formation of new and dissolving of existing defects is observed in zones of intensive plastic flow. Etching figure patterns similar to those observed for dislocations in crystals (such as straight chains of defects) are demonstrated. Fine structure and genealogy of defects are discussed from dislocation positions using existing and novel approaches.

DYNAMIC BEHAVIOR OF MATERIALS: Dynamic Fracture

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 Matis Sci & Engrg, Atlanta, GA 30332-0245; Ken S. Vecchio, University of California, Dept of Ames, San Diego, CA 92093 USA

Wednesday AM Room: Sydney
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Naresh N. Thadhani, Georgia Institute of Technology, Materials Science and Engineering, Atlanta, GA 30332-0245 USA; George T. Gray, Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

8:30 AM

DYNAMIC FRACTURE UNDER SPALL CONDITIONS: *Anna K. Zurek*¹; ¹LANL, MST-8, MS G755, Los Alamos, NM 87544 USA

The stress state of materials under spall conditions is drastically different than that for quasi-static loading. This difference arises from different stress triaxialities, global versus local strains, local porosity, and most of all from differences in strain rate and hardening due to the compressive wave preceding the spall inducing tensile wave. In this review paper we will describe the fracture process under spall conditions and compare it to fracture under quasi-static loading condition. We will concentrate on the shapes of the voids, their nucleation, growth and coalescence, as well as the total porosity arising from the voids dictated by the global and local stress triaxiality and strains. Finally, we will discuss recent advances in describing and quantifying damage accumulation specifically for incorporation into a micromechanical model of ductile fracture.

8:50 AM

DEFORMATION DYNAMICS OF AMORPHOUS SOLIDS: THE IMPLICATIONS FOR FRACTURE: *Michael L. Falk*¹; ¹University of California, Department of Physics, Santa Barbara, CA 93106 USA

Amorphous solids such as metallic glasses and non-crystalline ceramics exhibit both brittle and ductile fracture. Not coincidentally, these materials display complicated inelastic response to shear. Molecular dynamics investigations of the shear response of a simple two-dimensional two-component amorphous Lennard-Jones solid reveal analogous properties to those seen in actual materials. Examination of the rearrangements underlying these phenomena leads to the conclusion that the basic unit of inelastic shear can be understood as a microscopic two-state system. A theory of the dynamics of inelastic deformation under shear is developed from this assumption and compared to the simulation results. It is further conjectured that an understanding of this non-linear response sheds light on our recent simulations of brittle/ductile fracture behavior in computational models of amorphous solids as well as dynamic fracture experiments performed by others on polymeric systems in the laboratory.

9:10 AM

GOVERNING FACTORS OF THE SPALL FRACTURE: *Gennady I. Kanel*¹; ¹High Energy Density Research Center, IVTAN, Izhorskaya 13/19, Moscow, Russia 127412

Data on the resistance to spall fracture of a number of metals and alloys as a function of peak shock stress, load duration and orientation, material structure, and temperature are analyzed to determine the factors that govern metal fracture in uniaxial dynamic tensile fracture. It is shown that the dynamic strength is a structure-sensitive parameter. In commercial polycrystalline alloys fracture is nucleated at relatively large nonuniformities of the parent material. With an increase in the tension rate, the strength growth significantly and the excess stresses associated initiate fracture at increasingly smaller and more numerous damage nucleation sites. Initiation of the fracture on smallest defects which occurs in single crystals requires much greater stresses than that on grain boundaries, inclusions and other relatively coarse defects of polycrystalline alloys. When the spall thickness is less than the grain size, both in-granular and inter-granular strength can be recorded. The microscopic defects generated by shock compression do not appreciably affect the spall strength of commercial alloys. The dynamic strength of steels increases as a result of both quenching and the reversible high-pressure polymorphous transformation. The spall tests determine most fundamental bulk strength properties of materials. Besides short load duration, spalling conditions are characterized by small deformation at stressed states close to three-dimensional tension. Neither the surface of the body nor isolated coarse defects contribute to the main development of the spall fracture. A relationship between the yield stress, strain hardening, and the bulk strength indicates the material toughness. Due to that, the spall tests may be considered as a promising tool to characterize the materials.

9:30 AM

ROOM-TEMPERATURE DUCTILITY IN FINE-GRAINED ALUMINUM ALLOYS AT DYNAMIC STRAIN RATE: *Toshiji Mukai*¹; Koichi Ishikawa¹; Yoshihira Okanda¹; Kenji Higashi²; ¹Osaka Municipal Technical Research Institute, Mechanical Engineering Dept., 1-6-50 Morinomiya, Joto-ku, Osaka 532-8553 Japan; ²Osaka Prefecture University, Dept. of Metallurgy and Materials Science, 1-1 Gakuencho, Sakai, Osaka 593 Japan

Recently, microstructure of aluminum can be refined less than 1 micron by the advanced technique, such as mechanical alloying, crystallization from amorphous powder, consolidation of nano-crystalline powder, physical vapor deposition and/or severe plastic deformation. It has been extensively investigated mainly by compression and hardness tests that the strength of the fine-grained aluminum alloy increased with refining grain size. Very limited data on the tensile properties, however, has been reported for the fine-grained materials. To investigate the tensile properties at dynamic strain rates are very important for the application of these fine-grained materials to structural components. In this study, several fine-grained materials have been examined using a modified Hopkinson bar method for tensile tests. As a result, the fracture mechanism of the examined materials can be divided into two groups. The alloy strengthening by high volume fraction of second-phase particles exhibits almost brittle fracture, while the alloy strengthening by solute atoms or nano-dispersoids fractures with the ductile dimple. The ductile-fractured material exhibits the enhanced ductility with increasing strain rate, while the brittle-fractured one does the reduced ductility. The final goal of this study is to determine an optimum microstructure for high speed impact performance.

9:50 AM

SELF-SIMILARITY EFFECTS IN DYNAMIC FRACTURE OF MATERIALS: *Oleg Naimark*¹; ¹ICMM, Russian Academy of Science, Phys.Foundation of Strength, 1 Acad.Korolev Str., Perm 614013 Russia

Self-similar solid response on impact was established under spall failure of PMMA and ultraporcelain as stress amplitude independent response on fracture time ("dynamic branch" effect) with specific failure pattern (numerous mirror zones with different mean radius at spall surfaces). This result was predicted theoretically as resonance excitation of localised failure structures which subject failure kinetics under transition from damage to fracture. These structures represent areas with concordant blow-up microcrack growth kinetics and corre-

spond to eigen-function spectrum of self-similar solution. The existence of travelling structures allows the explanation of superdeep penetration effect for high-speed collision of particle flux with hard target. Eigenfunction spectrum is phase coordinates of strange attractor that determines the transition from steady-state to branching regime of crack propagation dynamics.

10:10 AM BREAK

10:20 AM

QUANTITATIVE DESCRIPTION OF DAMAGE EVOLUTION IN DUCTILE FRACTURE OF TANTALUM: *Jesus M. Rivas*¹; ¹Los Alamos National Laboratory, Materials Science and Technology Division, MST-8, MS G755, Los Alamos, NM 87545 USA

A systematic analysis of the evolution of several microstructural parameters during spallation of tantalum has been conducted in an effort to incorporate quantitative descriptors of damage accumulation into a micromechanical model of ductile fracture. In this paper, we describe a methodology to quantify important microstructural parameters of the ductile fracture process such as void size and void density. Four different samples of commercially pure and highly pure tantalum were incipiently spalled and recovered. Image analysis and optical profilometry techniques were used to study the recovered samples. The obtained results include statistical analyses of the void diameters, void volume fraction and its distribution with respect to the primary spall plane.

10:40 AM

QUANTITATIVE MICROSTRUCTURAL DAMAGE EVOLUTION IN COPPER UNDER SPALLATION CONDITIONS: *W. Richards Thissell*¹; *Anna K. Zurek*¹; *Jesus M. Rivas*¹; *Davis L. Tonks*²; *Robert S. Hixson*³; ¹Los Alamos National Laboratory, MST-8: Structure-Property Relationships, G755, Los Alamos, NM 87545 USA; ²Los Alamos National Laboratory, XNH: Nuclear and Hydrodynamic Applications, B216, Los Alamos, NM 87544 USA; ³Los Alamos National Laboratory, DX-1: Detonation Science and Technology, MS:P952, Los Alamos, NM 87545 USA

This paper quantitatively describes the damaged microstructures resulting from recovered incipient spallation experiments performed on 10100 OFHC. The starting material was annealed in vacuo at 600°C for one hour and has a mean grain size of 34 μm for all of the tests. We performed all of the experiments in a gas gun with identical flyer and target geometries, but at varying flyer plate velocities. Hence, the significant variable is the compressive and tensile stress amplitudes. The quantitative analysis combines two-dimensional image analysis with optical profilometry of metallographic sections cut normal to the incipient failure surface. The quantified microstructural parameters include void size (true and cross-sectional), position, aspect ratio, void clustering, void linking, and porosity versus position. We present statistical analyses of these parameters and describe the effect of stress pulse amplitude on the evolution of these parameters.

11:00 AM

INVESTIGATING THE ROLE OF VOID DAMAGE CLUSTERING IN DYNAMIC FRACTURE: *Michael J. Worswick*¹; *A. Keith Pilkey*¹; *Ahmed Kamal Kamel*¹; *Gang Liu*¹; *Pierre Pelletier*²; *Patrick J. Gallagher*³; *Denis Nandlall*⁴; ¹Carleton University, Mechanical and Aerospace Engineering, 1125 Colonel By Drive, Ottawa, Ontario K1S 5B6 Canada; ²SNC Industrial Technologies, 5 Montee des Arsenaux, LeGardeur, Quebec J5Z 2P4 Canada; ³Defence Research Establishment Suffield, Box 4000, Medicine Hat, Alberta T1A 8K6 Canada; ⁴Defence Research Establishment Valcartier, 2459 Pie XI Blvd. North, Courcelette, Quebec G0A 1R0 Canada

The role of void damage clusters during dynamic fracture is investigated on the basis of an experimental programme which combines high rate material testing with quantitative metallographic analyses of tested specimens. The nominal strain rate regime in this study is

2000/s, provided by a tensile split Hopkinson bar (TSHB) facility at Carleton University. Two very dissimilar materials are considered, namely free-machining brass and Armco iron, thereby facilitating an examination of clustering effects in the presence of many (brass) versus few (iron) void nucleation sites. For each material, several interrupted TSHB tests are conducted to obtain deformed microstructures at various strain states just below that of fracture. Spatial tessellation techniques are subsequently applied to large-scale high-resolution digital images of the potential fracture region in order to establish distinguishing characteristics of void clustering as damage evolves with applied strain. The significance of these stereological clustering measures in the prediction of dynamic fracture is supported by the results of a complementary numerical model which employs a characteristic damage length scale in its formulation.

11:20 AM

DYNAMIC DEFORMATION AND FRACTURE OF SHIP STEEL: *Gersom Pape*¹; ¹Delft University of Technology, Laboratory of Materials Science, Rotterdamseweg 137, Delft, - 2628 AL The Netherlands

The dynamic deformation of steel Fe E 355 for marine applications, along with the dynamic fracture process is the subject of this research work. For the prediction of the failure behaviour of dynamically loaded structures, several experiments are being done to examine the dynamic and static fracture characteristics for this kind of steel. For this purpose different geometries of tensile specimen will be considered to investigate the influence of the stress-triaxiality on fracture initiation. Finite element calculations will be used to determine the stress-triaxiality in the plastically deforming specimen. In these calculations an appropriate local fracture criterion predicts the onset of failure. To investigate the influence of the strain rate on the fracture initiation process, the experiments are being carried out at several strain rates, varying between approximately $1e-4$ and $1e+2$ per second. Use is made of a servo-hydraulic high rate single shot testing machine. Results of tensile tests at low and high strain rates will be presented along with some numerical results of calculations of the experiments.

11:40 AM

SPALL STRENGTH OF METALS AT HIGH TEMPERATURE AND HIGH PRESSURE: *Andrey Bogach*¹; ¹Institute of Chemical Physics, Combustion and Explosive, Institutskiy pr. 14, Chernogolovka, Moscow 142432 Russia

The spall strength of metals is known to have a threshold dependence on temperature [1] and peak pressure [2] and depends on the strain rate in unloading part of shock pulse as power function with small power index $1/m$ [1]. It is shown in present work that the power function take place for viscoplastic material, in which viscose stress depends on the strain rate as power function with power index $1/m$, and is directly connected to dynamic resistance of material to dislocation motion. The viscoplastic dissipation of deformation energy results to localized heating in vicinity of pore. In assuming of localized melting for these viscoplastic material the expressions for threshold temperature and for dependence of spall strength on temperature were received. According to the solution the threshold temperature and the spall strength have the same dependence on the stress gradient in shock pulse. In assuming that shock-induced and initial heating is equivalent the model gives the spall strength dependence on peak pressure. The dislocations density in vicinity of pore was estimated from experimental results of the threshold temperature and the spall strength for Al,Mg,Mo. 1. Kanel G.I.,Razorenov S.V.,Bogach A.A.,Utkin A.V.,Fortov V.E., J.Appl.Phys.79,8310-8317(96). 2. Kanel G. I., Razorenov S.V., Utkin A.V., Grady D.E. ShockCompression of condensed metter, Seattle, USA, 1995

EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Stress & Microstructure; 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; Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Dept.; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Wednesday AM Room: International Ballroom
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Peter Voorhees, Northwestern University

8:30 AM INVITED PAPER

THERMODYNAMICS AND STRESS: *Francis C. Larchie*¹; ¹University Montpellier, Materials Science and Technology, GDPC - Case 26, Montpellier, Cedex 5 34095 France

Our work with J.W. Cahn on the thermodynamics of stressed solids is first put in an historical perspective through a brief review of the literature since J.W. Gibbs. It is shown that the basic thermodynamics is the same; the various theories differ mostly through the models used to describe the physics of the system. The assumptions of our model are then examined in detail, in order to test its merits and limitations in representing the behavior of real solid materials. Possible extensions are presented or suggested. We also consider the question of macroscopic behavior (at a scale large compared to the microstructure) and see how this change of scale can be translated into the thermodynamic results.

9:15 AM INVITED PAPER

STRESS EFFECTS IN MICROSTRUCTURE EVOLUTION: *Perry H. Leo*¹; ¹University of Minnesota, Aerospace Engineering, 107 Akerman Hall, 110 Union St. SE, Minneapolis, MN 55455 USA

We study the effects of elastic stress on the evolution of precipitate-matrix microstructures in binary alloys. The precipitate and matrix are both assumed to obey anisotropic linear elasticity, although they can have different elastic constants and possibly different symmetries. Stresses arise from misfit between the phases and from applied loads. Both sharp interface (boundary integral) and diffuse interface (Cahn-Hilliard) methods are used to track the evolution of the precipitates. Results from both methods show that elastic stress has a strong influence on precipitate shapes and interactions as well as on the overall growth and coarsening behavior of the alloy. Further, relatively small difference in the elastic constants of the phases can play an important role in this evolution, suggesting that modeling elastic inhomogeneities is crucial in simulating the microstructure of alloys such as nickel based superalloys.

10:00 AM BREAK

WEDNESDAY AM

10:20 AM INVITED PAPER

GRAIN BOUNDARY FACETING AND DIGM: *Duk Yong Yoon*¹; ¹Korea Advanced Institute of Science and Technology, Materials Science and Engineering, 373-1 Kusungdong, Yuseonggu, Taejeon 305-701 Republic of Korea

Grain Boundaries in a variety of crystals undergo faceting-defaceting transition at critical temperatures which strongly depend on the impurity content. In a relatively pure polycrystalline Ni, for example, all grain boundaries are faceted at temperatures below 0.8 T_m (where T_m is the melting point) in a carburizing atmosphere. According to the analysis of Herring, Frank, Cabrera, and Cahn, any grain boundary can be faceted if the grain boundary energy varies strongly with the inclination angle. A faceted grain boundary can exhibit zig-zag migration behavior during DIGM, because the direction of DIGM is determined by the relative coherency strain energies at the boundary segments of the pair grains. Under certain conditions grain boundaries undergoing DIGM may develop faceting. Critical experiments have been performed to show that the driving force for the migration of grain boundaries and intergranular liquid films during alloying (and dealloying) and discontinuous precipitation is the diffusional coherency strain energy. The theoretical analysis of Cahn, Fife, and Penrose is consistent with these observations.

11:05 AM

PHASE FIELD COMPUTATIONS USING AN ADAPTIVE GRID TECHNIQUE: *N. Provatas*¹; *N. Goldenfeld*¹; *Jon Dantzig*¹; ¹University of Illinois, Mechanical and Industrial Engineering, 1206 West Green Street, Urbana, IL 61801 USA

In recent years, the phase field technique has been developed to model evolution of solidification microstructures. In this method, the liquid-solid interface is modeled as a diffuse region whose thickness is characterized by an order parameter, known as the phase field. One of the difficulties encountered when applying the phase field method is the conflicting requirements of high resolution needed to successfully capture the physical phenomena at the interface, and the simultaneous need to fully resolve the diffusion field ahead of the advancing front. This has led to the use of very dense grids, with correspondingly long computation times. Further, the calculations have been limited to the regime of high growth rates, where the diffusion field is relatively small. In this work, we describe an adaptive gridding procedure for solving the phase field equations, where high resolution is available near the interface, and more appropriate grid dimensions are used to resolve the diffusion field.

11:10 AM

COMPUTER SIMULATIONS ON THE PHASE TRANSFORMATION IN REAL ALLOY SYSTEMS BASED ON THE PHASE FIELD METHOD: *Toru Miyazaki*¹; *Toshiyuki Koyama*¹; ¹Nagoya Institute of Technology, Dept. of Materials Science and Engineering, Gokisocho, Showa-ku, Nagoya 466 Japan

The kinetic simulation based on the non-linear evolution equation become very powerful method in fundamental understanding the dynamics of phase transformation with the recent remarkable development of computer. In the present study, we calculate the dynamics of microstructure changes in real alloy systems, such as Fe-Al-Co, Fe-Si-V, Fe-Mo, Fe-Cr and Al-Zn based on the phase field method which is a new methodology to simulate the microstructure development by combining the Cahn-Hilliard diffusion equation and Cahn-Allen equation. The atomic interaction parameters, elasticity, and mobility are assumed to depend on local order parameters (composition, degree of order, etc) to calculate the phase transformation which is consistent with the equilibrium phase diagram of the actual alloys. Time dependent morphological changes of the microstructure (the formation of modulated structure by spinodal decomposition, strain induced morphological changes of precipitates, the interaction between the order-disorder phase transition and phase separation, discontinuous precipitation, and stable phase precipitation after the metastable phase decomposition, etc) will be demonstrated. The results simulated are quantitatively in good agreement with the experimental ones in the real alloy systems.

11:15 AM

INTERFACIAL STUDIES OF TERNARY ALLOYS: *Monica Olvera de la Cruz*¹; ¹Northwestern University, Dept. of Materials Science and Engineering, Evanston, IL 60208-3108 USA

We study interfaces of A-B-C ternary alloys decomposed into two and three phases. The effect of the gradient energy coefficients on the interface profiles of ternary alloys is examined. The interface profiles of ternary alloys are obtained numerically solving the steady-state solutions of the nonlinear spinodal decomposition equations. We also determine the composition profiles solving the two Euler-Lagrange equations resulting from minimizing the interfacial energy.

11:20 AM

THERMAL DRAG OF THE ANTIPHASE DOMAIN BOUNDARY MOTION: *Alexander Umantsev*¹; ¹Saint-Xavier University, Science Department, 3700 West 103rd Street, Chicago, IL 60655 USA

Antiphase domain boundary (APB) is a region in materials where ordering of atoms changes from one structural variant to another. Due to the surface tension, APB's constitute a network of structural defects which is in global disequilibrium with the crystal. Cahn-Allen's analysis of the structural coarsening of this network [Acta Metall, 27, 1085 (1979)] shed the light on the nature of the driving force in this problem: the free-energy change, if the material stays isothermal. We explore the influence of the internal energy transport on the dynamics of such boundary in the framework of the Onsager theory of linear response. The internal energy transport entails temperature humps in the transition regions and causes a drag effect as a consequence of the driving force reduction, implying that coarsening of the APB structure is not possible without heat conduction in the material. An evolution equation that takes into account the finite thermal conductivity is derived. Experimental setup to reveal the thermal drag is suggested.

11:25 AM

SIMULATING MICROSTRUCTURAL EVOLUTION IN MULTI-PHASE AND MULTI-DOMAIN MATERIALS USING PHASE FIELD METHOD: *Yunzhi Wang*¹; ¹The Ohio State University, Materials Science and Engineering, 2041 College Road, Columbus, OH 43210 USA

Many technologically important material systems are inherently heterogeneous. Structurally, they are multi-phase and multi-variant/domain aggregates whose morphology determines the important physical and mechanical properties. In this presentation, recent development of the continuum phase field method in simulating microstructural evolution in martensitic crystals and Ni-based superalloys will be discussed. The simulation models explicitly take into account the interactions between different orientation variants of the martensitic phase and the different antiphase domains of the L12 ordered phase in the elastic strain field generated by lattice misfit. Results of both 2D and 3D simulations illustrating the effect of these interactions on the kinetic and morphology of the multi-phase and multi-variant/domain microstructure development in these systems will be discussed. Also, the recent adoption of the phase field method to simulating the microstructural evolution and electrical conduction of ceramic thin film sensors will be presented. In this approach, detailed microstructural features developing during sintering, which are simulated using the phase field method, have been directly used in the mesoscopic resistor-lattice model for electrical conductivity calculations. Combination of these methods provides a direct link between the critical microstructural features developing during thermal processing and the electrical conducting behavior of the material. This work is supported by the National Science Foundation.

11:30 AM POSTER VIEWING

FATIGUE BEHAVIOR OF TITANIUM ALLOYS: Environmental and Surface Aspects of Fatigue

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

Wednesday AM Room: Athens/Berlin
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: P. Bowen; A. P. Woodfield

8:30 AM KEYNOTE

EFFECT OF ENVIRONMENT ON HIGH-TEMPERATURE FATIGUE OF IMI 834: O. Schauerte¹; A. Gysler¹; G. Lutjering¹; S. Mailly²; Y. Chabanne²; C. Sarrazin-Baudoux²; J. Mendez²; J. Petit²; ¹Technische Universität Hamburg-Harburg, Eissendorfer Str. 42, Hamburg, FRG 21073 Germany; ²L.M.P.M. - E.N.S.M.A., Site du Futuroscope - Chasseneuil-du-Poitou, B.P. 109, Futuroscope Cedex 86960 France

The fatigue properties of the Ti-alloy IMI 834 at high temperature (500°C) will be compared in air as an aggressive environment and in vacuum as an inert reference environment. This comparison includes the environmental effect on fatigue crack initiation in the LCF regime, as well as on the fatigue crack propagation behavior of self-initiated microcracks and in addition on the propagation behavior of long through cracks (macrocracks). Two different microstructural conditions will be considered, pure lamellar structures and bimodal microstructures, consisting of equiaxed primary α -phase in a transformed lamellar matrix. The characterization of the macrocrack propagation behavior includes the effects of mean stress variations and crack closure effects for both environmental conditions. The discussion will be focused on the environmental effects on the crack propagation mechanisms by detailed fracture surface studies and on variations in the through-thickness crack front profile appearances. The usually observed pronounced reduction in the resistance against high temperature fatigue crack nucleation as well as crack propagation in the aggressive air environment in comparison to vacuum will be discussed on the basis of hydrogen embrittlement caused by the water vapor in air environment.

9:00 AM INVITED PAPER

ENVIRONMENTAL INTERACTIONS IN HIGH TEMPERATURE FATIGUE CRACK GROWTH OF TITANIUM ALLOYS: J. Petit¹; C. Sarrazin-Baudoux¹; Y. Chabanne¹; G. Lutjering¹; G. Gysler¹; O. Schauerte¹; ¹L.M.P.M. - E.N.S.M.A., Site du Futuroscope - Chasseneuil-du-Poitou, B.P. 109, Futuroscope Cedex 86960 France

Titanium alloys are interesting to aerospace designers because of their low density, high specific strength and corrosion resistance. They are often used under cyclic conditions in aggressive environments such as moist air and at more and more elevated temperature levels. The present paper is addressing a study of the specific influence of atmospheric gaseous species (i.e. water vapor and oxygen) at 500°C on the fatigue cracking behavior of Ti6246 and IMI 834 Titanium alloys. A first comparison of the crack growth rates in air and in high vacuum confirms the existence of a detrimental effect of ambient environment which becomes more accentuated at high temperature. Complementary tests carried out at 500°C under selected environmental conditions as dry air, dry Argon and controlled low pressures of water vapour, demonstrate that the enhancement of the

fatigue crack growth rates in the near-threshold area observed in active environments in comparison with high vacuum is clearly related to the presence of water vapor even at very low partial pressure of about 1Pa. However comparison between growth data under atmospheric pressure of dry air and dry Argon containing few ppm of water vapor, suggests a complex interaction between oxygen and water vapour. Scanning electron microscope observations are investigated in order to identify the failure modes with respect to the microstructure of the specimens, and to precise the respective role of water vapor embrittlement and of oxidation. The contribution of crack closure is evaluated and the respective role of roughness induced closure and oxide wedging is discussed. A detailed analysis of the fatigue crack propagation resistance of the Ti6246 alloy is conducted at various loading frequencies ranging from 35Hz to 5x10⁻³ Hz. According to environment, loading conditions and test frequency, the respective contribution of potential time dependent damage cracking mechanisms assisted by water vapour, i.e. corrosion-fatigue and creep-fatigue, is discussed.

9:20 AM INVITED PAPER

EFFECT OF MECHANICAL SURFACE TREATMENTS ON FATIGUE PERFORMANCE OF TITANIUM ALLOYS: L. Wagner¹; ¹Technical University of Brandenburg at Cottbus, Department of Materials Technology, Postfach 101344, Cottbus 03013 Germany

Mechanical surface treatments such as shot peening, roller-burnishing and deep-rolling are widely used to improve the fatigue performance of titanium alloys. Generally, these treatments induce high dislocation densities in near-surface regions. Due to the local plastic deformation, residual stresses are developed and the surface topography is changed. In this overview, the effect of the various process parameters, e.g., Almen intensity, burnishing pressure and rolling force on the change in surface properties and resulting fatigue performance will be presented for the various titanium alloy classes, i.e., α , ($\alpha+\beta$) and β alloys. Results will be compared with electrolytically polished references. The different response of the various titanium alloy classes to mechanical surface treatments will be discussed on the basis of the degree of the cyclic stability of process induced residual compressive stresses and their influence on the microcrack growth.

9:40 AM

ROLLER-BURNISHING TO IMPROVE FATIGUE LIFE IN BETA TITANIUM ALLOYS: A. Berg¹; A. Drechsler¹; L. Wagner¹; ¹Technical University of Brandenburg at Cottbus, Department of Materials Technology, Postfach 101344, Cottbus 03013 Germany

Roller-burnishing is a common procedure for improving the fatigue performance of structural components which have a rotational symmetry, such as axles and shafts. Compared to shot peening, greater depths of induced plastic deformation combined with a much smoother surface finish can be achieved. In the present work, the effect of roller-burnishing on the change in surface layer properties and the resulting fatigue performance was studied on the metastable β titanium alloys Ti-10V-2Fe-3Al and Ti-3Al-8V-6Cr-4Mo-4Zr. On both alloys, suitable thermomechanical treatments were performed to cover conditions with low, intermediate and high strength levels. Hour-glass shaped specimens were roller-burnished in a conventional lathe using a hydraulic device by which a hard metal ball (6 mm diameter) is pressed on the surface of the specimen. For optimizing the fatigue response working pressures ranging from 25 to 400 bar were used. Induced dislocation densities and residual stresses were measured by X-ray diffraction techniques. Fatigue tests on the various roller-burnished conditions as well as on electrolytically polished references were performed in rotating beam loading (R = -1). Fatigue results will be discussed in terms of the effects of induced dislocation density and residual compressive stress on fatigue crack nucleation and microcrack growth.

10:00 AM

FATIGUE CRACK GROWTH OF TITANIUM ROTOR ALLOYS IN VACUUM AND AIR: R. Craig McClung¹; B. H. Lawless¹; M.

Gorelik¹; ¹Southwest Research Institute, Manager, Materials Integrity, P.O. Drawer 28510, San Antonio, TX 78228-0510 USA

A probabilistic damage tolerance design code for life management of aircraft turbine engine rotors is being developed under a major FAA grant (95-G-041), with an initial focus on fatigue cracking at hard alpha defects in titanium. Since HA defects are usually subsurface, any resulting cracks are embedded and hence isolated from the atmosphere (i.e., vacuum-like) for at least some of their life. FCG tests are conducted in vacuum at various temperatures for Ti-6-4 and Ti-6-2-4-2 rotor alloys, and results are compared with existing air data. The new design methodology is probabilistic in order to address uncertainties associated with the size, shape, and rate of occurrence of HA defects, as well as uncertainties in rotor inspection, stresses, and material properties. Replicate FCG data for titanium rotor alloys are analyzed statistically to characterize the FCG rate variability that could be significant for life calculations.

10:20 AM BREAK

10:40 AM

MODELING THE DAMAGING EFFECTS OF GASES ON THE FATIGUE CRACK GROWTH OF AN $\alpha+\beta$ TITANIUM ALLOY: *Stephen W. Smith*¹; Robert S. Plascik¹; ¹NASA-Langley Research Center, MS 188E, NRC Resident Research Associate, Hampton, VA 23681-0001 USA

The effects of air, oxygen, nitrogen, and water vapor on the fatigue crack growth behavior of an $\alpha+\beta$ titanium alloy (Ti 6-22-22, Ti - 6 Al - 2 Zr - 2 Cr - 2 Sn - 2 Mo) were evaluated to determine the mechanisms resulting in environmentally assisted fatigue. Tests were performed in laboratory air, ultrahigh vacuum, and high purity gaseous environments at various partial pressures for temperatures ranging from room temperature to 177°C. Accelerated fatigue crack growth rates (five to ten times greater) have been measured in laboratory air when compared to ultrahigh vacuum. Oxygen and water vapor are identified as the damaging species present in laboratory air. Fatigue crack growth tests were performed at constant ΔK , frequency, and stress ratio in high purity oxygen and water vapor environments at pressures ranging from ultrahigh vacuum ($\sim 9 \times 10^{-8}$ Pa) to atmospheric pressure at sea level ($\sim 1 \times 10^5$ Pa). From these tests, distinct transitions in the fatigue crack growth behavior have been observed. These transitions are related to three different mechanisms which affect fatigue crack growth at various pressure ranges. In low pressure oxygen and water vapor, an adsorption assisted damage process is suggested. This adsorption assisted process results in increasing fatigue crack growth rate with increasing pressure. A critical pressure is reached where a monolayer of adsorbed species is produced during each fatigue cycle. For pressures greater than the critical pressure, a crack-tip chemisorbed film and/or oxide is produced resulting in a decrease in fatigue crack growth rate with further increase in pressure. While the formation of this film results in decreased fatigue crack growth rates compared to the adsorption assisted regime, fatigue crack growth rates are accelerated compared to ultrahigh vacuum rates. A third damage mechanism is suggested for exposure to water vapor; here, increasing crack growth rate with increasing pressure is consistent with a hydrogen embrittlement mechanism.

11:00 AM

INTERFACE BETWEEN FATIGUE, CREEP, AND STRESS-CORROSION CRACKING ON Ti6246 TITANIUM ALLOY: *C. Sarrazin-Baudoux*¹; *Y. Chabanne*¹; *J. Petit*¹; ¹L.M.P.M.-E.N.S.M.A., Site du Futuroscope-Chasseneuil-du-Poitou, B.P. 109, Futuroscope Cedex 86960 France

Failure of structural materials due to cracking under coupled actions of mechanical stressing and environmental attack remains a safety and economical problem despite the effort that has been devoted to understand the cracking phenomena induced by stress-corrosion, corrosion-fatigue or creep-fatigue. Titanium alloys for aerospace applications are often used under a combination of cyclic and constant amplitude loading in aggressive environments such as moist

air, and at elevated temperature levels (up to 600°C). Then a well defined characterization of these alloys is required in order to ensure a good damage tolerance. The present paper is addressing a study of the influence of the mean stress on the fatigue crack growth behavior of a Ti6246 alloy, and is specially concentrated on the near-threshold condition at room temperature and 500°C. Threshold tests are performed at frequencies of 0.1 to 35 Hz and at a constant K_{max} level, which means increasing R ratio with decreasing ΔK ensuring the absence of closure during the threshold procedure. At room temperature and very low ΔK , it is shown that for a critical K_{max} level of about 70% to 80% of the fracture toughness, no threshold for crack propagation is reached and for ΔK range lower than 1.5 MPa m the growth rates appear to become time dependent instead of cycling dependent supporting a creep governing mechanism. At 500°C, tests performed in air and in humidified argon support a detrimental effect of water vapor consistent with a corrosion-fatigue mechanism in the mid-rate range and an additional process of stress-corrosion in the near threshold area. According to environment, loading conditions and test frequency, the respective contribution of the two involved competitive cracking mechanisms assisted by water vapor is discussed. Stress corrosion is shown to interact with corrosion-fatigue specially when the stress intensity factor range is lower than 2 MPa m and conjugated with K_{max} higher than 22 MPa m (rumpling tests). Such conditions of stress localization and environmental assistance can induce a failure by stress corrosion cracking for stress intensity level of about 40% of the fracture toughness. Scanning electron microscope observations and crack surface analysis are investigated in order to identify the failure modes and to precise the respective role of water vapor embrittlement and of oxidation with respect to the loading conditions.

11:20 AM

SLOW FATIGUE TESTING OF TITANIUM GRADE 29 IN AIR AND SEA WATER: *Lyder Atteraa*¹; *Gisle Hersvik*¹; *Haavard Solbakken*¹; ¹University of Bergen, Applied Physics and Technology, Allegaten 55, Bergen N-5007 Norway

Welded and unwelded specimens of ASTM Grade 29 titanium has been sinusoidally fatigue tested in air and seawater at 110°C at 0.2 Hz. The possible influence of seawater is completely masked by the fact that all the specimens of welded titanium, and most of the parent metal samples, had internal fracture initiations. A high maximum cyclic stress leads to a low fatigue life of the parent (unwelded) metal. Increasing the maximum stress from 75 to 85 % of the yield stress (at 110°C), the fatigue life is lowered by a factor of 3.6. Compared with the parent metal, the welded titanium has a significantly lower fatigue life. At 110°C, the fatigue life of weldmetal was reduced by ca. 93 % at the high stress level ($s_{max} = 0.85s_y$), and ca. 98 % at the low stress level ($s_{max} = 0.75s_y$). This drop in fatigue life is attributed to the presence of pores in the weld deposits. Fractographic studies indicate that the fracture initiation takes place in the material immediately surrounding a pore, with an abrupt crack formation, marking the beginning of the crack growth phase. The duration of the crack initiation phase shows large variations, whereas the growth phase duration varies little.

11:40 AM

FATIGUE OF TITANIUM GR. 29 UNDER CATHODIC "PROTECTION" IN HOT WATER: *Lyder Atteraa*¹; *Kjetil Foer*¹; ¹University of Bergen, Applied Physics and Technology, Allegaten 55, Bergen N-5007 Norway

Titanium Grade 29 has been tested in sinusoidal fatigue. The specimens were taken from a 340 mm pipe. To simulate a surface crack the specimens were notched. The results were obtained at 110°C. The testing was performed in air and in natural seawater. In water, specimens were subjected to three different corrosion potentials: Free corrosion potential, -1050 mV_{Ag/AgCl} and -1500mV_{Ag/AgCl}. The maximum nominal stress for all four series was 552 MPa (i.e. $0.75 * \sigma_y$ at 110°C) and the stress ratio was 0.1. The air testing frequency was 3 Hz, and the seawater test frequency 0.2 Hz. In each series 5 specimens were coupled in a column and tested together in

order to reduce the testing time. The post fracture examinations showed that all the fractures were initiated along the specimen surface, meaning that when submerged, seawater penetrated the crack from the start of the fatigue process. No significant reduction of the notched fatigue life due to seawater cathodic protection was found, but a tendency to a reduced fatigue life in the three seawater series was seen. The specimens experienced local plastic deformation in the notch during the fatigue testing. 10 of 21 specimens had more than one initiation site, at or close to the surface of the specimens. All fractures were transgranular and contained many cleavage-like facets near the ductile area. Fatigue testing in air showed that the fatigue life was reduced by 98% for the notched specimens compared to smooth specimens, when ignoring the elastic stress concentration factor. However, the static tensile strength was 60% higher in the notched specimens than in the unnotched specimens.

12:00 PM

EFFECTS OF TEMPERATURE AND FREQUENCY ON FATIGUE CRACK GROWTH RATE OF Ti-6Al-4V-0.1Ru IN AN OCEAN ENVIRONMENT: *Morten A Langoy*¹; *Stuart R. Stock*¹; ¹Georgia Institute of Technology, School of Materials Science and Engineering, 778 Atlantic Dr., Atlanta, GA 30332-0245 USA

The technological challenges associated with development of vessels for offshore oil production are considerable, and use of titanium for dynamically loaded production risers offers important economic advantages. However, limited experience exists with titanium risers. Recognizing that defects of some kind are inevitable in these types of components and that crack initiation will be only a minor portion of the lifetime. Hence, this study focuses on fatigue crack growth. In the offshore industry a fail safe design approach is commonly employed, and in this respect the synergic effects of temperature and frequency in ocean water is of interest. In titanium a unusual frequency cross-over effect has been reported (at low stress intensities higher frequencies promote more rapid fatigue crack growth while at high stress intensity, the converse is true). This paper reports the influence of temperature on the effect of frequency on fatigue crack growth in Ti-6Al-4V-0.1Ru.

systems. Heterogeneous nucleation occurs at the interface between the primary phase substrate and the liquid phase. During extensive thermal cycling, distinct changes in the nucleation behavior have been observed. These changes reflect transitions in the density of specific nucleation sites and can be correlated to a faceting tendency of the nucleating substrate during cycling, as observed by Scanning Electron Microscopy (SEM). Since it is expected that the development of smooth liquid-solid interfaces at the micron scale is reflected at the atomic scale in a similar structural configuration, the removal of large substrate features during consecutive cycles is anticipated to result in the removal of the more potent nucleation sites, exposing previously concealed sites. With controlled thermal cycles on drop-let samples as well as isothermal holding treatments, the nucleation temperatures can be correlated to particular sites on the primary substrate. In order to determine the catalytic potency of the investigated nucleation sites and to resolve closely spaced exotherms, a modification of the isothermal nucleation rate expression has been utilized to simulate the continuous cooling approach commonly used in heterogeneous nucleation analysis. The support of the NSF (DMR-9712523) is gratefully acknowledged.

8:55 AM

SOLUTE EFFECTS IN BAINITE IN Fe-C-X STEELS: *R. E. Hackenberg*¹; *D. G. Granada*¹; *G. J. Shiflet*¹; ¹University of Virginia, Department of Materials Science and Engineering, Thornton Hall, Charlottesville, VA 22903

Several ternary Fe-C-X steels were investigated with the goal of better understanding the role that the alloying element X plays in determining the kinetics and morphology of the bainite reaction. Particular attention has been paid to the "bay" region on the T-T-T diagram, in which the start curve passes through a minimum. The existence of a bay in Fe-C-W alloys is documented for the first time, and morphological similarities with Fe-C-Mo alloys are observed. This result is consistent with the theory that a solute drag-like effect (SDLE) accounts for the kinetic and morphological transitions seen in the bay region. The existence of SDLE in selected Fe-C-Mo alloys is tested with analytical TEM to determine the distribution of solute in and around the growth front. Particular attention is paid to the time-temperature regions (above the bay) where growth stasis is known to occur. Early results from this study will be presented, and attention will be given to the role played by carbide precipitation. This research was supported by the National Science Foundation.

9:20 AM

IDENTIFICATION OF THE SOLUTE DIFFUSION PATHS AT THE REACTION FRONT IN LAMELLAR PHASE TRANSFORMATIONS: *C. R. Hutchinson*¹; *G. J. Shiflet*¹; ¹University of Virginia, Department of Materials Science and Engineering, Thornton Hall, Charlottesville, VA 22903

Modeling of growth kinetics in lamellar phase transformations usually assumes that a high diffusivity path is employed for substitutional solute redistribution at the reaction front. Previous research in ternary Fe-C-X pearlite using analytical microscopy has not been of sufficient spatial resolution to confirm the diffusion path. In addition to ferrous pearlite transformations, the cellular transformation in alloys, such as Cu-Ti, has also been investigated. Although a precipitation reaction, the synchronous growth of the precipitate phase and depleted matrix results in a similar reaction front to that observed in pearlite. Mathematically modeling this phase transformation also incorporates boundary diffusion of the solute element and closely follows the approach for pearlite. To obtain chemical information, High Resolution Analytical Electron Microscopy has been used to critically evaluate alloy content in the various interfaces associated with these two lamellar phase transformations as a function of temperature. Concentration profiles in the vicinity of the reaction front and the interlamellar interphase boundaries will be reported. These chemical profiles will be correlated with the interfacial structure. The authors gratefully acknowledge support from the National Science Foundation.

GENERAL ABSTRACTS: Phase Transformations

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 Sizak, Inco Alloys International, Mechanical Testing, Huntington, WV 25705

Wednesday AM Room: Zurich/Tokyo
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Zi Kui Liu, Questek Innovations, Evanston, IL 60201

8:30 AM

HETEROGENEOUS NUCLEATION BEHAVIOR AS A FUNCTION OF SUBSTRATE MORPHOLOGY: *Peter G. Höckel*¹; *John H. Perepezko*¹; ¹University of Wisconsin - Madison, Materials Science and Engineering, 1500 Engineering Drive, Madison, WI 53706 USA

The droplet emulsion technique (DET) and Differential Scanning Calorimetry (DSC) have been used to investigate the undercooling and nucleation behavior of powder samples from several binary alloy

9:45 AM

A KINETICS MODEL FOR CONTINUOUS COOLING TRANSFORMATION AND ITS APPLICATION TO PLAIN CARBON STEELS: *Choo Se Don*¹; Choo Wung Yong¹; ¹Pohang Iron & Steel Co., Technical Research Labs, Pohang P.O.Box 36.1, Koedong-dong, Nam-ku, Pohang-shi, Kyungbuk 790-785 South Korea

A new model was suggested to describe the kinetics of the austenite-to-ferrite phase transformation during continuous cooling. It was based on the simplified Fe-C phase diagram and some thermodynamic theories were considered. The final equation derived was as follows; $\ln(-\ln(1-X))=K\alpha t + \text{constant}$ where, X is the transformed volume fraction, K is a kinetic parameter, t is the time and α is the cooling rate. It is somewhat different from the Avrami type equation which is for isothermal transformation behaviors. Using a dilatometer the phase transformation has been investigated in a kind of plain steels with compositions C:0.05~0.2, Mn:0.5~2.0, Ti:0.01 under the various cooling rate 0.2°C/sec up to 30°C/sec. The Experimental data were statically treated and the kinetic parameter K in the above equation was revealed to depend only on the chemical compositions. The predicted time-transformation curves using the above equation and the measured ones were compared and reasonably good agreement was obtained. The equation has been integrated into a full microstructural model which has been applied to prediction of mechanical properties. It was found that the predicted values of tensile strength of plates processed under various manufacturing were in reasonable agreement with the measured ones.

10:10 AM

PHASE EQUILIBRIA AND REACTIONS IN THE Ni-Bi SYSTEM: *Chengheng R. Kao*¹; M. S. Lee¹; C. M. Liu¹; ¹National Central University, Dept. of Chemical Engineering, Chungli, Taiwan ROC

The phase equilibria and the reactions between Ni and liquid Bi were studied. It was found that the only intermetallic compound formed in the reactions was NiBi₃. The other intermetallic compound NiBi, which is thermodynamically stable at these temperatures, did not form. Reaction at 300 C produced a thick reaction zone, which is a two-phase mixture of NiBi₃ needles dispersed in Bi matrix. The thickness of the reaction zone increased rapidly with reaction time, reaching 400 microns after 360 minutes. Reactions at 360 and 420 C produced very thin reaction zones, and the major interaction was the dissolution of Ni into liquid Bi. Reaction at 480 C produced extremely thin reaction zone, and the dissolution of Ni into liquid Bi was very fast and was the major interaction. It is proposed that the formation of the reaction zone is controlled by two factors: the solubility limit and the diffusivity of Ni in liquid Bi. Small diffusivity and small solubility limit, i.e. lower temperature, tend to favor the formation of thin reaction zone. In addition to the NiBi₃ formed within the reaction zone, NiBi₃ also formed outside the reaction zone as long needles with hexagonal cross-section. It is believed that these NiBi₃ needles formed during the solidification of liquid Bi.

10:35 AM BREAK

10:50 AM

A TEM STUDY OF THE EFFECT OF COHERENCY STRAIN ON THE COARSENING RATE OF THE GAMMA' PRECIPITATES IN NICKEL BASE SUPERALLOYS: *Michael J. Pollard*¹; Steve L. Weber¹; *Haydn H. Chen*¹; ¹University of Illinois at Urbana-Champaign, Materials Science and Engineering, 1304 W. Green, Urbana, IL 60565 USA

The objective of this study was to determine the coarsening rate of γ' precipitates in several alloys of the Ni-Al-Si system in the composition region near complete coherency in order to observe the effect of coherent misfit strain on coarsening rates. Samples with total nickel concentration fixed at 88 at%, but with varying Al/Si ratio, were aged at 550°C and 600°C for 2, 4, 6, 8, 10, and 12 hours. The samples were then viewed in a TEM in the dark field of a superlattice spot to see the precipitate structure. The micrographs were scanned and analyzed to obtain the average size of the precipitates versus time for each composition and temperature, and there-

fore the coarsening rate. The coarsening rates for these compositions were compared to the results of the study of the same alloys by Muralidharan and Chen (SANS), who demonstrated that small misfit strains in either the positive or negative direction caused increases in coarsening rate. The TEM study was able to reproduce the coarsening rate increases on either side of zero misfit.

11:15 AM

SOLIDIFICATION PROCESSING OF HIGH-TEMPERATURE Mo-B-Si ALLOYS: *Ridwan Sakidja*¹; H. Sieber¹; G. Wilde¹; H. Perepezko¹; ¹University of Wisconsin-Madison, Dept. of Materials Science and Engineering, 1509 University Ave., Madison, WI 53705 USA

Some of the microstructural options available in high-temperature Mo-B-Si alloys have been identified in an examination of different solidification processing treatments. In-situ intermetallic matrix composites consisting of Mo solid solution phase dispersed in a Mo₅SiB₂ (T₂) intermetallic phase were produced using arc-casting (AC) and rapid solidification processing (RSP). In conventionally cast Mo-B-Si samples, significant solidification segregation involving the formation of additional high melting phases such as MoB and Mo₅Si₃ was found. These phases showed a very slow dissolution rate even at temperatures as high as 1600C. From the arc-cast sample, the stable (Mo + T₂) two-phase alloys could be attained only after a prolonged subsequent annealing treatment at high temperature. In contrast, the same Mo-B-Si samples produced by means of RSP such as splat-quenching (SQ) and drop-tube processing (DTP) yielded a homogeneous two-phase microstructure. The in-situ formation of the two-phase microstructure directly from the melt allows for new options in the microstructural design of Mo-B-Si alloys. The advantages of using RSP for solidification processing of high temperature alloys will be discussed. In addition, a Mo precipitation reaction that develops in a T₂ phase upon subsequent annealing provides an additional means of microstructural control. The support of AFOSR (F49620-96-1-0286) is gratefully acknowledged.

11:40 AM

EXAMINATION OF SPINODAL DECOMPOSITION USING ANALYTICAL TRANSMISSION ELECTRON MICROSCOPY: *K. J. Doherty*¹; D. J. Li²; G. J. Shiflet¹; S. J. Poon²; ¹University of Virginia, Department of Materials Science, Thornton Hall, Charlottesville, VA 22903 ; ²University of Virginia, Department of Physics, McCormick Road, Charlottesville, VA 22901

The spinodal reaction is commonly observed as a precursor to devitrification of bulk amorphous metal alloys. An objective of this study is to re-examine spinodal decomposition within the miscibility gap in the Nb-Zr system using high-resolution analytical transmission electron microscopy with electron energy loss spectroscopy and small probe energy dispersive spectroscopy. These advanced techniques are employed to physically measure composition profiles along the spinodally transformed <100> directions as a function of time and temperature with the objective of forming three dimensional chemical maps. Following a discussion of the binary alloy results, this procedure is expanded to study the phase separation in a 5-component metastable bcc (β) alloy Ti₆₅Cr₁₃Cu₁₆Mn₄Fe₂ where the spinodal reaction also occurs. The authors acknowledge the Army Research Office for their support.

12:05 AM

A NEW ANALYSIS FOR THE DETERMINATION OF TERNARY INTERDIFFUSION COEFFICIENTS FROM A SINGLE DIFFUSION COUPLE: *Mysore A Dayananda*¹; *Yong-Ho Sohn*¹; ¹Purdue University, School of Materials Engineering, 1289 MSEE Building, West Lafayette, IN 47907 USA

Concentration profiles that develop in a ternary diffusion couple during an isothermal anneal can be analyzed directly for average ternary interdiffusion coefficients. A new analysis is presented for the determination of average values for the main and cross interdiffusion coefficients over selected regions in the diffusion zone from an

integration of interdiffusion fluxes, which are calculated directly from experimental concentration profiles. The analysis is applied to selected isothermal diffusion couples investigated with alpha(fcc) Cu-Ni-Zn alloys at 775°C, beta (bcc) Fe-Ni-Al alloys at 1000°C and gamma (fcc) Ni-Cr-Al alloys at 1100°C. Average ternary interdiffusion coefficients are calculated over composition ranges on either side of the Matano plane and examined for the diffusional interactions among the diffusing components. The ternary interdiffusion coefficients determined from the new analysis are observed to be consistent with those determined by the Boltzmann-Matano analysis at selected compositions in the diffusion zone. The average ternary interdiffusion coefficients are also employed in the analytical solutions based on an error function for the generation of concentration profiles for the selected diffusion couples. The generated profiles are a good representation of the experimental profiles exhibiting up-hill diffusion or zero-flux plane development for the individual components.

HOT DEFORMATION OF ALUMINUM ALLOYS: Process Simulation and Developmet - I

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

Wednesday AM Room: Orchard
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Stuart MacEwen, Alcan International, Kingston, Ontario K7L 5L9 Canada; E. Nes, Norwegian University of Science and Technology, Department of Metallurgy, Trondheim, N-7034 Norway

8:30 AM INVITED PAPER

MICROSTRUCTURE CONTROL ON AN ALUMINIUM HOT REVERSING FINISHING MILL: *Jurgen Hirsch*¹; Rainer Grenz²; ¹VAW Aluminium AG, R&D, Bonn 53014 Germany; ²Mannesmann Demag AG Metallurgy, Ratingen Germany

With most Aluminium alloys the hot rolling process has a major effect on finished material quality. Special attention must be paid to the process configuration selected and the factors affecting the development of the microstructure and hence metallurgical properties of the rolled strip. The strength and forming properties of the strip depend to a high degree on the complex control of rolling parameters such as temperature, strain, strain rate and interstand time - especially critical in the hot reversing finishing process. By utilizing precise simulation models the effect of recrystallization and cube texture formation during the hot rolling process can be predicted and the corresponding rolling parameters optimized. In combination with powerful process control and automation systems the high product quality standards required, for example, for can body sheet can be achieved. The new hot mill complex comprising a hot reversing roughing mill coupled with a hot reversing finishing mill currently being implemented with Hulett Aluminium in South Africa illustrates the trend towards low capital cost, high-tech "minimill" configurations. The two-stand process configuration has been selected to improve mill productivity, plant flexibility and product quality. The Hulett hot mill concept is used as a case study and reviewed within this paper.

9:00 AM INVITED PAPER

MODELING THE EFFECTS OF FRICTION AND GEOMETRY ON DEFORMATION PATH DURING HOT ROLLING OF ALUMINUM: *D. A. Korzekwa*¹; A. J. Beaudoin²; ¹Los Alamos National Laboratory, Los Alamos, NM 87545 USA; ²University of Illinois Urbana-Champaign, Dept. Mechanical Engineering, Urbana-Champaign, IL 61801 USA

Processing conditions in the hot rolling of aluminum alloys lead to non-uniform deformation within the roll bite. The ratio of slab thickness to arc of contact in the roll gap can vary over a wide range because of the large initial slab thickness and the very large reductions that are possible for thinner slabs. The degree of non-uniform deformation also depends on friction conditions and the rate dependence of the material. It is necessary to know the regime of deformation rate and temperature space that is developed in the roll bite to make proper application of evolving material models. In this work, a parametric study of hot rolling is conducted. The effects of friction model, friction coefficient, roll gap geometry and temperature on the deformation rate field are demonstrated. The degree and nature of redundant work (shearing) is contrasted for different streamline locations within the bite. Recommendations for the application of material models in analysis of rolling are made with consideration of the simulation predictions.

9:30 AM INVITED PAPER

FORMATION OF SURFACE COARSE GRAINS IN ALUMINUM ALLOYS DURING EXTRUSION: *P. T. Wang*¹; R. D. Dogerty²; ¹Alcoa Technical Center, Aluminum Co. of America, Alcoa Center, PA 15069 USA; ²Drexel University, Department of Material Engineering, Philadelphia, PA 19104 USA

Surface coarse grain formation in aluminum alloys during extrusion has been investigated using a sub-scale indirect extrusion approach where various thermomechanical histories under extremely large strains and elevated temperatures could be imposed on extrudates. During the course of coarse grain formation in extrusion, three types of recrystallization behavior could be observed: in-situ recrystallization during extrusion, primary and secondary (coarsening) recrystallization during post cooling and solution heat treatment. A recrystallization front criterion is proposed and is used as a constitutive model to describe the thickness of in-situ surface recrystallized layers formed during extrusion. In conjunction with finite element analyses, a recrystallization front from the surface to the near surface areas and the formation of coarse grains behind the front for direct extrusion can be described. For tracking a given material point or an extruded grain along a material flow line, the proposed recrystallization criterion is sensitive to thermal and deformation histories, which realistically reflects experimental observations. It is suggested that surface temperature and strain rate, and surface strain profiles are key parameters for the control of surface quality. Additional research effort in modeling primary and secondary recrystallization is mentioned. The methodology presented here can be utilized for studying the effect of dispersoid on recrystallization for extrusion products.

10:00 AM INVITED PAPER

MODELLING RECRYSTALLISATION IN MULTI-PASS HOT ROLLING AND EXTRUSION OF COMMERCIAL ALUMINIUM ALLOYS: *Hans Erik Vatne*¹; ¹Aluminium, R&D Materials Technology, Sunndalsora N-6600 Norway

A model for recrystallisation after hot deformation of aluminium alloys has recently been developed [1,2]. The model is based on thorough experimental investigations of recrystallisation behaviour and deformed microstructure and texture of hot plane strain compressed AA1050 and AA3104 alloys, where special attention has been paid to the industrially relevant cube orientation [3,4]. The model differs from other recrystallisation model in the sense that several of the basic equations are physically based rather than purely empirical. In the present paper the model has been compared to multipass hot deformation of AA3104 alloys and extruded AA6060 and AA6082

types of alloys. Model validation for multi-pass hot rolling is not easy, as industrial tests are commercially sensitive and specimen collection is difficult from a practical point of view. Here, the multi-pass aspect has been tested in several ways. Firstly, by 2-pass plane strain compression laboratory tests, where the inter-annealing time between the 2 deformation passes was varied to give different amounts of recrystallisation between the 2 deformation passes. Secondly, by comparing to a fairly controllable laboratory hot rolling mill. Thirdly, the multi-pass aspect was tested by industrial hot rolling trials by a reversible break-down mill in connection with a 2-stand tandem mill. The model has also been applied to extrusion of 6xxx series alloys (by a vertical laboratory press), where recrystallisation kinetics, texture and grain size was modelled. The alloy AA6082 is particularly challenging, as precipitation of Mn-bearing dispersoids before and during annealing strongly interacts with the recrystallisation process.

10:30 AM INVITED PAPER

QUANTIFICATION AND SIMULATION OF TEXTURE GRADIENTS IN ROLLED ALUMINUM PLATE: *Paul Dawson*¹; Matthew P. Miller¹; Todd J. Turner¹; ¹Cornell University, Sibley School of Mechanical and Aerospace Engineering, 196 Rhodes Hall, Ithaca, NY 14853 USA

Rolled, thick gauge plate, such as that used for the fabrication of airframe components, exhibits through-thickness gradients in the crystallographic texture which arise from the heterogeneous nature of processing deformations. Near-surface material is subject to more intensive shearing than material that is closer to the plate centerplane, shearing that reverses its sense as material passes the neutral point. The prediction of the crystallographic texture, including the strength of its gradients, is a goal of process simulation, and having an effective means of representing the texture is critical to being able to quantify its gradients with accuracy. The crystallographic texture for a sufficiently large volume of material (sufficient in the sense that the volume contains on the order of thousands of crystals) can be represented in a number of ways. A discrete collection of orientations is one possibility, while an orientation distribution function is another. Orientation distribution functions specify the probability density (a scalar) as a function of position in orientation space. The representation can be constructed in terms of various basis functions, such as harmonics in which approximating functions each span the entire space or finite elements in which the basis functions are piecewise. Further, the choice of spaces are many, including conventional Euler angle representations as well as angle-axis representations. Here we utilize a combination of an angle-axis representation (Rodrigues vectors) and finite element interpolation, noting that the parameters of this representation are nodal values which coincide with the orientation distribution value at the nodal point position and which can be interpolated easily between neighboring points. In this paper we discuss the robustness of this methodology for quantifying texture gradients in relation to measurements made on rolled aluminum plate using SEM electron backscatter patterns. The texture obtained by interpolating between two known distributions is compared to the measured texture. Further, we discuss the distributions computed using a finite element, polycrystal plasticity simulation for aluminum rolling in terms of the ability to capture the texture gradient, especially in terms of important texture components.

11:00 AM INVITED PAPER

MODELLING TEXTURE EVOLUTION DURING BREAK-DOWN ROLLING OF AA3104: *M. R. Van der Winden*¹; H. E. Vatne²; C. M. Sellars³; ¹Koninklijke Hoogovens NV, P.O. Box 10.000, 1970 CA IJmuiden The Netherlands; ²Hydro Aluminium a.s., P.O. Box 219, Sunndalsora N-6601 Norway; ³The University of Sheffield, Engineering Materials, Mappin Street, Sheffield S1 3JD United Kingdom

Recently a new experimental technique has been developed to simulate industrial break-down rolling. The Sheffield Mill for Aluminium Rolling at Temperature provides a possibility to study the microstructural development of a specimen at each stage between as-cast ingot to transfer gauge. In the present work, this technique has been used to determine the texture evolution during break-down roll-

ing. In parallel to the experimental technique, a model was developed that can be applied to break-down rolling. One of the parameters calculated by the model is texture. So far, this aspect of the model has not been validated. It is the objective of the present work to do this. Also, the model has been refined. The main alteration enables the changes in the second phase particle distribution to be taken into account.

11:30 AM INVITED PAPER

EXPERIMENTAL TECHNIQUES AND APPLICATIONS OF SUB SCALE PHYSICAL SIMULATIONS: *Vivek M. Sample*¹; ¹Alcoa Tech Center, 100 Tech Dr., Alcoa Center, PA 15069 USA

Physical simulations of thermo mechanical histories can be successfully conducted to gain a fundamental understanding of microstructure evolution as well as to obtain an empirical relationships of process-microstructure and microstructure-properties. Such simulations often provide several distinct advantages over full scale plant experiments such as: economic and expeditious means of obtaining desired data; independent and precise control of process parameters; better data of the actual history applied; ability to understand the microstructure evolution through a sequence of operations; and, ability to go beyond the process envelope of production equipment (e.g. temperatures, strain rate and stress). Techniques and limitations of conducting sub scale thermo mechanical simulations will be discussed.

MECHANISMS AND MECHANICS OF COMPOSITE FRACTURE: Particle 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, UES, Inc., Dayton, OH 45432-1894 USA; Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

Wednesday AM Room: Paris A
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: M. L. Gambone, Air Research Laboratories, Materials and Manufacturing Directorate, Wright Patterson Air Force Base, OH 45433 USA

8:30 AM

FRACTURE BEHAVIOR OF SiC PARTICULATE-REINFORCED ALUMINUM-ALLOY COMPOSITES: *A. B. Pandey*¹; B. S. Majumdar¹; D. B. Miracle²; ¹UES, Inc., 4401 Dayton-Xenia Road, Dayton, OH 45432 USA; ²Air Force Research Laboratory, AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

Discontinuously reinforced aluminum (DRA) composites have poorer ductility and fracture toughness than the base matrix alloy. The effects of microstructural parameters, such as particle size and volume fraction, are not well established as yet, and are the subject of this controlled study. Two matrix alloys were selected: 2009 and 7091 alloys. Additionally, two different particle sizes, 4.9 μm and 10.4 μm , and volume fractions, 15 and 25 vol.%, of SiC, were considered. These composites were processed using a powder metallurgy technique, and tested in the nominally underaged and peak-aged conditions. The composite containing finer SiC particles showed slightly higher yield strength and ductility than that with the coarse particles. The volume fraction of SiC had considerable influence on the ductility of the composite. The fracture toughness exhibited some depen-

dence on the volume fraction, but was negligibly influenced by the particle size. Fractography and damage observations will be presented to explain some of the results.

8:55 AM INVITED PAPER

STUDIES OF DAMAGE IN A MODEL ZrO_2 PARTICLE REINFORCED Al-6061 MATRIX COMPOSITE USING DUAL ENERGY X-RAY TOMOGRAPHY: Mr. Justice¹; B. Derby¹; P. Anderson²; G. Davis²; J. Elliott²; ¹University of Oxford, Department of Materials, Parks Road, Oxford, Oxi 3PH UK; ²Queen Mary and Westfield College, Department of Biophysics, School of Dentistry, Mile End Road, London, England E1 4NS UK

It is well known that particle fracture or particle/matrix decohesion are important damage mechanisms which occur in particulate reinforced metal matrix composites soon after the onset of tensile plastic flow. There has been considerable discussion in the literature concerning the role of reinforcement particle clustering in controlling the onset of damage nucleation in these materials. In this study we use a dual energy X-ray tomography technique which allows the separation of reinforcement and void spatial distribution from the same reconstructed slice. Unfortunately, to achieve this either the matrix or reinforcement must contain a component with a strong X-ray absorption edge. Here we study a model ZrO_2 particulate reinforced Al-6061 alloy. In this system our results indicate a correlation between void distribution and regions of higher than normal reinforcement density.

9:20 AM

FAILURE OF MMCS UNDER CYLIC LOADING - MEASUREMENT AND INTERPRETATION OF STRESSES USING NEUTRON DIFFRACTION: M. R. Daymond¹; M. E. Fitzpatrick²; ¹IRIS, Rutherford Appleton Labs, Chilton, Oxon UK; ²Open University, Dept. of Materials, Milton Keynes UK

Neutron diffraction allows non-destructive monitoring of the stress state within the phases of a composite. Multiple phases can be simultaneously examined in situ while macroscopic temperature or stress loads are applied. The technique has been used to characterise the deformation and load sharing characteristics of Metal Matrix Composites undergoing isothermal creep, thermal cycling creep and cyclic fatigue loading. This talk will focus on the latter form of failure in MMCS. The residual stress state of a composite material is known to affect the initiation and growth of fatigue cracks, and thus fatigue resistance. For instance the compressive stress produced in the reinforcement phase of an MMC due to thermal misfit upon cooling from fabrication retards the early stages of fatigue crack growth. We have explored the effect of fatigue cycling on the residual stress state of an Al/20%SiC particulate reinforced MMC, using pulsed neutron diffraction. A novel stroboscopic data collection system has been used to allow data collection in situ during fatigue cycles at various stress levels. Comparisons are made with finite element model predictions.

9:45 AM INVITED PAPER

EFFECT OF REINFORCEMENT CLUSTERING ON THE STRENGTH AND DUCTILITY OF PARTICLE-REINFORCED COMPOSITES: J. Llorca¹; M. Elices¹; C. Gonzalez¹; ¹Polytechnic University of Madrid, Department of Materials Science, E.T.S. de Ingenieros de Caminos, Madrid 28040 Spain

A micromechanical model is developed to simulate the mechanical response of particle-reinforced metal-matrix composites including the effect of reinforcement clustering. The microstructure of the composite is represented as a three-dimensional array of hexagonal prisms with one reinforcement at the center of each prism. The shape, volume fraction and state (either intact or broken) of the reinforcement is independent for each cell, so it is possible to study the interaction among regions with large and small reinforcement volume fractions. The tensile response of the composite is determined from the behavior of the intact and damaged cells, the fraction of damaged cells being calculated on the assumption that the

reinforcement strength follows the Weibull statistics. The model is used to determine the effect of the heterogeneous distribution of the reinforcements with different shape within the composite on the tensile strength and ductility.

10:10 AM

EFFECTS OF STRESS TRIAXIALITY ON THE DAMAGE EVOLUTION OF DISCONTINUOUSLY REINFORCED COMPOSITES: Peravudh Lowhaphandu¹; John J. Lewandowski¹; ¹Case Western Reserve University, Dept. Materials Science and Engineering, 10900 Euclid Ave., Cleveland, OH 44106 USA

The effects of changes in stress triaxiality on the damage evolution were determined for AZ91 Mg matrix composites in addition to 7093Al matrix composites containing several types of reinforcement. The stress triaxiality variation was obtained via utilizing notched axisymmetrical tensile specimen and superimposed hydrostatic pressure. SEM fractography and optical metallography were used to quantify the damage levels. The damage levels (i.e. amount of particle cracking) observed were correlated with the level of imposed stress triaxiality and particle geometry.

10:35 AM BREAK

10:50 AM INVITED PAPER

A MICROSTRUCTURE BASED COMPUTATIONAL MODEL FOR DAMAGE EVOLUTION: S. Moorthy¹; S. Ghosh¹; B. S. Majumdar²; ¹The Ohio State University, Dept. of Aerospace Engineering, Applied Mechanics and Aviation, Columbus, OH 43210 USA; ²UES Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

A Voronoi Cell finite element model (VCFEM) is developed for damage initiation and evolution in composite microstructures. An elastic constitutive relation is used for the inclusion, for which particle cracking is modeled by the Mohr's theory of maximum principal stress or the Rankine criterion. To represent ductile failure, the matrix material is represented by a pressure dependent elastic-plastic constitutive relation for porous materials. In particular a non-local form of the Tvergaard-Gurson model with void nucleation, growth and coalescence is used for modeling the ductile response of the matrix. Evolution of damage in the microstructure is monitored through growth in porosity of the microstructure. In the experimental component of this work, metal matrix composites with different particle volume fractions and particle sizes subjected to different strain levels by uniaxial tension, were studied. Microstructural images formed input for the VCFEM. Damage evolution from VCFEM are compared with the experimental observations. In particular, damage in the microstructure near a structural flaw is compared with experimental observations by modeling a distribution of particles ahead of the flaw in a compact tension test. Damage in the form of brittle particle cracking and ductile matrix softening are compared with experimental observations obtained by image analysis.

11:15 AM INVITED PAPER

INFILTRATED BORON CARBIDE AND ALUMINA PARTICLE REINFORCED ALUMINUM COMPOSITES: M. Kouzeli¹; T. Grant¹; L. Weber¹; B. Viguier¹; C. San Marchi¹; A. Mortensen¹; ¹Swiss Federal Institute of Technology in Lausanne, Department of Materials, Laboratory for Mechanical Metallurgy, Lausanne, EPFL CH-1015 Switzerland

The fracture and deformation characteristics of particle reinforced metal-matrix composites are often complicated by the large number of microstructural variables typically present in this class of material, such as particle distribution, porosity and secondary phases. The present study is thus partly motivated by the comparatively greater microstructural simplicity of pressure-infiltrated composites, which are pore-free, contain a uniform distribution of particles, and can feature microstructurally simple matrix and reinforcement structures. Focus is placed on composites which are based on aluminium reinforced with approximately 50% of boron carbide or aluminum oxide particles, to

highlight the importance of particle characteristics. The incidence of particle size and other microstructural variables on the mechanical behavior of these materials is investigated with a dual focus on tensile behavior and fracture toughness

11:40 AM INVITED PAPER

THE CONSTRAINT EFFECT ANALYSIS OF SiC PARTICLE REINFORCED ALUMINUM MATRIX COMPOSITES: *M. Kikuchi*¹; *M. Geni*¹; ¹University of Tokyo, Dept. of Mechanical Engineering, Noda, Chiba 278 Japan

The dimple fracture test is conducted by changing the stress triaxiality using notched specimen of SiC particle reinforced aluminum alloy. The fracture surface is observed by SEM, and it is found that the stress triaxiality has large effect on the dimple size and the critical void volume fraction. Using the three dimensional FEM, based on the large deformation theory and Gurson's constitutive equation, the interaction effect of SiC particles during dimple fracture is evaluated. The distribution of the stress triaxiality is also obtained and the effect on the dimple fracture process is discussed.

12:05 PM INVITED PAPER

OPTIMIZING THE FRACTURE RESISTANCE OF PARTICULATE COMPOSITES BY MICROSTRUCTURAL MODIFICATION: *David L. Davidson*¹; ¹Southwest Research Institute, 6220 Culebra Road, San Antonio, TX 78238 USA

Somerday and Gangloff found that the fracture of notched specimens of particulate containing composites is insensitive to notch shape. This result indicates that the effect of macroscopic notch geometry related constraint is not applicable to particulate composites. Measurement of constraint on a microstructural scale (Davidson and McClung) confirms that particles cause microscale constraint. Particle induced constraint, together with other fracture related parameters, has been combined into a model for computing fracture toughness based on particle volume fraction, size, fracture toughness, and matrix strength and fracture strain. The model is calibrated with results from some Nb-based composites. Using the model allows estimates to be made of the effects on fracture toughness of changing particle and matrix characteristics. Thus, the microstructural characteristics that optimize the fracture resistance are predicted, which can provide guidance for further development of tough particulate composites.

MODELLING OF TEXTURE AND ANISOTROPIC PROPERTIES IN STEEL AND ALUMINUM INDUSTRIES FOR OPTIMUM MATERIALS PERFORMANCE: Session 1

Sponsored by: ASM International: Materials Science Critical Technology Sector, Texture & Anisotropy Committee

Program Organizers: Hasso Weiland, Alcoa, Alcoa, PA 15069 USA; J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

Wednesday AM Room: Madrid
October 14, 1998 Location: O'Hare Hilton Hotel

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

8:30 AM INVITED PAPER

SURFACE ROUGHENING AND MICROSTRUCTURE IN SHEET METALS: *A. D. Rollett*¹; *P. S. Lee*¹; *G. Jarvis*²; *H. R. Piehler*²; *B. L. Adams*²; *S. Saigal*³; *C. Shet*³; ¹Dept. of Mater. Sci. & Eng., Carnegie

Mellon University, Pittsburgh, PA 15213-3890; ²Dept. of Civil and Environmental Eng., Carnegie Mellon University; ³Alcoa Technical Center, 100 Technical Drive, Alcoa Center, PA 15069-0001

Considerable effort has been expended over the years to develop processing methods and sheet forming techniques that preserve a high quality surface finish. The crystallographic nature of plastic deformation in sheet metals means, however, that a free surface will tend to deform non-uniformly. Our studies of surface roughening have shown that, in addition to roping and L-ders bands, distributed linear localization can occur. This form of localization which occurs in a range of straining modes may be linked to shear banding in the sheet materials. Evidence will be presented from Orientation Imaging Microscopy (OIM) on 6111-T4 aluminum sheet that this form of surface roughening is associated with strain inhomogeneities through the thickness of the sheet. The microtexture in the sheet was also observed to be inhomogeneous and the relationship between microtexture and shear banding will be explored. Techniques used in this work include OIM, interference microscopy, digital image analysis, polycrystal plasticity simulations and finite element simulations with constitutive descriptions of materials that incorporate crystal plasticity.

9:00 AM

TEXTURE EVOLUTION IN ALUMINUM ALLOY SHEETS DURING DEEP DRAWING : *Shi-Hoon Choi*¹; *Jae-Hyung Cho*¹; *Kwansoo Chung*²; *Frederic Barlat*³; *Kyu Hwan Oh*¹; ¹Seoul National University, School of Materials Science and Engineering, Research Institute of Advanced Materials, San 56-1 Shinrim-dong, Kwanak-ku, Seoul, Korea; ²Seoul National University, Department of Fiber and Polymer Science, San 56-1 Shinrim-dong, Kwanak-ku, Seoul, 151-742 Korea; ³Aluminum Company of America, Alcoa Technical Center, 100 Technical Drive, Alcoa Center, Pittsburgh, PA USA

The texture evolution by deep drawing of AA1050 sheet metals was investigated and the stable orientation was predicted using rate sensitive model. The measured textures show the different behavior with the initial crystal orientations and strain state. Among the crystal orientations of RD sample, the crystal orientations around the D component rotated toward the Cu component and the crystal orientations along the α fibre rotated toward the $\{1\ 1\ 0\} \langle 0\ 0\ 1 \rangle$ and $\{1\ 1\ 0\} \langle 1\ 1\ 1 \rangle$ components during deep drawing. In the case of the TD sample, the crystal orientations around the D component rotated toward the Rotated Cube and the crystal orientations along α fibre rotated toward the $\{1\ 1\ 0\} \langle 1\ 1\ 3 \rangle$ component. In order to calculate the strain path during deep drawing, deep drawing for aluminum alloy sheet was simulated using anisotropic elasto-plastic finite element analysis. In this study, the orientation density in the wall part which undergoes tensile deformation was interpreted by considering sequential deformation states such as plane strain and tensile strain state.

9:30 AM INVITED PAPER

ASSESSMENT OF VARIOUS YIELD CRITERIA FOR PREDICTION OF FORMING LIMIT STRAINS IN AN ALUMINUM SHEET: *J. Savoie*¹; ¹Alcan, Kingston Canada

In order to simulate adequately the formability of sheet materials, it is important to use a yield criterion which accurately captures the anisotropy of the material due to crystallographic texture. These yield functions must be in a form that can be used easily in finite element analysis. This work investigates the use of two commonly used yield criteria by Hill [1990] and Barlat [1989], as well as a 4th order inverse potential function [1996] for forming limit predictions. User-material subroutines, based on these functions, have been written and implemented into a commercial finite element code - Abaqus/Implicit. Finite element simulations of hemispherical punch stretching process are carried out for an automotive aluminium sheet. Different values of coefficient of friction between the punch and sheet surfaces are employed in the models to simulate strain paths in the range between plane strain and equi-biaxial tension. The model results are compared with a range of experimental parameters such as punch load versus displacement, strain profiles along the longitudinal and transverse orientations of the sheet (with respect to the rolling direction), and limit strains, to assess the suitability of various yield

criteria. It is observed that not only necking but also fracture mechanisms are controlling the forming limits close to equi-biaxial tension. Thus, a fracture criterion was incorporated into the models to improve the predictions of limit strains.

10:00 AM

THE ROLE OF INHOMOGENEITY IN PLASTIC FLOW ON PLASTIC ANISOTROPY IN AA5XXX ALLOYS: *G. Burger*¹; ¹Alcan International Limited, Kingston Research and Development Center, Kingston, Ontario K7L 5L9 Canada

The yield strength, R value and work hardening behaviour of AA5754 and AA5182 sheet material in the O temper were evaluated along orientations at 0, 45 and 90 degrees to the rolling direction. AA5182 sheet in a hard-rolled temper was also considered. Plastic straining at room temperature occurs inhomogeneously, exhibiting serrated flow. To assess the role of serrated flow, testing was also carried out at -70 C, at which plastic flow occurs homogeneously. In O-temper sheet, the yield point at -70 C was almost identical to that at room temperature, although the Lüders elongation was smaller. Lüdering phenomenon was not observed in hard-rolled material. Low temperature did promote an increase in work hardening rate and uniform elongation, however, the increases in d/d_0 in different orientations were equivalent. The elimination of serrated flow had no significant influence on measured R values in any orientation. The development of crystallographic texture during tensile straining at these two temperatures was also identical. Thus it was concluded that the inhomogeneous plastic straining occurring during serrated flow did not influence the macroscopic plastic behaviour of the sheet. The anisotropy in R value and in flow stress in these materials was not well predicted based on a full constraints Taylor-Bishop-Hill calculation utilizing the measured crystallographic textures of the sheet in O temper.

10:30 AM

DO SOLUTE SHEAR BANDS PROMOTE THE BRASS DEFORMATION TEXTURE IN HEAVILY COLD ROLLED 2024 AND 7475?: *Sabir Doherty*¹; Carl T. Necker²; Roger D. Dougherty¹; Surya Kalidindi¹; Ehab El-Danaf¹; ¹Drexel University, Dept. of Materials Engineering, Philadelphia, PA 19104 USA; ²Los Alamos National Laboratory, Los Alamos, NM USA

The texture transition from a copper-type rolling texture to a brass type one associated with low stacking fault energy driven deformation twinning is believed by some investigators to arise not directly with the onset of twinning but with the twinning and high strain hardening induced shear banding. In order to test this idea the deformation textures in heavily rolled aluminum alloys containing high solute contents (solution treated quenched 2024 and 7475 known to form many shear bands) are being studied to determine if shear banding in these high stacking fault energy alloys, which do not twin, give a conventional copper type Texture or one more similar to the Brass texture.

11:00 AM

PLASTIC ANISOTROPY IN PRECIPITATION-STRENGTHENED ALUMINUM ALLOYS DEFORMED IN UNIAZIAL AND PLANE STRAIN COMPRESSION: *Barlat and J. Liu*¹; ¹Alcoa Technical Center, 100 Technical Drive, Alcoa Center, PA 15069-0001 USA

The goal of this work was to quantitatively assess the plastic anisotropy of strongly textured polycrystals with and without a distribution of hard (non-shearable) precipitates. The aluminum-copper system was selected as a model material. The strong texture in the experimental design provided a high level of sensitivity for evaluating plastic anisotropy. The choice of polycrystals rather than single crystals was to favor multiple slip, which is the behavior more representative of commercial materials. The anisotropic behavior was characterized by the shape change of the specimen cross-sections and the shape of the stress strain curves after uniaxial and plane strain compression tests. A model based on kinematic hardening concepts

was developed to quantitatively describe the plastic behavior of materials containing non-shearable precipitates such as q' in Al-Cu alloys. This model was validated by the experimental results.

11:30 AM

THE EFFECT OF SPECIAL PROCESSING TECHNIQUE ON THE ANISOTROPY OF Al-Li ALLOY: *L. Brandao*¹; P. N. Kalu¹; H. Garmestani¹; O. Es-Said²; Eui Lee³; ¹FAMU-FSU College of Engineering, Department of Mechanical Engineering and Center for Materials Research and Technology, (MARTECH), Tallahassee, FL 32310-6046 USA; ²Loyola Marymount University, Department of Mechanical Engineering, Los Angeles, CA 90045 USA; ³Naval Air Warfare Center, A/C Division, Code 4.3.4.2, Warminster, PA 18974 USA

The use of aluminum-lithium alloys in aerospace applications has been limited due to their undesirable characteristics, e.g. anisotropy. A distinct thermomechanical processing, which combines rolling and stretching (at five different angles) was used to process Al-2095 alloy with the view to eliminate anisotropy. The material was tested in tension and characterized. This paper presents a preliminary attempt to correlate the mechanical anisotropy and texture development with some texture theories. Analyses based on upper bound model using restricted and pencil glide approaches suggested that the contribution of crystallographic texture to anisotropy was only part of the total mechanical anisotropy.

12:00 PM

UNIAXIAL AND BIAXIAL ANISOTROPY IN ALUMINUM ALLOYS: *C. T. Necker*¹; C. N. Tomel¹; D. A. Korzedowa¹; ¹Los Alamos National Laboratory, Los Alamos, NM USA

The mechanical anisotropy of sheet material is often measured using the uniaxial tensile test and is represented as Lankford coefficients (R values), the ratio of width strain to thickness strain. There is a correlation between R values and formability in steel however the same can not be said for aluminum. R values less than unity are typical for aluminum. However when the formability of sheet material is tested under biaxial tensile (bulge) conditions the measured through-thickness strength is often found to be greater than the in-plane strength, counter-intuitive to the R value measurement. These apparently contradictory strain proportionalities are investigated computationally using a visco-plastic self-consistent approach to model both uniaxial and biaxial conditions. The approach uses the initial texture combined with imposed stress state conditions to model the evolution of the yield surface and calculate R values. The calculated R values generally relate reasonably well with the measured values. The modeling effort is extended into the plastic regime by combining the texture and imposed stress state conditions with experimentally determined material hardening information. Modeling of both uniaxial and biaxial conditions out to the ultimate strength of the sheet shows that the differences between experimental conditions are captured by this model.

12:30 PM INVITED PAPER

TEXTURE AND EARING AL CAN-SHEET: *J. Hirsch*¹; ¹VAW Aluminium AG, R&D, Bonn Germany

The earing profile obtained in deep drawing of cups must be kept low in order to maintain optimum output and performance during can manufacturing operations. Since sheets of the Aluminium (AA3004) alloy are used in a highly cold rolled state form strong 45° ears this effect must be balanced by the 0°/90° earing characteristic of the initial hot band cube texture. During cold rolling the earing profile changes from 0°/90° to 45° earing with characteristic intermediate profiles (with 6-ears). This transition is analyzed systematically for various initial hot band cube textures and for different rolling reductions. A new method for the evaluation of earing profiles in Al-sheet is presented and applied which gives a quantitative description of the profiles and their transition and the correlation between textures and earing profiles. The variation of initial textures and the minimum rolling strain to achieve sufficient strength can be predicted and the

rolling schedule can be optimized in order to obtain the best combination of low earing and required strength properties.

PAUL A. BECK MEMORIAL SYMPOSIUM: Paul A. Beck Memorial Symposium - III Magnetism

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee

Program Organizers: Karl A. Gschneidner, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Michael V. Nevitt, Clemson University, Dept of Physics & Astronomy, Clemson, SC 29634 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899

Wednesday AM Room: Rome
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Charles A. Wert, University of Illinois, Materials Science & Engin., Urbana, Illinois 61801 USA

8:30 AM INVITED PAPER

IRON IN GOLD, PAUL A. BECK: *Anthony Arrott*¹; ¹Virginia State University, Physics Department, P.O. Box 9325, Petersburg, VA 23806 USA

An alloy of iron in gold is inhomogeneous at various length scales depending on the metallurgical treatment. The magnetic moment and anisotropy of each iron atom depend on the arrangement of neighboring iron atoms. The exchange interactions vary with the distances between each pair of iron atoms and the distribution of neighboring iron atoms. These interactions are both ferromagnetic and antiferromagnetic in sign. The exchange interactions are tensors with off diagonal elements that contribute to non-collinearity of magnetic moments. Spin-spin correlation effects are entwined with atom-atom correlation effects. Armed with a phenomenological understanding of metallurgy, thermodynamics and magnetism, Paul A. Beck sought to understand the results of specific heat and magnetic moment measurements and their dependence on the metallurgy. He was able to show that a simple primary assumption of all previous and most continuing theoretical discussion was in doubt. The so called 'spin glass' state seen at low temperatures for concentrations less and greater than the critical concentration for the onset of ferromagnetism is often called the 'reentrant spin glass' phase at the higher concentrations. This is because it was assumed that the higher temperature phase is usual ferromagnetism. Beck's analysis showed that, on a diagram of temperature and concentration, the higher temperature phase was continuous with both the reentrant spin glass phase at low temperatures and the usual ferromagnetic phase at higher concentrations. He called this 'mictomagnetism'. In this state there are clusters of magnetic moments acting together as giant magnetic molecules, but there is an absence of long range order among these clusters. In Au(Fe) the occurrence of mictomagnetism disturbs the clarity of simple models in which the complexities of mictomagnetism are overlooked. The problem sat there and smiled during the years in which Beck worked on it, now that he is gone, the problem still sits there but sadly neglected. Today there are materials that are clearly mictomagnetic as envisioned by Beck, but what about Au(Fe)?

9:05 AM INVITED PAPER

LOCAL ATOMIC ARRANGEMENT AND MAGNETIC PROPERTIES IN Au-Fe SPIN GLASS SYSTEM: *Haydn H. Chen*¹; ¹University of Illinois, Department of Materials Science & Engineering, 1304 West Green Street, Urbana, IL 61801 USA

The Au-rich Au-Fe face-centered cubic alloy system has long been a topic of considerable study due to the spin-glass magnetic behavior displayed for compositions >0.5 at. pct Fe at temperatures below ~ 100 K. The interpretation of the magnetic behavior is not without controversy. For instance, magnetic susceptibility and neutron scattering measurements have given evidence of a "double" magnetic transition corresponding to an interim ferromagnetic state existing between the low-temperature spin-glass state and the high-temperature paramagnetic state. However, Raker and Beck showed through low applied fields and special specimen geometry that no ferromagnetic state exists in water-quenched Au-17.5 at. pct Fe. This paper will review those most recent magnetic results as well as present a detailed local atomic arrangement study based upon diffuse x-ray scattering measurements utilized synchrotron radiation and computer simulation of the short-range ordered structure. Results show for the first time that two different short-range order environments exist. The Fe-enriched environment, although quite disordered, showed a Fe-segregation preference toward {100} and {110} matrix planes. These become more predominant with aging. The Fe-depleted environment is best described using the SCWP (Standing Concentration Wave Packet) model, and the results derived from this work give a real-space visualization of the SCWP. (* Work supported by the US National Science Foundation.)

9:40 AM INVITED PAPER

MAGNETIC PHASE TRANSITIONS PROBED AT VERY LOW MAGNETIC FIELDS: *Helmut Claus*¹; ¹University of Illinois at Chicago, Dept. of Physics, 845 W. Taylor St., Chicago, IL 60607-7059 USA

With the availability of high sensitive SQUID magnetometers it has become possible to probe magnetic phase transitions down to very low magnetic fields. This has led to the discovery of new interesting effects. One is the paramagnetic Meissner effect in superconductors another the diamagnetic response of ferromagnets. As examples we will present data for two paramagnetic superconductors, metallic Nb and the high temperature superconductor YBCO, as well as for the diamagnetic ferromagnet Pd-0.5%Fe. We will demonstrate that in these cases the unexpected sign of the magnetic response is caused by the interaction of a surface layers of slightly enhanced transition temperatures, with the bulk of the samples. Whereas the diamagnetic response of the ferromagnet is well understood, there is still no explanation for the paramagnetic signal for the two superconductors. Part of this research was supported by the US Department of Energy, Basic Energy Science, Materials Science, under Contract No. W-31-109-ENG-38

10:15 AM BREAK

10:35 AM INVITED PAPER

MAGNETICALLY GRANULAR, BUT STRUCTURALLY GLASSY Nd-Fe-Al BASED EXCHANGE COUPLED HARD MAGNETS: *K. V. Rao*¹; *R. J. Ortega*¹; *A. Inoue*²; ¹Royal Inst. of Technology, Condensed Matter Physics, Stockholm SE-100 44 Sweden; ²Inst. of Metals Research, Tohoku University, Sendai, 980 Japan

An unusual maximum of $H_c = 3.5$ Tesla around 30K in the temperature dependence of coercivity is observed in a new class of bulky glassy Fe-Nd-Al based hard magnets. These glassy materials in the form of 3-7 mm rods, fabricated perhaps by the slowest known cooling rates, show isotropic hard magnetic behaviour with room temperature coercivities as high as one Tesla in 30 Tesla fields, but still cannot be magnetically saturated. The origin of the hard magnetic properties appears to be due to exchange hardening provided by the coupling between Fe clusters and an antiferromagnetic matrix. Our studies suggest a new class of Nd-Fe-Al based bulk glassy materials which are magnetically granular although structurally glassy.

11:10 AM

MAGNETIC EXCHANGE BIAS IN A NiO/FeNi ANTIFERRO-MAGNETIC/FERROMAGNETIC BILAYER: Robert D. Shull¹; A. J. Shapiro¹; V. Nikitenko²; V. Gornakov²; A. Khapikov²; L. M. Dedukh²; A. Chaiken³; ¹National Institute of Standards and Technology, Metallurgy Division, Bldg. 223, Rm. B152, Gaithersburg, MD 20899 USA; ²Institute of Solid State Physics, RAS, 14232, Chernogolovka Russia; ³Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

The magnetic domain structure in an antiferromagnet (AF) - ferromagnet (FM) bilayer crystal possessing unidirectional magnetic anisotropy was imaged using a special magneto-optic imaging technique (MOIF) developed jointly by NIST and the Institute of Solid State Physics in Chernogolovka, Russia. In this MOIF technique, a thin film of a material with a large magneto-optic Faraday effect is placed on top of the sample of interest. Leakage fields from the sample (primarily near domain walls) locally polarize the imaging film and reveal the domain structure of the sample. By applying a magnetic field to the sample, domain dynamics are then observable. For a NiO/permalloy bilayer crystal with unidirectional magnetic anisotropy, magnetization reversal upon application of a reversed magnetic field was found to occur by domain nucleation and growth. Different domain nucleation sites were observed in such an AF/FM sandwich for the remagnetization processes in opposite directions while cycling the field, as in the measurement of a magnetic hysteresis loop. These findings indicate that the nucleation of domains in such a material is intimately connected with the magnetic bias imposed on the material, as well as on the sample's microstructure. This finding explains the high coercivity and reduced exchange bias always observed. This has important ramifications for magnetic recording devices.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VII: Exotic Processing Techniques for Materials

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

Wednesday AM
October 14, 1998

Room: Chicago
Location: O'Hare Hilton Hotel

Session Chairs: Dr. Patrick Taylor, University of Idaho, Plasma Processing Laboratory, Moscow, ID 83844-3024 USA; Dr. L. V. Ramanathan, Cidade Universitaria, Energy and Nuclear Research Institute, Travessa R 400, CEP 05508-900

8:30 AM INVITED PAPER

PLASMA SPRAYING OF HYDROXYAPATITE COMPOSITE COATINGS: K. A. Khor¹; C. H. Quek¹; V. J. P. Lim¹; P. Cheang¹; ¹Nanyang Technological University, Singapore, School of Mechanical & Production Engineering, Singapore 63978

Biomedical implants using metals, polymers, ceramics and composites are increasingly being designed to restore bio-function and to facilitate healing of the ailing body part. It is often the material design aspects of such implants that will determine the stability of the biomedical implant in-vivo. This paper investigates the design of hydroxyapatite (HA) based composite layers using dc plasma spraying. Composites based on the combination of HA with Ti-6Al-4V and HA with YSZ are investigated. HA being a bioactive material

combined with non-toxicity and bioinert. Ti-6Al-4V provides an implant that is biocompatible, light and stable. YSZ is known for imbuing improved fracture toughness to many ceramic matrices. In the HA/Ti-6Al-4V composites, the powder feedstock using calcined HA and Ti-6Al-4V was prepared by the ceramic slurry mixing method in the ratio of 80 wt%: 20 wt % and 50 wt%: 50wt% respectively. Yttria stabilized zirconia (YSZ) was ball milled together with HA in the compositions 10% HA, 30% HA and 50% HA, and then plasma sprayed together with the HA onto a Ti-6Al-4V substrate under various spraying parameters. The composite powder was deposited onto the titanium substrate by means of a robot-controlled 100kW plasma gun (Praxair Surface Technologies, USA) equipped with an advanced computerized closed-loop powder feed system. Mechanical properties of the individual layers were tested using the tensile adhesion test. The surface morphology and microstructure of the composite layers were examined by a scanning electron microscope. The phase composition and crystallinity were determined by the X-ray diffractometer. The investigation shows that the as-sprayed coating of the individual layers possess a higher porosity level in the HA-rich regions. Tensile adhesion test revealed a higher bond strength for the composite coatings compared to pure HA coatings.

8:55 AM INVITED PAPER

PRODUCTION OF HOLLOW FIBERS FROM AN IMIDE COPOLYMER: Gaajanan S. Bhat¹; ¹University of Tennessee, 230 Jessie Harris Building, Knoxville, TN 37996-1900 USA

Hollow fibers spun from synthetic polymers have been investigated for a long time, especially for producing high-bulk, low-density fabrics. Such fibers produced from a high temperature resistant polymer have some advantages in intended applications. Ultem, a polyetherimide resin marketed by General Electric company is a copolymer with ether molecules between imide groups. The fully reacted polyimide with the imide group being part of the linear polymer chain makes this polymer thermoplastic and easily dissolvable. Being an amorphous thermoplastic polyimide, the Ultem resin combines the high performance associated with exotic specialty polymers and the good processability of typical engineering plastics. In addition to high strength and modulus, and heat resistance, the polymer has high dielectric strength, broad chemical resistance, transparency and good processability. A special set-up was built at the Phillips Laboratory of Edwards AFB, CA to spin hollow fibers. The spinneret designed has a hollow tube supported in the center of the orifice and an inert gas is injected through the needle to maintain the tubular shape until the solidification of the fiber. Using that set-up, fibers were spun from two grades of Ultem under several different conditions. Effect of these processing conditions on the structure and properties of the formed hollow fibers will be discussed.

9:20 AM INVITED PAPER

RESPONSE SURFACE STUDY ON PRODUCTION OF EXPLOSIVELY-WELDED ALUMINUM-TITANIUM LAMINATES: Efe S. Ege¹; Osman T. Inal¹; C. A. Zimmerman¹; ¹New Mexico Tech, Materials and Metallurgical Engineering, Socorro, NM 87801 USA

Aluminum-titanium multi-layered composites (laminates) were produced through explosive welding, for light weight, structural applications. The purpose of lamination was to have a material exhibiting superior mechanical properties due to the, explosive-welding introduced, plastic deformation throughout each layer and to the lamination-introduced strengthening. A total of eighteen laminates, nine of which were used as points defined by central composite design, were produced. Yield strength, ultimate tensile strength, elongation, and toughness data were collected from tensile tests. A second-order model was fitted and a three dimensional response surface was built to see the relationship between the mechanical properties of the laminates, namely yield strength and toughness and two design variables, abundance of interfaces (or total interfacial area) and volume percentage of the more ductile component. The fitted second-order model clearly shows that the mechanical properties of the laminates strongly depend on the relative amounts of the components but not on the abundance of interfaces within the selected operability region.

Further characterization of the laminates were done by microhardness testing, optical microscopy (OM) and scanning electron microscopy (SEM). Microhardness values revealed extensive hardening, over 25%, especially in titanium layers. Optical microscopy revealed plastic flow in the layers. SEM studies showed that laminates failed in a ductile manner. The presentation will detail our efforts.

10:00 AM INVITED PAPER
TAPE CASTING OF COMPOSITE CERAMIC SUBSTRATES USING HOLLOW MICRO-SPHERICAL POWDERS: Alfred I.Y. Tok¹; *Freddy Y. C. Boey*¹; K. A. Khor¹; ¹Nanyang Technological University, School of Mechanical & Production, Nanyang Avenue, Singapore 639798

The current device of miniaturization and higher device counts in IC (integrated circuit) packages has significantly increased the use of both multilayer ceramic packages (MLCP) and multilayer capacitors (MLC). Both employ thin ceramic tapes with very high dielectric properties which are cast using solid powders. Crucial to the production of these ceramic substrates is the use of a tape caster with precise control of tape thickness, uniformity and surface finishes. A novel approach is presently taken in producing a composite ceramic substrate using hollow micro-spherical powders via the tape casting method. The advantage being significantly lower density and better dielectric behaviour. This involved the proper control of both the formulation as well as the processing parameters, both of which were critical to the quality and reproducibility of the substrates.

10:25 AM INVITED PAPER
THE SOLVENT LAMINATION FOR POWDER INJECTION MOLDING FEEDSTOCKS: *Ting-Chu Ko*¹; James D. Cawley¹; Arthur H. Heuer¹; ¹Case Western Reserve University, Department of Materials Science and Engineering, 10900 Euclid Avenue, Cleveland, OH 44106-7204 USA

Rohm & Haas stainless steel 316L powder injection feedstocks have been evaluated as a raw material for solvent lamination. This was performed by compression molding the feedstocks into sheet that was cut into the desired dimension for lamination aided by solvent, and then debinded and sintered. Various solvent-based techniques were evaluated for sheet lamination, and the optimum set of lamination parameter results was determined. Employing to this optimized lamination formulation, complex objects were generated. In some, a fugitive material has been used to aid in registration. The effect of solvents on mechanical properties will be also reported. Modeling of the solvent concentration profile in the tape was performed in order to confirm and explain the measured property change after the solvent treatment. The polymers in feedstocks were identified and the kinetics of phase separation for those polymers was determined using capillary rheometer and Dynamic Mechanical Analysis (DMA).

10:50 AM INVITED PAPER
THE DEVELOPMENT OF AMORPHOUS Fe-Cr-P ELECTRODEPOSITS: C. T. Kunioshi¹; O. V. Correal¹; N. B. de Lima¹; *L. V. Ramanathan*¹; ¹Cidade Universitaria, Energy and Nuclear Research Institute, Travessa R., CEP CDP 05508-900

Some metallic amorphous alloys have shown superior corrosion resistance and could thus be used for protecting other less resistant alloys in aggressive environments. A number of techniques have been used to obtain amorphous alloy coatings. One of the techniques, aqueous electrodeposition is simple and economical. This paper presents the effect of electroplating parameters on the structure, morphology, composition and corrosion resistance of Fe-Cr-P deposits. The effect of various complexing agents and other bath additives as well as current density, duration of deposition and the use of ion selective membranes on deposit characteristics have been investigated. Deposits with average grain size as low as 15Å were obtained. Corrosion measurements on uncoated and amorphous alloy coated mild steel specimens in acidic media revealed significant increases in corrosion resistance of the coated specimens. These data along with

the effect of varying the plating parameters during electrodeposition to obtain gradient amorphous deposits will be presented and discussed.

11:15 AM INVITED PAPER
PRODUCTION OF BARIUM FERRITE FILMS USING AN INDUCTIVELY COUPLED PLASMA REACTOR: Edgar E. Vidal¹; *Patrick R. Taylor*¹; ¹University of Idaho, Plasma Processing Laboratory, Department of Metallurgical and Mining Engineering, Moscow, ID 83844-3024 USA

The production of barium ferrite thin films has been evaluated experimentally using Plasma Assisted Chemical Vapor Deposition in an Inductively Coupled Thermal Plasma (ICP) Reactor. Thermal plasma appears to demonstrate technological and economical advantages over existing methods. The films were characterized using Scanning and Transmission Electron Microscopy (SEM and TEM) and X-Ray Diffraction (XRD). Magnetic properties of the films were also determined: saturation magnetization, coercivity, and squareness ratio. A mathematical model that describes the deposition process at the substrate is presented and compared with experimental results. Solutions prepared with the salts of the desired elements are atomized in the plasma region, ensuring complete vaporization of the precursors. Experimental variables studied include plasma torch power, relative substrate-plasma position, solution stoichiometry, and substrate material. It was observed that under certain conditions the resultant films were amorphous, thus requiring annealing.

11:40 AM
THE EFFECT OF PRIOR HEAT TREATMENT AND PROCESSING VARIABLES ON THE HARDENED LAYER OF NITRIDED EN40B STEEL: *S. Mridha*¹; D. H. Jack¹; ¹Nanyang Technological University, School of Applied Science, Nanyang Avenue, Singapore 63978

The growth kinetics of nitrided layer is investigated by nitriding EN40B steel at 470°, 520°, and 570°C for 6h to 96h in gaseous environments containing different amounts of ammonia. The effects of austenitizing and tempering temperatures, and surface preparation prior to nitriding are also discussed. The nitrided layer thickness is found to increase parabolically with increasing processing time. The growth of this hardened layer shows a linear relationship with nitriding potential at all temperatures up to a certain point where white layer forms. At 470°C the growth rate is constant where presumably γ -Fe₄N is present while at 520° this constancy appears in the presence of ϵ -Fe₃N. At 570° C the growth rate continues to increase; but not linearly, even when γ -Fe₄N is present and constancy appears well ahead of the presence of ϵ -Fe₃N phase. The diffusivities of nitrogen in alpha-iron at different nitriding temperatures, calculated for this steel using experimentally observed $r[X]$ values and the growth rates in the internal nitriding model, are about 50% lower than those for pure iron. The presence of grain boundary cementite and/or the counterflow of carbon from the carburized layer in the nitrided steel is proposed to oppose the diffusion of nitrogen, and the internal nitriding model; therefore, cannot be used for any quantitative prediction in steels.

12:00 PM
WETTING AND INTERFACIAL REACTIONS BETWEEN SNPB SOLDER AND BULK Ni SUBSTRATE: *Kwan Hoe Han*¹; Soong Joon Jeon¹; ¹Yeungnam University, Department of Metallurgical and Materials Engineering, 214-1 Dae-Dong, Kyongsan, Kyongbuk 712-749 Korea

Ni has been being widely used as a Barrier-coating layer, in microelectronics industries, for making chip mount and lead frame Interconnects through soldering. In spite of the technological importance, however, the soldering reactions between the molten solder and Ni, were not fully explored. In an effort to obtain better understanding of the wetting and interfacial reactions between eutectic SnPb solder and Ni an experimental work has been carried out in this study. We used pure Ni plate and eutectic, SnPb solder beads as a substrate and solder, respectively. The experiments were carried out for various reflow

times in mildly activated resin (type RMA) flux at temperatures between 200 and 250°C. The morphology and microstructural observation and the phase analysis were performed using optical and scanning electron microscope equipped with EDX. The spread radius and the wetting angle Cross-sectional microstructural examination showed that the intermetallic compounds forming at the interface possess a rod-shaped morphology, being different from the same phase formed in the solid state. The morphology and the growth kinetics of the compounds will be presented as well as the wetting/spreading characteristics.

12:20 PM

TITANIUM CARBIDE WHISKERS SYNTHESIZED USING METHANE AS A CARBON SOURCE: T. Hashishin¹; N. Tsukamoto¹; Y. Kaneko¹; ¹Ritsumeikan University, Department of Chemistry, Faculty of Science and Engineering, Shiga Prefecture, Kusatsu-shi, 1-1-1, Noji-Higashi

Titanium carbide (TiC) whiskers were deposited on the outer surface of a graphite tube and on a graphite boat in 10vol% methane gas stream following a heat treatment at 1000-1400°C. This temperature range was broader and lower than the 1250-1400°C range in which TiC whiskers were obtained when 5vol% methane gas was used in a thermal-decomposition-reduction-carbonization reaction. The lattice constant of TiC whiskers deposited on the outer surface of a graphite tube after the heat treatment at 1400°C was 0.432084 ± 0.007 nm, indicating that this TiC had a stoichiometric composition. The cylindrical whiskers formed at 1050-1200°C were considered to have been grown via a vapor-solid (VS) growth mechanism. Because the side faces of the rectangular prismatic whiskers formed at 1300-1400°C were dented, whisker growth in the radial direction may be caused by the layer stacking growth mechanism (a VS mechanism) due to two-dimensional nucleation on the {110} plane.

12:40 PM

POLYSILOXANE MODIFIED EPOXY - OLIGOIMIDE COMPOSITES: A. Ashok Kumar¹; M. Alagar¹; S. Anand Kumar¹; K. P. O. Madesh¹; ¹Anna University, Department of Chemical Engineering, Chennai 600 025 India

A novel hybrid variety of polydimethylsiloxane modified epoxy - oligoimide matrix resin systems have been developed from diglycidyl ethers of bis phenol-A and hydroxyl terminated polydimethylsiloxane with 3-aminopropyltriethoxysilane as a crosslinking agent. The objective of development of modified epoxy resin system is to improve the impact, thermal, dielectric and aging characteristics of conventional epoxy systems and to use them for the fabrication of high performance advanced composites for engineering and aerospace applications. The different types of curatives viz. aromatic bis(maleimide)-diaminodiphenylmethane oligomers having multifunctional amine groups are synthesized through the Michael addition reaction. The amine terminated oligomers are used for the curing of polydimethylsiloxane modified epoxy resin and the results are compared with those of conventional aromatic amine cured epoxy systems. The mechanical, electrical, thermal and aging characteristics of cured siliconized epoxy systems have been carried out as per ASTM standards. The optimum siloxane introduction into epoxy resins to achieve reasonable impact characteristics have been ascertained from experimental data. The siloxane introduction influences and improves the impact, dielectric, thermal and aging characteristics of the cured matrix systems and reinforced composites according to the percentage composition of siloxane content and the nature of curative involved.

PROCESSING & PROPERTIES OF ADVANCED STRUCTURAL CERAMICS: Advanced Structural Ceramics: Properties

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Rajiv S. Mishra, University of California, Dept of Chemical Engineering & Materials Science; Amiya K. Mukherjee, University of California, Dept of Chemical Engineering & Materials Science

Wednesday AM Room: London
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Arvid Pasto, Oak Ridge National Laboratory, Director, Oak Ridge, TN 37831-6062 USA

8:30 AM KEYNOTE

WHAT MAKES SILICON NITRIDE WORK: Sheldon M. Wiederhorn¹; ¹National Institute of Standards and Technology, MSEL Bldg 223 Room B309, Gaithersburg, MD 20899 USA

Silicon nitride has now reached the stage where it is an economic material. There are some areas where silicon nitride is accepted commercially (bearings, seals etc.), others that look promising (automobile valves) and still others that look like a promise on the horizon (turbine blades and nozzles). In this talk, I address the subject from the point of view of these practical applications and discuss those property that make the material work. Silicon nitride is a family of materials, and the optimum microstructure is application sensitive. The toughness-strength relation is the critical parameter in valves for example, where high toughness and high strength result in high reliability and long life. In wear applications material parameters are selected to avoid the formation of Hertzian cracks under sliding conditions. Now the properties that control distributed damage in silicon nitride are important. Short crack toughness is now the important design parameter. At high temperature, creep dominates the mechanical behavior. The damage that eventually leads to failure is generated by deformation and oxidation. Now the refractoriness of the intergranular phase that bonds the silicon nitride grains together is the dominate property that leads to low creep rates and long life. In the course of the lecture, we consider economic factors, since these are the deciding factors as to whether silicon nitride is to be used in a given application.

9:10 AM INVITED PAPER

CERAMIC LAMINATES WITH THE FIBROUS MONOLITH ARCHITECTURE: B. King¹; John W. Halloran¹; R. Trice¹; D. Kovar²; G. Hilmas³; ¹University of Michigan, Materials Science and Engineering Department, 2205 H.H. Dow Bldg., Ann Arbor, MI 48109-2125 USA; ²University of Texas, Austin, TX USA; ³Advanced Ceramics Research Inc., Tucson, AZ USA

Fibrous monoliths are two phase monolithic ceramics fabricated from powders using coextrusion methods to create a woody fibrous texture of strong silicon nitride cells and weak boron nitride cell boundaries. With this texture, the fibrous monoliths fracture gracefully, splintering like wood. Uniaxially aligned Si₃N₄/BN fibrous monoliths have peak flexural strengths around 600 MPa and retain about half of their load bearing ability after fracture initiates, with apparent fracture energies around 6000 J/m². Multiaxial materials have about half this strength and fracture energy, but have quasi-isotropic mechanical properties. This paper will discuss the mechanics of these materials, and the influence of the strong cell/weak cell boundary architecture

on properties, elevated temperature properties, and behavior of turbine engine components.

9:40 AM INVITED PAPER

OXIDE LAMINATED COMPOSITES WITH GRACEFUL FAILURE: *Waltraud M. Kriven*¹; ¹Univ. of Illinois at Urbana-Champaign, Materials Science and Engineering, Urbana, IL 61801 USA

A new "transformation weakening" mechanism for inducing debonding in an oxide interphase is introduced. It is based on inducing a displacive phase transformation accompanied by a significant unit cell volume contraction and/or shape change, in a coating around a reinforcement or between laminates of a ceramic matrix. In a thermally-induced transformation, all interphases are pre-transformed before the approach of a crack, with some consequent loss of overall strength of the material. In the ideal, shear-stress induced case, an oncoming crack induces a transformation in its immediate environment as necessary, with strength only minimally reduced throughout the bulk. Maximum toughening may then be achieved, since the propagating crack needs to do work to overcome the nucleation barrier and cause transformation, as well as the onset of other synergistic toughening mechanisms. A model laminate system has been designed and fabricated with a cristobalite (SiO₂) interphase between mullite (3Al₂O₃*2SiO₂) / cordierite (2MgO*2Al₂O₃*5SiO₂) mixed-matrix laminates. The mixed matrix optimized the sinterability of the laminate system, as well as the thermal expansion mismatch between the matrix and interphase layers. In pure SiO₂ the beta (cubic) to alpha (tetragonal) transformation occurs at 265°C on cooling, with a volume change of -3.2 %. For better control of the critical particle size for transformation, the SiO₂ was Al and Ca-doped. Depending on the interphase grain size then, the cristobalite interphase underwent either predominantly thermal or shear-induced transformation, exhibiting characteristics of graceful failure.

10:10 AM BREAK

10:20 AM INVITED PAPER

INTERFACE CONTROL FOR DURABLE CERAMIC COMPOSITES: *Ronald J. Kerans*¹; ¹Air Force Research Laboratory, Materials Directorate, AFRL/MLLN, Wright-Patterson AFB, OH 45433-7817 USA

Interest in ceramic composites is a consequence of their potential for dramatically improved damage tolerance as compared to monolithic ceramics while delivering similar high temperature properties. The remarkable toughening effect resulting from proper combination of three brittle phases in the form of fiber, coating and matrix is a consequence of a particular sequence in the development of damage. It is understood that there are both beneficial and detrimental ways for damage to develop; hence, successful design of composites requires the management of the progression of such damage. The situation is greatly complicated by the fact that the most productive uses of ceramic composites are dependent upon the development of fiber coatings that not only promote the desired interfacial failure, but satisfy the additional requirement of being oxidation resistant. Identification of suitable materials and their evaluation, development and incorporation into composites is key to the development of broadly useful composites. These and other issues related to the development of ceramic composites will be discussed with the intention of identifying topics of interest for future research.

10:50 AM INVITED PAPER

MECHANICAL PERFORMANCE OF POROUS-MATRIX ALL-OXIDE CERAMIC COMPOSITES: *F. W. Zok*¹; *J. A. Heathcote*¹; *X.-Y. Gong*¹; *J. Yang*¹; *U. Ramamurty*²; *C. G. Levi*¹; *F. A. Leckie*³; ¹University of California, Materials Department, Santa Barbara, California 93106 USA; ²Nanyang Technological University, School of Mechanical and Production Engineering Singapore; ³University of California, Mechanical and Environmental Engineering, Santa Barbara, CA 93106 USA

The conventional strategy for imparting high toughness and damage tolerance in fiber-reinforced ceramic composites involves the use of weak interphases between fibers and matrix, providing a path for crack deflection. An alternate approach for promoting such crack deflection is to utilize a highly-porous matrix that is well-bonded to the fibers. Because of its low elastic modulus and low fracture toughness, the matrix acts as the crack-deflecting medium, analogous to the weak interphase in conventional (dense-matrix) composites. The present paper will highlight recent progress in the development and performance of porous-matrix all-oxide ceramic composites. The emphasis will be on the mechanisms and mechanics associated with the in-plane tensile properties, both in the absence and in the presence of holes or notches. To provide a proper perspective, comparisons will be drawn with the behavior of conventional SiC-based composites with weak fiber-matrix interphases.

11:20 AM INVITED PAPER

ADVANCED CHARACTERIZATION TECHNIQUES FOR ADVANCED MATERIALS: *Arvid Pasto*¹; ¹Oak Ridge National Laboratory, Director, High Temperature Materials Laboratory, Oak Ridge, TN 37831-6062 USA

The HTML (High Temperature Materials Laboratory) is a U. S. Department of Energy User Facility, offering opportunities for in-depth characterization of advanced materials, specializing in high-temperature-capable structural ceramics. Available are electron microscopy for microstructural and microchemical analysis, equipment for measurement of the thermophysical and mechanical properties of ceramics to elevated temperatures, x-ray and neutron diffraction for structure and residual stress analysis, and high speed grinding machines with capability for measurement of component shape, tolerances, surface finish, and friction and wear properties. This presentation will focus on structural materials characterization, illustrated with examples of work performed on heat engine materials such as silicon nitride, industrial refractories, metal-and ceramic-matrix composites, and structural alloys. * Research sponsored by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program, Oak Ridge National Laboratory, managed by Lockheed Martin Energy Research Corp. for the U.S. Department of Energy under contract number DE-AC05-96OR22464.

11:50 AM

ELECTRON MICROSCOPE INVESTIGATION OF SILICON CARBIDE FIBER REINFORCED SILICON CARBIDE COMPOSITES: *Mani Gopal*¹; *Gareth Thomas*¹; ¹Department of Materials Science and Mineral Engineering, University of California, Berkeley CA 94720 and, Center for Advanced Materials, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA

One class of silicon carbide (SiC) composites is that reinforced with SiC fibers. The fibers are coated with boron nitride (BN) to improve the mechanical properties, by contributing to crack deflection and fiber pull-out. Transmission electron microscopy has been used to characterize the coated fibers and the fiber-reinforced composites. The BN coating on the fiber has been found to be textured, with its basal plane parallel to the surface of the fiber. This texture permits easy deflection of the crack as BN readily cleaves along its basal plane. Analysis of the composite indicates that during processing, interfacial reaction occurs between the fiber and the Si-C matrix. Further, EELS imaging coupled with conventional imaging shows the presence of elemental silicon saturated with carbon. This implies that the reaction between molten silicon and carbon, used to form the SiC matrix, is incomplete. The mechanisms by which this occurs, and steps to improve the processing will be presented. The authors thank T. Kameda of Toshiba for the specimens, NCEM for their cooperation and DOE for the funding.

DAMAGE PROCESSES IN ADVANCED MATERIALS: Intermetallics and Hydrogen Effects

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee, Corrosion and Environmental Effects Committee, Physical Metallurgy Committee

Program Organizers: Russ H. Jones, Pacific Northwest Natl. Lab., Richland, WA 99352 USA; Richard Ricker, NIST, Materials Rm B254, Gaithersburg, MD 20899; Norman S. Stoloff, Rensselaer Polytechnic Inst., MS&E Dept., Troy, NY 12190-3590 USA

Wednesday PM Room: Paris C
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: R. H. Jones, Pacific Northwest National Laboratory, Richland, WA 99352 USA; N. S. Stoloff, Rensselaer Polytechnic Institute, Troy, NY 12180-3590 USA

2:00 PM INVITED PAPER

FATIGUE DAMAGE IN INTERMETALLICS: *N. S. Stoloff*¹; ¹Rensselaer Polytechnic Institute, Materials Science and Engineering Department, Troy, NY 12180-3590 USA

Studies of fatigue damage in intermetallics have lagged behind investigations of tensile ductility and creep behavior. However, there is now a sizeable body of stress-controlled fatigue crack growth data in aluminides and silicides (especially Fe₃Al, TiAl and MoSi₂) as well as strain-controlled low cycle fatigue data for Ni₃Al, NiAl and TiAl. This paper will summarize recent observations of fatigue damage in these intermetallics and some of their composites, and will attempt to correlate microstructure and observed fatigue resistance. In addition, the influence of external environments on fatigue crack growth behavior will be described.

2:40 PM

IMPACT DAMAGE AND HIGH TEMPERATURE FATIGUE BEHAVIOR OF Γ -TiAl: *T. S. Harding*¹; J. W. Jones¹; T. M. Pollack²; M. P. Rubal²; P. S. Steif²; ¹University of Michigan, Ann Arbor, MI 48019 USA; ²Carnegie-Mellon University, Pittsburgh, PA 15213 USA

The elevated temperature fatigue response of gamma titanium aluminide following impact has been investigated. Axial fatigue specimens machined from cast Ti-47.9Al-2.0Cr-1.9Nb and Ti-47.3Al-2.2Nb-0.5Mn-0.4W-0.4Mo-0.23Si alloys, were impacted at low speeds with a 60Y; wedge shaped indenter under controlled conditions in order to simulate manufacturing related damage. Additional specimens from the same alloys were impacted at high speeds with a 1.6 mm steel ball bearing near the specimen edge to simulate leading edge FOD/DOD impacts on turbine blades. Damage was characterized and related to different impact parameters in both scenarios. A step-loading fatigue test procedure is used to determine the fatigue failure stress of the impacted specimens at 650Y. A threshold based fracture mechanics approximation of the failure stress is presented for both impacting conditions. Fractography has been conducted to determine the extent of the initial impact damage within the specimen, and to identify the mechanisms responsible for the propagation of the crack from the impact site. These results are used to verify the threshold based approximation.

3:00 PM

MICROSTRUCTURAL EVOLUTION, CREEP AND TENSILE BEHAVIOR OF A Ti13Al-39Nb BETA+ORTHORHOMBIC ALLOY: *C. J. Boehlert*¹; B. S. Majundar¹; V. Seetharaman¹; D. B. Miracle²; ¹UES Inc., 3301 Dayton-Xenia Road, Dayton, OH 45420-1894 USA; ²Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

The microstructural evolution, creep and tensile deformation behavior of a Ti-13Al-39Nb(at.%) alloy were studied. Monolithic sheet materials were produced through conventional thermomechanical processing techniques comprising nonisothermal forging and pack rolling. TEM studies showed that, depending on the heat-treatment schedule, this alloy contains two constituent phases including: (disordered body-centered cubic) and O (ordered orthorhombic based on Ti₂AlNb). Heat treatments above 800Y, followed by water quenching, resulted in fully- β microstructures. Below 800Y, fine O-phase needles precipitated within the β grains. The fully- β microstructure exhibited a room temperature elongation of more than 27% and a yield strength of 550 MPa. The O+ β microstructures, containing approximately 30 volume percent O, exhibited elongations of more than 12% and yield strengths up to 810 MPa. Metallographic observations revealed that slip was compatible between the two phases. Constant load tensile creep experiments were conducted in the temperature range 650-705Y and stresses were varied between 35-172 MPa. Measured creep exponents and activation energies suggested that the creep mechanisms were dependent on stress. For applied stresses less than 125 MPa, the creep exponents were between 1.6-2.0. For higher stresses, the stress exponents were between 3.5-7.2. The calculated activation energies for the low-stress regime were approximately half those calculated for the high-stress regime. These data suggest that Coble creep operates at low-stress levels and dislocation climb is active at high stresses. The β grain size did not significantly influence the creep rates within the high-stress regime. Within the low-stress regime, grain size did influence the creep behavior and a low dislocation density was observed. Microstructural effects on the tensile properties and creep behavior are discussed in the light of existing models. Overall, the elevated and room-temperature properties of this O+ β alloy are quite attractive.

3:20 PM

MECHANISMS OF FRACTURE AND FATIGUE-CRACK PROPAGATION IN A SHAPE-MEMORY ALLOY TiNi: *A. L. McKelvey*¹; R. O. Ritchie¹; ¹University of California, Department of Materials Science and Mineral Engineering, Berkeley, CA 94720-1760 USA

The effect of stress-induced phase transformations upon fracture and fatigue-crack growth resistance in shape-memory and super-elastic TiNi (Nitinol) are investigated. Particular emphasis is placed on the damage processes during monotonic and cyclic crack growth as a function of temperature, microstructure, and environment. Resistance curve tests indicate that super-elasticity results in crack-tip shielding under monotonic loading. The degradation of crack-tip shielding during cyclic loading and its effect on crack-growth rates are examined. Tests are conducted at temperatures to correspond with non-transforming martensite, transforming martensite (i.e., shape-memory or thermoelastic), transforming austenite (i.e., super-elastic), and non-transforming austenite. As super-elastic Nitinol is a candidate material for cardiovascular stents, additional tests are being conducted to examine the role of crack-tip corrosion at 37Y in deionized water and Hank's solution (a simulated body fluid). The mutual competition of intrinsic microstructural damage mechanisms ahead of the crack tip, which promote crack advance, and extrinsic crack-tip shielding (e.g., transformational toughening and closure) behind the tip, which act to impede it, is investigated.

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HYDROGEN ENVIRONMENT-ASSISTED CRACKING IN BETA-TITANIUM: *B. P. Somerday*¹; R. P. Gangloff²; ¹Sandia National Laboratories, Livermore, CA 94550 USA; ²University of Virginia, Department of Materials Science, Charlottesville, VA USA

The objective of this research is to determine the critical metallurgical factor(s) and associated embrittlement mechanism that govern hydrogen environment-assisted cracking (HEAC) in beta-titanium alloys. The metallurgical variable(s) that govern intergranular (IG) HEAC of Ti-8V-6Cr-4Mo-4Zr-3Al (Beta-C) in aqueous NaCl are examined by fracture mechanics experiments on both immune and susceptible microstructures; including solution heat treated, cold worked, isothermally aged, and double-aged conditions; coupled with high-resolution imaging and analytical electron microscopy. Segregated Si at beta grain boundaries is the likely requisite for IG HEAC in Beta-C. The fracture mechanics measurements and fractographic results for susceptible isothermally aged and double-aged Beta-C microstructures are consistent with effects of heat treatment time and temperature on solute segregation-induced hydrogen embrittlement, and both small-probe TEM EDS and Auger measurements of grain boundary composition reveal elevated Si concentrations. Immunity to HEAC may be achieved in high-strength beta+alpha microstructures by a two-step heat treatment, where initial high-temperature aging for short times depletes Si from the single-phase beta matrix by forming Ti-Zr silicide particles, then subsequent low-temperature aging precipitates alpha. The hydrogen embrittlement mechanism that operates at beta grain boundaries in HEAC-susceptible Beta-C is probed using a field-emission gun SEM, which allows resolution of fine IG fracture surface features. Intergranular cracking of Beta-C in aqueous NaCl is likely from hydrogen-assisted decohesion of grain boundaries, based on the presence of contiguous featureless areas on all fracture surface facets that suggest cracking along a continuous beta/beta interface path. The complex beta+alpha microstructure causes a variety of distributed fine-scale features on decohered grain boundary surfaces. Neither fine dimples nor slip offsets are observed on the surface of most IG facets, indicating that hydrogen-enhanced plasticity does not operate in Beta-C.

DYNAMIC BEHAVIOR OF MATERIALS: Dynamic Behavior III

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

Wednesday PM Room: Sydney
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: John J. Gilman, University of CA, Materials Science and Engineering, Los Angeles, CA 90095 USA; Marc A. Meyers, University of CA, Dept. of AMES, La Jolla, CA 92093-0411 USA

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LOW-VELOCITY-TO-HYPERVELOCITY PENETRATION TRANSITION FOR IMPACT CRATERS IN METAL TARGETS:

Lawrence E. Murr¹; S. A. Quinones¹; *E. T. Ferreyra*¹; A. Ayala²; O. L. Valerio¹; ¹The University of Texas at El Paso, Dept. of Metallurgical

and Materials Engineering, Engineering Science Complex M-201, El Paso, TX 79968-0520 USA

Projectile/target behavior for 1100 Al Cu, soda-lime glass/Cu, soda-lime glass/1100 Al, ferritic stainless steel/Cu, and ferritic stainless steel/1100 Al for a range of spherical (3.18 mm diameter) projectiles at impact velocities ranging from 0.8 km/s to ~6 km/s has been examined by light metallography, SEM, and TEM. At a reference velocity of 1 km/s, the crater depth/crater diameter ratio (p/D_c) is observed to be linearly related to $(\rho_p/\rho_t)^{1/2}$ and $(E_p/E_t)(\rho_p/\rho_t)^{1/2}$; and to vary from about 0.2 to 2.95. The hypervelocity threshold value for p/D_c ($u_p > 5$ km/s) is also shown to be linearly related to these functionalities; ranging from $p/D_c = 0.4$ for the 1100 Al/Cu system and 0.85 for the ferritic stainless steel/1100 Al system.

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INTERFACIAL FRICTIONAL RESISTANCE OF SLIDING INTERFACES AT HIGH SLIP SPEEDS AND HIGH TEMPERATURES:

*Vikas Prakash*¹; Mohammad A. Irfan¹; ¹Case Western Reserve University, Department of Mechanical and Aerospace Engineering, 10900 Euclid Avenue, Cleveland, OH 44106-7222. USA

In the present study, a plate impact pressure-shear loading device is used to investigate the frictional characteristics of sliding interfaces at high slip speeds, high pressures, high temperatures, and time varying normal and shear tractions. The experimental configuration employed represents a significant improvement over conventional dynamic friction experiments by allowing the control of the interfacial tractions through the use of combined pressure-shear waves instead of manipulating actuator motion. The use of planar pressure shear waves enables critical frictional parameters such as the interfacial slip resistance, slip speeds, interfacial temperatures, and the applied normal pressures to be interpreted by the framework of one-dimensional plane wave analysis. Results will be presented for pressure-shear friction experiments conducted on a Ti-6Al-4V/steel tribo pair.

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DYNAMIC BEHAVIOR OF Zr-2.5Nb PRESSURE TUBE MATERIAL: *Darren D. Radford*¹; Mike J. Worswick²; ¹Atomic Energy of Canada, Reactor Core Integrity, WL, Pinawa, Manitoba R0E-1L0 Canada; ²Carleton University, Mechanical and Aerospace Engineering, Ottawa, Ontario K16-5B6 Canada

The current design of Canada Deuterium Uranium (CANDU) nuclear reactors utilize cold-worked Zr-2.5Nb pressure tubes to contain the uranium fuel bundles and the heavy water (D2O) coolant [1]. The tubes are required to operate at temperatures in the range of 250 to 310°C, an internal pressure up to 13 MPa, and a fast ($E > 1$ MeV) neutron flux of 2.5×10^{17} n/m²/s. In certain hypothetical accident scenarios it is postulated that a pressure tube could burst prior to the detection of a through-wall longitudinal crack and the shut-down of the reactor. Assuming also, as a worst-case scenario, that the surrounding calandria tube fails, the longitudinal crack could continue to propagate down the length of the tube. In this situation the pressure tube would deflect vertically due to the release of the pressurized coolant, thereby dynamically loading the ends of the pressure tube in tension. Depending on the final crack geometry and thermal-hydraulic conditions, the ruptured tube could impact surrounding fuel channels causing further in-core damage. Preliminary analyses indicated that plastic deformation at the ends of a rupturing pressure tube would occur at strain rates in the range of 500 to 3000 s⁻¹. In order to accurately simulate pressure tube deformation and the consequences of pressure tube rupture, it is necessary to have an accurate description of the stress-strain behavior of Zr-2.5Nb pressure tube material for the relevant conditions. Although there is a considerable amount of data related to the deformation of Zr [2-6], and Zr-Nb alloys [7-8], it was either obtained at conditions not applicable to pressure tube rupture scenarios, or determined from material with considerably different crystallographic structure and texture. In this investigation, results from high strain rate tensile tests conducted on non-irradiated [9] and irradiated Zr-2.5Nb pressure tube material using a direct-tension version of the split Hopkinson bar apparatus [10, 11] are reported. Specimens were manufactured from both the longitudinal and

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transverse tube orientations and tested at temperatures in the range of 20 to 300°C and strain rates in the range of 500 to 3000 s⁻¹. The irradiated specimens were manufactured from pressure tubes that had been in service for up to 18 years and had received a total neutron fluence of up to 7.4×10^{25} n/m². Results from tests performed on non-irradiated material at 20°C demonstrate that the pressure tube material remains anisotropic at high strain rates [9]. As observed in low strain rate tests [12], the ultimate tensile strength (UTS) in the transverse direction is higher than in the longitudinal direction. The stress-strain behavior for Zr-2.5Nb pressure tube material is quite different depending on the specimens orientation, due to the relative abundance of slip systems in the longitudinal direction and lack of primary slip systems in the transverse direction [13]. The effect of increasing the strain rate was to increase the strength in both directions. The values of flow stress at high strain rates increased approximately 40% above quasi-static values for both specimen orientations. Also, as seen in a number of materials [14] a bilinear dependence of flow stress on logarithm of strain rate was observed, with the strain rate sensitivity being higher at high strain rates. At elevated temperatures, the pressure tube material remained anisotropic and the strength increased above quasi-static values as the strain rate was increased for specimens tested in both directions. The increase in strength was approximately in the same proportions as observed in the 20 °C tests and a bilinear dependence of flow stress on logarithm of strain rate was also exhibited. UTS values obtained at high strain rates and 300 °C were approximately 20% lower than those obtained at high strain rates and 20 °C, for both specimen orientations. Previous results from tensile tests performed on irradiated Zr-2.5Nb pressure tube material at quasi-static strain rates [8] demonstrated that irradiation increases the strength and decreases the ductility in both the longitudinal and transverse directions. These changes occurred at low levels of fluence (1×10^{25} n/m²) and did not increase further as fluence levels increased to approximately 7×10^{25} n/m². The overall stress-strain responses, as a function of tube orientation, were consistent with that of the non-irradiated material, and UTS values remained higher in the transverse direction. The effect of irradiation on the tensile properties at high strain rates was to increase the strength and decrease the ductility for all test temperatures. The reduction in ductility at high strain rates, however, was less than that observed at quasi-static strain rates. As observed in the quasi-static tests, the increase in strength due to irradiation took place at low fluences and did not significantly change for fluence levels up to approximately 7×10^{25} n/m². A bilinear dependence of flow stress on logarithm of strain rate was also observed in the irradiated material for strain rates ranging from 1×10^{-4} to 3×10^3 s⁻¹.

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DYNAMIC HARDNESS AND RATE SENSITIVITY IN METALS: *Ghatu Subhashi*¹; Brian J. Koepfel¹; ¹Michigan Technological University, Mechanical Engineering-Engineering Mechanics Department, 1400 Townsend Drive, Houghton, MI 49931 USA

Indentation hardness is one of the simplest and the most commonly used measures for quickly characterizing material response under static loads. Hardness may mean resistance to cutting to a machinist, resistance to wear to a tribologist, or a measure of flow stress to a design engineer. In this simple technique, a predetermined force is applied to an indenter for 5-30 seconds causing it to penetrate a specimen. By measuring the load and the indentation size, a hardness value is determined. However, the rate of deformation during indenter penetration is of the order of 10⁻⁴ s⁻¹. In most practical applications, such as high speed machining or impact, material deformation occurs at strain rates in excess of 10³-10⁵ s⁻¹. At such high strain rates, it is well established that the plastic behavior of materials is considerably different from their static counterpart. For example, materials exhibit an increase in their yield stress, flow stress, fracture stress, and fracture toughness at high strain rates. Hence, the use of static hardness as an indicator of material response under dynamic loads may not be appropriate. Accordingly, a simple dynamic indentation hardness tester is developed for characterizing materials at strain rates similar to those encountered in dynamic situations. The experimental technique uses elastic stress wave propagation phenom-

ena in a slender rod. The technique is designed to deliver a single indentation load of 100-200 microseconds duration. Similar to static measurements, the dynamic hardness is determined from the measured load and indentation size. Hardness measurements on a range of metals have revealed that the dynamic hardness is consistently greater than the static hardness. The percent increase in hardness under dynamic conditions is found to be similar in magnitude to the percent increase in yield stress under uniaxial compression. Therefore, it is suggested that the current technique can be used to quickly assess the rate sensitive nature of engineering materials. To further characterize the plastic strains within the indentation volume, static microhardness measurements were performed within this region. The contours of microhardness values thus generated give a measure of accumulated plastic strains beneath the indenter. It was observed that this plastic zone is typically smaller under dynamic conditions compared to static loading for rate sensitive materials. To assess the influence of the elastic modulus, yield stress, and work hardening coefficient on the induced plastic volume, finite element simulations were performed using the explicit finite element code LS-DYNA3D. The parametric study revealed that the yield stress has the most significant influence on the size and shape of the plastic zone. The above microstructural and numerical results can be used as guidelines for proper selection and design of engineering materials in applications involving high strain rate loading. Moreover, the contours of microhardness variation can be used to verify the suitability of analytical models developed for characterizing the deformation behavior of engineering materials under complex three dimensional loading conditions.

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DAMAGE EVOLUTION OF DYNAMIC DEFORMATION OF SILICON CARBIDE: *C. James Shih*¹; Marc A. Meyers²; Vitali F. Nesterenko²; ¹Ceradyne Inc, 3169 Redhill Avenue, Costa Mesa, CA 92626 USA; ²UCSD, Materials Science, Mail Code 0411, 9500 Gilman Drive, La Jolla, CA 92093-0411 USA

Damage evolution was investigated in silicon carbide by subjecting it to dynamic deformation in (a) a compression Hopkinson bar (compressive stresses of 5 GPa), and (b) high-velocity impact under confinement (compressive stresses of 19 to 32 GPa) by a cylindrical tungsten alloy projectile. Considerable evidence of plastic deformation, as dislocations and stacking faults, was observed in the fractured specimens. A polytype transformation was observed through a significant increase in the 6H-SiC phase at compressive stresses higher than 4.5 GPa (in the vicinity of the dynamic compressive failure strength). Profuse dislocation activity was observed in the frontal layer in the specimen recovered from the projectile impact. The formation of this frontal layer is proposed to be related to the high lateral confinement, imposed by the surrounding material. The microstructural defects and evolution were found to be dependent on the concentration of boron and aluminum, which were added as sintering aids. Finite element calculations showed that tensile stresses are generated due to elastic compatibility strains. Two mechanisms are proposed for the initiation of fracture: dilatant cracks and Zener-Stroh cracks. Dilatant cracks are induced by mismatch in the effective elastic moduli between two adjacent grains, leading to internal tensile stresses and creating transgranular fracture. Zener-Stroh cracks are nucleated by the piled up dislocations along grain boundaries, and result in intergranular fracture. The high dislocation density observed in the impacted specimen is consistent with existing models of microplasticity. This research is supported by the U.S. Army Research Office through AASERT (DAAH04-94-G-0314) and MURI (DAAH04-96-1-0376) programs, and the National Science Foundation through the Institute for Mechanics and Materials at the University of California, San Diego.

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INFLUENCE OF COLD ROLLING ON PLASTIC RESPONSE OF PM AND CVD RHENIUM: *Ghatu Subhashi*¹; Brian J. Koepfel¹;

¹Michigan Technological University, Mechanical Engineering-Engineering Mechanics Department, 1400 Townsend Drive, Houghton, MI 49931 USA

Plastic response of rhenium (Re) produced by powder metallurgy (PM) and chemical vapor deposition (CVD) was investigated under uniaxial compression at a range of strain rates. The PM Re was also cold rolled by 50% and 80% reduction (denoted as PM50 and PM80, respectively) in thickness. Both cold rolled and CVD Re have been found to have a strong basal texture. Rhenium exhibited a two stage hardening in its plastic response. It revealed a high strain hardening rate and a strong strain rate sensitivity of flow stress. Increased cold work in the PM Re resulted in an increase in the initial yield stress with a concurrent reduction in the hardening rate and failure strain. CVD Re exhibited a distinctly higher hardening rate and ultimate flow stress than the above cold rolled PM Re. After accumulating a certain amount of plastic strain, the deformation in the specimens localized leading to formation of shear bands at an angle to the loading axis. Fracture immediately followed the shear bands. Twinning was found to be a dominant deformation mode in both PM and CVD Re. Extensive twinning was observed in larger grains of CVD Re. Twins in fine grained PM specimens were less common and occurred primarily in the regions close to the shearband. Based on the experimental results and microscopic investigations, the micromechanisms of deformation responsible for the observed behavior of Re will be discussed.

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QUASI-STATIC AND DYNAMIC CRUSHING OF AN ALUMINUM CLOSED CELL FOAM: *Ian W. Hall*¹; *Mustafa Guden*²; ¹Department of Mechanical Engineering, University of Delaware, Spencer Laboratory, Newark, DE 19716 USA; ²Izmir Institute of Technology, Gaziosmanpasa Bulvarı No:16, Cankaya, Izmir Turkey

An aluminum 6061 closed cell foam has been compression tested at quasi-static (1.5x10⁻³s⁻¹) and high strain rates (400 and 1700 s⁻¹) in order to assess strain rate effect on the flow stress /strain behavior of the foam. Experimental results have shown that increasing strain rate from quasi-static to dynamic increased the flow stress and hence energy absorption capacity of the foam. Application of scaling relations to the flow stress vs. relative density of the foam suggested that increased flow stress at high strain rates compared to quasi-static rates was due to the strain rate sensitive flow stress behavior of the foam alloy. Microscopic observations clearly showed that compression of the foam resulted in crushing of the cell walls by buckling and tearing in localized regions and deformation was progressed from localized through the undeformed regions of the foam. The final deformation stage, densification, was the result of cell touching which significantly increased the flow stress of the foam at relatively large strains.

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AN ANALYSIS OF THE CRSS FOR TWINNING IN LOW-SYMMETRY METALS: *George C. Kaschner*¹; *Thomas A. Mason*²; *George T. Gray*¹; ¹Los Alamos National Laboratory, MST-8, M/S G755, Los Alamos, NM 87545 USA; ²Los Alamos National Laboratory, MST-6, M/S G755, Los Alamos, NM 87545 USA

Previous work has demonstrated that the deformation of low-symmetry metals is a balance of accommodation by dislocation slip and deformation twinning. Zirconium was selected as the model material to explore the correlation of local crystallographic orientation and the CRSS for twinning. Samples were tested in cryogenic quasi-static conditions and characterized using the recently developed automatic electron backscatter diffraction techniques of OIM. This analysis will attempt to correlate the volume of twinned matrix and the accommodation of strain in textured h.c.p. metals.

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INFLUENCE OF ALLOYING ELEMENTS ON THE STRAIN RATE AND TEMPERATURE DEPENDENCE OF THE FLOW STRESS OF STEELS: *Volker Schulze*¹; *Otmar Voehringer*¹; ¹Universitaet

Karlsruhe (TH), Institut fuer Werkstoffkunde I, Kaiserstr. 12, Karlsruhe, FRG 76128 Germany

The relation between flow stress σ , strain rate $\dot{\epsilon}$ and temperature T of metallic materials is controlled for $\dot{\epsilon} < 10^4 \text{s}^{-1}$ and $T < 0.3 T_m$ (T_m = melting temperature in K) by the thermally activated slip of dislocations over short-range obstacles. The quantitative description of the effect of strain rate and T on the flow stress is given by the constitutive equation $\sigma = \sigma_G + \sigma_o^* (1 - (T/T_o)^n)^m$ with G_o = free activation enthalpy, k = Boltzmann constant, $\dot{\epsilon}_o$ = strain rate constant, σ_G = athermal flow stress, σ^* = thermal flow stress at $T = 0$ K and m, n = exponents which characterize the force-distance shape of the short range obstacle. The properties of the constitutive equation were estimated for several plain carbon steels in normalized conditions as well as quenched and tempered alloyed steels based on their temperature dependence and strain rate sensitivity of the flow stress at different $\dot{\epsilon}$ -values and low temperatures. Increasing contents of soluted alloying elements lead to decreasing values of σ_o^* and ΔG_o . This effect is called pseudo-alloying-softening and depends strongly on type and content of the alloying element. It will be discussed from a microstructural point of view.

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STRUCTURAL TRANSITIONS IN ENSEMBLES OF MESODEFECTS AS MECHANISM OF SHEAR LOCALISATION UNDER IMPACT LOADING: *Oleg Borisovich Naimark*¹; ¹ICMM, Russian Acad.Sci., Phys.Foundation of Strength, 1 Acad.Korolev Str., Perm 614013 Russia

Surprising universality for viscosity value of shocked materials obtained first by Sakharov's group is explained. This property is caused by orientation transition in microshear ensemble under subjection of relaxation material ability to self-similar defect ensemble behaviour which corresponds to resonance excitation of solitary waves of orientation defect instability. The latter leads to strain localisation waves propagating with velocities determined by orientation transition kinetics. New nature of shear strain instability is shown. Phenomenology modification and minimum testing extension is proposed for dynamic problem studying. Conditions of resonance excitation of self-similar regimes are studied theoretically and experimentally for projectile-target interaction.

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EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Thermodynamics and Concluding Talk by J. W. Cahn

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

Wednesday PM Room: International Ballroom
 October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Sam Allen, MIT

2:00 PM INVITED PAPER

CLASSIFICATION OF PHASE TRANSFORMATIONS: *John A. L. Ågren*¹; ¹Royal Institute of Technology, Materials Science and Engineering, S-100 44, Stockholm 100 44 Sweden

Phase transformations may be classified in many different ways. The division introduced by Gibbs into homogenous and heterogeneous transformation as well as Ehrenfest division into first, second and higher order transformations are well known to all students in physics, chemistry and materials science. Other ways of classifying the transformations are based on the most important atomistic or molecular mechanisms, e.g. diffusional and displacive transformations. In the talk various classification methods are discussed and criticized from a practical as well as a more fundamental point of view. For example, it is shown that it is generally not meaningful to consider first and second order transformations as equivalent with heterogeneous and homogeneous transformations respectively.

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FROM NON-EQUILIBRIUM TO EQUILIBRIUM STUDIED BY CLUSTER VARIATION AND PATH PROBABILITY METHOD: *Tetsuo Mohri*¹; ¹Hokkaido University, Division of Materials Science and Engineering, Graduate School of Engineering, Sapporo 060-8628 Japan

Cluster Variation Method (CVM) has been recognized as one of the powerful theoretical tools to predict and to analyze phase equilibria in an alloy system. The combination with electronic structure calculations enable us to investigate the phase equilibria even from the first-principles. Path Probability Method (PPM) is the natural extension of the CVM to time domain, and it has been confirmed that the derived quantities in the long time limit exactly reproduce the equilibrium ones independently obtained by the CVM. Hence, the combination of the CVM and PPM is best suited to study time evolution and devolution behavior of microscopic quantities starting from non-equilibrium state towards the equilibrium state. Among the various calculations attempted, phase transition kinetics and relaxation kinetics are discussed.

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THE JOYS AND NEVER-ENDING CHALLENGES OF PARADIGM BUILDING; WILL OURS EVER BE FLAWLESS?: *John W. Cahn*¹; ¹NIST, Materials Science and Engineering Laboratory, Materials Building, Room A153, Gaithersburg, MD 20899 Xanadu

Fundamental ideas have played a central role in the long history of our field and led to many useful inventions and discoveries, even when they were later proven wrong. The scientific revolution has changed many of these ideas, and has even changed their nature. The pace of these changes is accelerating as our field keeps expanding. Paradigms provide a framework for the development of ideas, but ones created for fluids are severely challenged when applied to solids; ones advanced for metals may not survive without modification when applied to polymers or ceramics. New experimental and theoretical tools permit the asking of new questions and examination of previously inaccessible details. Some of the recent changes will be examined for the challenges they still pose.

FATIGUE BEHAVIOR OF TITANIUM ALLOYS: Application, Life Prediction and Design

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

Wednesday PM Room: Athens/Berlin
 October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: J. Larsen; D. Eylon

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APPLICATION AND LIFE PREDICTION OF TITANIUM ALLOYS IN AIRCRAFT GAS TURBINE ENGINES: *B. A. Cowles*¹; ¹Pratt & Whitney, P.O. Box 109600, MS 707-22, West Palm Beach, FL 33410-9600 USA

Since initial introduction in the 1950's, application of titanium alloys has steadily increased in aircraft gas turbine engines. The low density and high specific strength of titanium alloys have contributed significantly toward attainment of today's high thrust, light weight, extremely efficient engines. Today, titanium alloys comprise more than one-third of total engine weight, much of it in structurally critical parts such as fan and compressor rotors and airfoils, and engine mainframe structures. Materials processing, and structural design, durability and life prediction practices have continuously evolved to facilitate such applications. This paper presents an overview of current titanium applications in gas turbine engines, the associated structural and durability considerations, and areas that appear significant for future applications.

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APPLICATION OF Ti-ALLOYS AS COMPRESSOR DISKS AND BLADES: *D. Helm*¹; ¹MTU, Munchen, FRG 80976 Germany

Ti-alloys are widely used as materials for compressor discs, blades, vanes and housings in modern aero-engines due to their excellent strength-to-weight ratio. Their high corrosion resistance and good weldability are additional factors for their application as main com-

pressor components. A variety of different Ti-alloys have been developed in recent years and introduced into jet engines in order to fulfill the large spectrum of required mechanical properties. The main topic of the present paper is to describe the correlation between essential microstructural features of a number of Ti-alloys for compressor disc and blade applications and such mechanical properties as strength, creep and fatigue resistance, and crack propagation behavior. It will be shown that for different engine requirements the choice of the suitable alloy, as well as tailoring of microstructural features with the aim to achieve an optimized balance of relevant mechanical properties, is necessary. The influence of thermomechanical treatment on morphology and dimensions of phases, and the resulting correlation to mechanical properties, will be discussed in detail for selected Ti-alloys, i.e. Ti-6Al-4V, Ti-6Al-2Sn-4Zr-2Mo and IMI 834 (Ti-5.8Al-4Sn-3.5Zr-0.5Mo-0.7Nb-0.35Si). Additionally, effects of surface conditions caused by surface treatment (shot peening) or by service exposure (surface oxidation) will be presented.

2:50 PM INVITED PAPER

TITANIUM IN FATIGUE CRITICAL MILITARY AIRCRAFT STRUCTURES: *Franna Gillespie*¹; ¹The Boeing Company, Information, Space and Defense Systems; Military Airplanes, P.O. Box 3707, Seattle, WA 98124 USA

Titanium comprises a major portion of military aircraft structure, in a large part because of its temperature capability, corrosion resistance, and high specific strength. Today's military aircraft have increased performance requirements to the point that static strength in titanium is much less important than fatigue performance, fatigue crack growth and fracture toughness. A combination of analysis techniques and manufacturing processes are used by the designer to ensure that future military aircraft meet the customer's requirements. This paper describes the impact of fatigue analyses and designated manufacturing processes on titanium structure.

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FATIGUE CRACK INITIATION AND FATIGUE LIFE PREDICTION OF Ti-6Al-4V ELI: *M. Niinomi*¹; *T. Akahori*¹; ¹Toyohashi University of Technology, Department of Production Systems Engineering, 1-1, Hibarigaoka, Tempaku-cho, Toyohashi 441-8580 Japan

The specimen of Ti-6Al-4V ELI with Widmanstätten α structure fractures before adequate fatigue hardening because a lot of microstructure-cracks are easily formed while the specimen with equiaxed α structure fractures after adequate fatigue hardening. The mechanical properties, that is, tensile strength and hardness tend to increase clearly according to the fatigue steps in particular in the low cycle fatigue (LCF) region, while impact toughness and elongation show the reverse trend. The hardness far from the specimen surface is greater than that near the specimen surface at the early stage of LCF. Both hardnesses are however nearly equal each other at the latter stage of fatigue. It can be understood therefore that the dislocation multiplies near the specimen surface at the early stage of fatigue while at the latter stage of fatigue, the dislocation multiplies also near the center of the specimen. The dislocation multiplication will finally saturate in the whole specimen.

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DESIGN AND QUALIFICATION OF TITANIUM FOR DEEPWATER HIGH PRESSURE DRILLING RISERS: *J. Murali*¹; *M. M. Salama*²; *M. W. Joosten*²; ¹Norske Conoco AS, Tangen 7, P.O. Box 488, 4070 Randaberg, Stavanger 4001 Norway; ²Conoco Inc., 100 South Pine, P.O. Box 1267, Ponca City, OK 74603 USA

Titanium alloy Ti-6Al-4V ELI was selected for fabricating Heidrun's high pressure drilling riser because of its high specific strength, corrosion resistance, and favorable elastic properties. The qualification of this titanium alloy required assessing its resistance to hydrogen embrittlement and stress corrosion cracking due to seawater

with/without cathodic protection, evaluating its wear resistance against a rotating steel drill string, and studying the influence of service induced defects on fatigue and crack growth behavior when subjected to the operating environment. The paper presents an overview of the design requirement and the materials performance properties which were proposed, evaluated, specified and validated for the Ti-6Al-4V ELI used in this application.

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MICROSTRUCTURE & TEXTURE MODEL FOR THE MEAN STRESS SENSITIVITY OF HCF STRENGTH IN A+B TITANIUM ALLOYS: *Frederic Cohen*¹; *S. Ivanova*²; *R. D. Sisson*²; *R. R. Biederman*²; ¹Pratt & Whitney, Mail Stop 114-40, 400 Main Street, East Hartford, CT 06108 USA; ²Worcester Polytechnic Institute, 100 Institute Road, Worcester, MA 01609-2280 USA

The effect of microstructure and crystallographic texture on the mean stress sensitivity of the fatigue strength has been investigated for equiaxed and bi-modal microstructures in Ti-6Al-4V. Most microstructures exhibited a pronounced mean stress sensitivity. A quantitative model has been developed which accurately predicts the fatigue strength and its mean stress dependence as a function of the microstructure and texture characteristics of the $\alpha + \beta$ titanium alloys.

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FATIGUE CHARACTERISTICS OF BIOMEDICAL TITANIUM ALLOYS: *Mitsuo Niinomi*¹; ¹Toyohashi University of Technology, Department of Production Systems Engineering, 1-1, Hibarigaoka, Tempaku-cho, Toyohashi 441-8580 Japan

Fatigue characteristics and microstructure relation of various biomedical titanium alloys like Ti-5Al-2.5Fe, Ti-6Al-7Nb and Ti-6Al-4V ELI were investigated in air and simulated body environment, that is, Ringer's solution. Fatigue strength of Ti-5Al-2.5Fe with various microstructures is not degraded in ordinary Ringer's solution while that of stainless steel, 316L, degraded. However, fatigue strength of Ti-5Al-2.5Fe is degraded in Ringer's solution with N_2 gas flow, that is, low oxygen content. The long fatigue crack propagation rate, da/dN, of Ti-5Al-2.5Fe is accelerated in ordinary Ringer's solution when da/dN is related with nominal cyclic intensity factor range, ΔK , while da/dN in ordinary Ringer's solution is nearly the same as that in the air when da/dN is related with effective cyclic stress intensity factor range, ΔK_{eff} . The short fatigue crack propagation characteristics of Ti-6Al-7Nb with various microstructures will be also reported, comparing with that of Ti-6Al-4V ELI.

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EFFECT OF SURFACE FINISHING ON FATIGUE PERFORMANCE OF THE SURGICAL IMPLANT ALLOY Ti-6Al-7Nb: *J. Kiese*¹; *U. Holzwarth*¹; *L. Wagner*¹; ¹Technical University of Brandenburg at Cottbus, Department of Materials Technology, Postfach 101344, Cottbus 03013 Germany

Latest technologies in designing and testing of new implants require higher strength in addition to improved fatigue performance in comparison to the properties of the as-delivered condition. The decision to use the reliable alloy Ti-6Al-7Nb was made as a result of its high biocompatibility and its high potential of property improvement. Thermomechanical treatments were used to develop duplex microstructures in Ti-6Al-7Nb, containing 20 and 60% primary- α . By varying the cooling rate from the duplex anneal, two strength levels of the lamellar matrix were achieved. In addition, mechanical surface treatments such as shot peening and roller-burnishing were performed to improve the fatigue resistance. Hour-glass shaped specimens were shot peened and roller-burnished by a wide range of Almen intensity and working pressure, respectively, to evaluate optimum conditions for fatigue performance. A part of specimens was additionally grit blasted with Al₂O₃ to increase the surface roughness and prevent iron contamination of the titanium surface that may be caused by embedded steel particles from the shot peening process. Fatigue behavior was studied in rotating beam loading at frequencies of 60 Hz in air and corrosive Ringer's solution and compared to the electrolyti-

cally polished reference. A combination of suitable microstructure and optimum surface treatment led to an increase in fatigue strength of Ti-6Al-7Nb of up to 60%. The results will be explained in terms of the influence of surface roughness, induced dislocation density and residual stresses on crack nucleation and microcrack growth.

HOT DEFORMATION OF ALUMINUM ALLOYS: Process Simulation and Development - II

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

Wednesday PM Room: Orchard
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Terrence Langdon, University of Southern California, Departments of Material Science & Mechanical Engineering, Los Angeles, CA 90089-1453 USA; M. E. Kassner, Oregon State University, Dept. of Mechanical Engineering, Corvallis, OR 97331 USA

2:00 PM INVITED PAPER

SIMULATION OF COMMERCIAL HOT ROLLING BY LABORATORY PLANE STRAIN COMPRESSION AND ITS APPLICATION TO ALUMINUM INDUSTRY CHALLENGES: *G. J. Marshall*¹; ¹Alcan International, Banbury Laboratory, Southam Road, Banbury OX167SP UK

The majority of semi-fabricated, flat rolled products are manufactured by the aluminium industry using large and expensive hot and cold rolling facilities to meet the high standards required for subsequent downstream processing and in-service performance. For many the hot rolling stage is crucial in determining the correct microstructure, including crystallographic texture, prior to cold rolling and, in some cases, final annealing. On-line monitoring for the control of metallurgical parameters is in its infancy within the industry and strict practices for process control are enforced to achieve the correct metallurgy. These practices are traditionally established from experience and plant trials. In recent years the determination of process-microstructure-property relationships has relied on extensive use of laboratory plane strain compression (PSC) testing and the introduction of this information into physically based models. In 1991 Alcan's Banbury Laboratory designed and commissioned a PSC rig to simulate the deformation history of commercial hot multi-stand rolling (tandem mill rolling), typical of the major Aluminium rolling companies. This paper will review the capability of the Alcan PSC tester with reference to current industrial challenges in the production of packaging, architectural and transportation products. Examples will be provided to demonstrate particular issues, progress towards solutions and future objectives.

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RESTORATION MECHANISMS AND HOT ROLLING OF AL ALLOYS: *H. J. McQueen*¹; ¹Concordia University, Mechanical Engineering, Montreal H3G 1M8 Canada

The industrial rolling of Al alloys is generally conducted in multi-stage schedules of 10-15 passes, partly on reversing and partly on continuous mills, with temperature declining from 500 to between

300 and 250°C (which might be called warm rolling). Such schedules have been physically simulated on a torsion machine with measurement of flow stresses and interpass softening microstructures were examined at many stages. During the passes of 10-40% reduction, recrystallized material undergoes monotonic hardening possibly to steady state, with development of a dynamically recovered substructure in elongated grains. During most intervals, only static recovery takes place so that in the following passes, the flow curves exhibit higher initial stresses but less strain hardening than described above. Static recrystallization may take place to some degree in long intervals after passes with higher temperature and strain so that the behavior in the following pass is some combination of those described above according to the role of mixtures. The constitutive equations determined for recrystallized aluminum and particle alloys are not applicable to calculation of the mean stresses in the passes of such schedules because of the recovered substructure carried over. However, those determined for Al-Mg alloys appear to be applicable because the solute drag effect has a more significant role in defining initial flow stress than the substructure.

3:00 PM

COMPARISON OF EXTRUSION MODELING OF AA7075 WITH OTHER ALLOYS AND COMPOSITES: *E. V. Konopleva*¹; *M. Sauerborn*¹; *H. J. McQueen*¹; ¹Concordia University, Mechanical Engineering, Montreal H3G 1M8 Canada

The extrusion of AA7075 alloy was modeled by DEFORM™ for comparison with AA6061 and AA2618 alloys and particulate composites of those three alloys that had been prepared by liquid metal mixing. The modeling was based on constitutive data established over ranges 250-540°C and 0.1 to 4.0 s⁻¹ by torsion testing. In these tests hot ductility of 7075 declined above 400°C become much lower than 2618 which has low incipient melting (503°C) and 6061 which has very high ductility. The extrusion was modeled for a range of billet temperatures 350-500°C, ram speeds 2.6 and 5 mm/s and extrusion ration of 31. Distribution of velocity, strain rate, temperature and mean stress were determined to clarify the process details. The maximum loads and temperatures are compared to understand the relative extrudability.

3:30 PM INVITED PAPER

MICROSTRUCTURAL DEVELOPMENT FOR SUPERPLASTICITY IN Al-Mg-Mn ALLOYS: *J. S. Vetrano*¹; *M. T. Smith*¹; *C. H. Henager*¹; *S. M. Bruemmer*¹; ¹Pacific Northwest National Laboratory, P.O. Box 999, Richland, WA 99352 USA

Thermomechanical processing and alloying additions have been utilized in Al-Mg-Mn alloys to create microstructures suitable for superplastic deformation. Superplasticity typically requires a grain size of <10 μm that is stable against growth at temperatures close to the melting point of the material. We have examined the role of Mn, Sc and Zr additions on recrystallization and grain growth during thermomechanical processing and superplastic deformation of cast Al-Mg alloys. Particles containing Mn which formed during solidification were observed to be efficient nuclei for recrystallization but their effectiveness was reduced when Zr was present. This was a result of a shape change in these particles which did not allow them to break up and disperse during hot and cold rolling. Smaller Al₆Mn dispersoids, Al₃Zr and Al₃Sc particles acted to pin grain boundary migration. Their effectiveness was maximized by tailored heat treatments to create an optimum dispersion and particle size. Grain sizes less than 5 μm could be achieved by conventional cold-rolling and recrystallization treatments, resulting in superplastic elongations of more than 700%. *Work supported by the Materials Division, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC06-76RLO 1830

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FLOW AND FRACTURE BEHAVIOR DURING WARM FORMING OF ALUMINUM ALLOYS: *A. K. Ghosh*¹; *Z. Guo*¹; ¹The University of Michigan, Ann Arbor, MI 48109-2136 USA

Constitutive Behavior — temperature and strain rate effects, microstructural and textural changes, cavitation and plastic growth of voids.

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REFINEMENT OF CONE TESTING METHODOLOGY FOR CAVITATION STUDIES OF SUPERPLASTIC ALUMINUM ALLOYS: *V. I. Levit*¹; R. Crooks¹; R. N. Shenoy¹; Yu N. Gornostyrev²; ¹Analytical Services and Materials, Inc., 107 Research Drive, Hampton, VA 23666 USA; ²Institute of Metal Physics RAS, 18 Kovalevskaya St., Yekaterinburg 620219 Russia

Cavitation occurs during superplastic forming (SPF) of aluminum alloys primarily as a result of decohesion at grain or interphase boundaries. Cone testing allowed the evaluation of temperature, pressure differential and back pressure effects on: maximum strain at fracture, strain distribution inside the cone, total porosity level and porosity size and morphology. A few geometrical parameters were used to both characterize and simulate strain distribution in the cone test. The cavitation behavior in a 2xxx series and selected other superplastic aluminum alloys was characterized in the cone test samples using quantitative metallography and density measurements. Images showing pore morphologies and distributions were collected by scanning electron microscopy (SEM) of metallographically sectioned cones. A commercial image analysis package was then used to determine the dimensions, volume fraction and various distribution statistics of the cavities. The total porosity measured by SEM was in good agreement with the density measurements. Characteristic morphologies were attributable to forming parameters, especially temperature and strain rate. A useful correlation was found for representations of the distribution of pore volume fraction based on total volume within given pore-size ranges. Using this approach it is possible to distinguish between "ordinary" cavitation, which accompanies superplastic deformation processes, and the stage of void coalescence which precedes fracture.* Work performed at NASA Langley Research Center under contract NAS1-96014.

MATERIALS CHARACTERIZATION VIA THE INTERNET

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

Program Organizers: L. Allard, Oak Ridge National Laboratory; K. Alexander, Oak Ridge National Laboratory; U. Kahmen, Lawrence Berkeley National Laboratory

Wednesday PM Room: Dublin
October 15, 1998 Location: O'Hare Hilton Hotel

Session Chair: L. Allard, Oak Ridge National Laboratory, Oak Ridge, TN

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MATERIALS CHARACTERIZATION VIA THE INTERNET: The combination of high speed communication via the Internet and the increasing capabilities of desktop computers has made it possible to extend the control of expensive scientific instruments such as electron microscopes from the laboratory to distant locations. It is now possible for scientists at widely disparate locations to participate in a "TelePresence Microscopy" session, using inexpensive teleconferencing tools and their own personal computers. This includes the ability to assume full control of the microscope operation, if desired, so that a local operator need not even be present. Collaborators doing materials research can view results and share ideas in real time, improving the potential to obtain reliable interpretation and understanding of the

data. The implications of this new way for scientists to work together, unencumbered by time and space, to do better science will be explored via live demonstrations of TelePresence Microscopy with several national laboratory microscopy facilities.

MECHANISMS AND MECHANICS OF COMPOSITE FRACTURE: Particle 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, UES, Inc., Dayton, OH 45432-1894 USA; Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

Wednesday PM Room: Paris A
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Benji Maruyama, Air Force Research Laboratories, Materials and Manufacturing Directorate, Wright Patterson Air Force Base, OH 45433 USA

2:00 PM INVITED PAPER

FACTORS AFFECTING THE FRACTURE TOUGHNESS OF COMPOSITES: *John J. Lewandowski*¹; ¹Case Western Reserve University, Dept. Materials Science and Engineering, 10900 Euclid Ave., Cleveland, OH 44106 USA

The fracture toughness of discontinuously reinforced aluminum (DRA) is affected by a number of factors controlled by the reinforcement volume fraction, size, and distribution, while equally important microstructural effects on the evolution of damage and fracture toughness have been recorded. The presentation will provide a review of ongoing work in the field which has been conducted on such systems where systematic changes to the variables outlined above have been conducted. The talk will conclude with a brief review of opportunities for toughness enhancement in such systems and the variables which have been identified that contribute to the magnitude of toughening possible.

2:25 PM INVITED PAPER

EFFECTS OF INTERFACIAL PFZ LAYERS AND PRECIPITATES ON STRENGTH OF AGE-HARDENABLE ALUMINUM MATRIX MMCS: *Toshiro Kobayashi*¹; Hiroyuki Toda¹; ¹Toyohashi University of Technology, Department of Production Systems Engineering, Toyohashi, Aichi 441-8580 Japan

The existence of the thin layers of precipitate free zone have been observed around reinforcements in several MMCs having age-hardenable aluminum alloys as matrices, together with the formation of coarse interfacial precipitates and local solute atom segregation. The effects of such inhomogeneity of the matrix microstructure are analyzed by means of the elastic-plastic finite element analysis. The plastic flow of a matrix is concentrated within the PFZ layers due to its ductile nature, thereby reducing the overall strength of MMCs. On the other hand, the interfacial precipitates effectively retard the concentrated plastic flow within the PFZ layers. The interfacial precipitates never exhibit such preferable behavior when the reinforcements are surrounded by the homogeneous matrix. Both the axial stresses within the reinforcements and the cavity formation rate at the interfaces increase with increasing the thickness of the PFZ layers and the size of the interfacial precipitates. The cavity formation rate is re-

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markedly affected by the morphology and density of the interfacial precipitates.

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FAILURE MECHANISMS AND MODELING OF FRACTURE OF

DRA: *B. S. Majumdar*¹; D. B. Miracle²; ¹AFRL, Materials Directorate, Wright Patterson Air Force Base, OH 45433 USA; ²UES, Inc., Dayton, OH 45432 USA

In this study, the mechanisms of failure of discontinuously reinforced aluminum (DRA) alloy composites were investigated using a 15 volume percent 7093-Al-alloy/SiCp composite. Different heat treatments were employed to change the flow stress of the matrix, to allow probing of the damage mechanisms and the elongation and fracture toughness of the DRA. Except for the highly overaged condition, the sequence of damage consisted of particle fracture, followed by damage and failure of the matrix ligaments. Elastic-plastic analysis and probability statistics were used to extract the Weibull strength characteristics of the particles. The matrix ligament failure was then modeled using the damage model of Cockcroft and Latham, to derive the failure strain of the composites. The results show reasonable agreement with experimental data, and suggest that at high volume fraction it is critical to retain high particle strength. The fracture toughness of the materials were also analyzed, and issues related to testing and analyses, as well as the inverse dependence of toughness on strength will be discussed.

3:15 PM INVITED PAPER

THE CREEP STRENGTHENING OF SiC PARTICULATES IN

POWDER METALLURGY SiC-AL COMPOSITES: *Farghalli A. Mohamed*¹; ¹University of California, Department of Chemical and Biochemical Engineering and Materials Science, Irvine, CA 92697-2575 USA

A systematic comparison between the creep behavior of discontinuous SiC-Al composites and that of their unreinforced matrix alloys under similar experimental conditions is of practical and scientific significance. From a practical point of view, such a comparison is essential to the process of assessing the potential of these composites for use as structural materials for high temperature applications. From a scientific point of view, information obtained from such a comparison is critical to the identification of various processes that may limit the creep strengthening imparted by SiC particulates or whiskers. In this paper, a comparison between the steady-state creep rates of SiC-2124 Al composites and those of the unreinforced matrix alloy, 2124 Al, over eight orders of magnitude of strain rate reveals that, for constant temperature, strengthening arising from SiC particulates is eliminated at high strain rates. An examination of the microstructure of deformed samples suggests that the loss of strengthening at high strain rates is due to the occurrence of debonding between SiC particulates and 2124 Al (interfacial debonding). This suggestion is supported by the observation that no significant interfacial debonding is detected in a SiC-6061 Al composite which exhibits better creep resistance than 6061 Al over the entire strain rate range measured (no loss of the creep strengthening at high strain rates).

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IMPROVEMENT OF FRACTURE BEHAVIOUR OF SiCP REIN-

FORCED 6XXX AL-MATRIX COMPOSITES BY TAILORING MATRIX MICROSTRUCTURE: J. K. Chakravarty¹; R. Nagarajan¹; I. Dutta¹; T. R. McNelley¹; ¹Naval Postgraduate School, Center for Materials Science and Engineering, Department of Mechanical Engineering, Monterey, CA 93943 USA

The influence of matrix microstructure on the fracture behaviour of 6092 Al-17.5 vol. % SiC particulate composites has been studied. Both crack initiation and propagation phenomena have been investigated to predict overall fracture behaviour of this composite. It has been successfully demonstrated that with a combination of thermo-mechanical processing and heat treatment, it is possible to modify crack nucleation behaviour and to obtain a desirable strength-fracture toughness combinations. Novel heat treatment methods have

been employed to obtain a ductile layer at the SiC-matrix interface by the introduction of precipitate free zones (PFZs), with the objective of inhibiting propagation of cracks from fractured SiC particles to the surrounding matrix. The impact of this ductile layer on the fracture behaviour of the composites will be discussed. In order to assess the damage tolerance of various microstructural states, evolution of young's modulus, poisson's ration and dilatational strain as a function of strain has been studied. An attempt will be made to rationalize the observed effect of microstructure on fracture behaviour and damage tolerance. * Supported by the US Army Research Office and the Wright Materials Laboratory of the US Air Force

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FATIGUE BEHAVIOR OF PARTICLE REINFORCED METAL MATRIX COMPOSITES:

*Nikhilesh Chawla*¹; J. Wayne Jones¹; John E. Allison²; ¹University of Michigan, Department of Materials Science and Engineering, 2300 Hayward Dr., Ann Arbor, MI 48109-2136 USA; ²Ford Motor Co., Ford Research Laboratory, Dearborn, MI 48124 USA

SiC particle reinforced Al matrix composites, for example, are being targeted for use as automotive components, where high cycle fatigue resistance (at least 10⁷ cycles) and low cost are the primary considerations. The cyclic fatigue behavior of SiC particle reinforced Al 2080 matrix composites was investigated. The fatigue response of these materials can be described in terms of combinations of classical composite strengthening (direct strengthening) and changes in matrix microstructure and deformation characteristics (indirect effects) which arise from the presence of the reinforcement. By keeping the matrix microstructure constant, a systematic investigation of the effect of reinforcement size and volume fraction on fatigue life was obtained. Matrix microstructure was also varied to examine the effect of precipitate size and distribution on the fatigue behavior of the composites with a given amount of reinforcement. The extent of damage during fatigue in the composite and the unreinforced alloy was characterized by the evolution of stress-strain hysteresis loop shape, plastic strain amplitude, and modulus reduction. Finally, fatigue crack initiation and propagation processes were evaluated and related to matrix microstructure and the presence of reinforcement.

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THE QUASI-STATIC AND CYCLIC FATIGUE FRACTURE BE-

HAVIOR OF A DISCONTINUOUSLY-REINFORCED TITANIUM

ALLOY METAL MATRIX COMPOSITE: *T. S. Srivatsan*¹; W. O.

Soboyejo²; R. J. Lederich³; ¹The University of Akron, Department of Mechanical Engineering, Akron, OH 44325 USA; ²The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; ³Boeing Aerospace Company, St. Louis, OH USA

A study was undertaken with the primary objective of rationalizing the underlying mechanisms and intrinsic micromechanisms governing the quasi-static fracture, cyclic deformation and cyclic strain-controlled and cyclic stress controlled fatigue fracture of a discontinuously reinforced titanium alloy metal-matrix composite. The discontinuous titanium boride whisker-reinforced Ti-6Al-4V alloy was cyclically deformed under fully-reversed, tension-compression loading over a range of strain amplitudes giving cyclic fatigue lives of less than 104 cycles. The whisker-reinforced titanium alloy matrix was also cyclically deformed under total stress amplitude-control, over a range of stress amplitudes giving fatigue lives in the high-cycle range (> 104 cycles), at both ambient and elevated temperatures. The fundamental kinetics governing the quasi-static and final fatigue fracture processes will be discussed and rationalized in light of the mutually interactive influences of composite microstructural effects, matrix deformation characteristics, cyclic strain and stress amplitudes, composite response stress, and test temperature.

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MECHANICS AND MECHANISMS OF FRACTURE IN TOUGHENED NiAl COMPOSITES: M. Li¹; R. Wang²; D. Zheng²; W. O. Soboyejo¹; ¹The Ohio State University, Department of Materials of Science and Engineering, 2041 College Road, Columbus, OH 43210 USA; ²The Ohio State University, Department of Aerospace Engineering, Applied Mechanics and Aviation, 155 West Avenue, Applied Mechanics Section, Columbus, OH 43210 USA

The mechanics and micromechanisms of fracture in toughened NiAl composites are elucidated in this paper. Following an initial discussion on transformation toughening of NiAl, the effects of layer thickness on the fracture behavior of NiAl composites will be examined within a combined mechanics and materials framework. The synergistic interactions between shielding contributions from crack bridging and transformation toughening are also elucidated. The implications of the results are discussed for the design of toughened NiAl composites.

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STRAIN ACCUMULATION DURING SLIDING WEAR OF SiC WHISKER REINFORCED ALUMINUM COMPOSITES: A. P. Sannino¹; H. J. Rack²; ¹Sengate Technology, Bloomington, MN 55435 USA; ²Clemson University, Clemson, SC 29634-0907 USA

Extensive tribological investigations of discontinuously reinforced metal matrix composites have generally shown that these materials provide significantly improved wear resistance vis a vis their unreinforced matrices. However when delamination is the controlling wear mechanism composite wear performance is similar to and may actually be inferior to the corresponding unreinforced alloy. This presentation will discuss the development and implementation of a coupled finite element - computerized image analysis model for predicting the precursor subsurface strains developed prior to delamination during surface deformation. Comparison between these computational results and experimental examination of global damage accumulation in a SiC whisker reinforced 2124 Al/17-4 PH tribo-couple will also be presented.

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CREEP CRACK GROWTH BEHAVIOR OF BRIDGED CRACKS IN COMPOSITES: S. B. Biner¹; ¹Iowa State University, Ames Laboratory, Ames, IA 50011 USA

In this study, the creep crack growth behavior of bridged cracks that occurs in the composites is investigated numerically. In the analysis, crack growths resulting from the growth of pre-nucleated creep cavities with diffusional and dislocation assisted mechanisms are fully accounted for. The bridging elements are assumed to be elastic. The results indicate that the bridging tractions significantly relax even with the creep deformation of the composite alone. A much weaker stress singularity than the ones described by K or C* occurs ahead of the bridged cracks in the creep regime. In this weak singularity region the cavities grow at comparably high rates to each other.

MODELLING OF TEXTURE AND ANISOTROPIC PROPERTIES IN STEEL AND ALUMINUM INDUSTRIES FOR OPTIMUM MATERIALS PERFORMANCE: Session II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Texture & Anisotropy Committee

Program Organizers: Hasso Weiland, Alcoa, Alcoa, PA 15069 USA; J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

Wednesday PM

Room: Madrid

October 14, 1998

Location: O'Hare Hilton Hotel

Session Chair: J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

2:00 PM INVITED PAPER

NEW PARAMETER MODEL FOR TEXTURE DESCRIPTION IN STEEL SHEETS: L. Delanny¹; P. Van Houtte¹; D. Vanderschueren¹; ¹Katholieke Universteit Leuven, Department MTM, de Croylaan 2, Heverlee B-3001 Belgium

A new model is proposed for the characterisation of steel sheet textures. This model relies on the identification of 25 relevant parameters in the Orientation Distribution Function (ODF). Textures consisting of alpha and gamma fibres and/or cube and Goss orientations can be generated. It was found that the model can quantitatively reproduce almost any industrial steel sheet texture. A first application which was conducted consisted in the study of the r-value sensitivity on the model parameters.

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THE DESIGN OF A BIAXIAL TENSILE TEST AND ITS USE FOR THE VALIDATION OF CRYSTOGRAPHIC YIELD LOCI: E. Hoferlin¹; A. Van Bael¹; P. Van Houtte¹; G. Steyaert²; C. De Mare²; ¹K.U. Leuven, Department MTM, de Croyoann 2, B-3001, Herverlee Belgium; ²OCAS nv, John Kennedylann 3, Zelzate B-9060 Belgium

A biaxial tensile test has been designed for the experimental determination of yield locus points of thin steel sheets. Using texture-based anisotropic finite element (FE) simulations, the geometry of the test sample has been optimised and the accuracy of the conversion procedure has been validated. Experimental tests have been carried out on low carbon steels and both yield stresses and ratios of plastic strains have been compared to the theoretical predictions obtained with the Taylor-Bishop-Hill (TBH) model using the experimentally determined crystallographic textures.

3:00 PM INVITED PAPER

THE INFLUENCES OF DEFORMATION BANDING AND SLIP SYSTEMS ON MODELING BCC ROLLING TEXTURES: Guanglei Liu¹; B. J. Duggan¹; Lixin Zhang²; ¹The University of Hong Kong, Department of Mechanical Engineering, Pokfulam Road, Hong Kong; ²Institute of Corrosion and Protection of Metals, Academia Sinica, 62 WenCui Road, Shenyang 110015 P. R. China

Deformation banding has been established in cold rolled iron by microstructure investigation with a series of SEM micrographs taken from progressively rolled pure iron. Such deformation modes should be included in texture modeling. Of particular significance to simulating bcc rolling texture is the occurrence of deformation banding in the rolling plane since this allows another degree of freedom, ϵ_{32} , in modeling texture evolution with DB model. Simulations including deformation banding and $\{111\} \langle 110 \rangle$ slip show a better agreement with experimental observations. On the other hand, $\{112\}$ slip yields peak type texture and $\{123\}$ slip supports the a-fiber.

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SIMULATION AND EXPERIMENTAL INVESTIGATION OF ROLLING TEXTURES OF FERRITIC STEELS: HOMOGENIZATION METHODS VERSUS Fe METHODS: *D. Raabe*¹; ¹CMU, Pittsburgh, PA USA

The paper focusses on two large-scale approaches to simulate the evolution of rolling textures of polycrystalline metals. The first part of the paper presents predictions obtained by various homogenization approaches, such as Taylor-Bishop-Hill-type and Self-Consistent Modeling. The simulations are conducted for simple plane-strain deformation modes. The texture predictions are conducted for a ferritic stainless steel. The simulations are compared with corresponding experimental data. The second part of the paper presents predictions obtained by the integrated use of viscoplastic finite element simulations in conjunction with Taylor-Bishop-Hill-theory. The simulation results are compared with experimental results for a commercial low-carbon steel.

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RESIDUAL STRESSES IN HY100 POLYCRYSTALS: COMPARISONS OF EXPERIMENTS AND SIMULATIONS: *Paul Dawson*¹; Donald Boyce¹; Ronald Rogge²; Stuart MacEwen³; ¹Cornell University, Sibley School of Mechanics and Aerospace Engineering, 196 Rhodes Hall, Ithaca, NY 14853 USA; ²Steacie Institute for Molecular Science, National Research Council, Ottawa, Ontario K1A 0R9 Canada; ³Alcan International Limited, Kingston Research and Development Center, P.O. Box 8400, Kingston K7L4L9 ONT Canada

Residual stresses arise in polycrystals when they are unloaded after being inelastically deformed. Inelastic deformations are inhomogeneous over the scale of polycrystals due to the anisotropy of individual crystals and to differences in lattice orientations from crystal to crystal. Some crystallographic orientations tend to exhibit greater residual stresses than do other orientations, again a consequence of the single crystal anisotropy both elastic and plastic. Residual stress levels in polycrystals can be determined from neutron diffraction measurements of elastic lattice strains. In this paper we discuss tensile testing of HY100 in which diffraction measurements were made after various levels of plastic strain, both while the specimens were loaded and while they were unloaded. Finite element simulations of the experiments are also presented which are based on an elastoplastic polycrystal constitutive theory. Identical sequences of loading and unloading of specimens have been simulated, allowing the residual elastic lattice strains to be computed at several stages in the process of applying a large overall plastic strain. Comparisons of the residual lattice strains are presented for crystals having particular planes aligned either with the specimen tensile axis or with a transverse axis. The levels of the corresponding residual stresses are discussed relative to aspects of the crystal orientations.

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COMPUTER MODELLING OF GOSS TEXTURE DEVELOPMENT IN Fe-Si STEELS: *N. Rajmohan*¹; J. A. Szpunar¹; Y. Hayakawa²; ¹McGill University, Department of Mining and Metallurgical Engineering, Montreal, Quebec H3A 2B2 Canada; ²Kawasaki Steel Corporation, Technical Research Laboratories, 1-Kawasakidouri, Mizushima, Kurashika, Okayama 712 Japan

Abnormal growth of Goss grain is studied in detail by various computer models which use computer specimens representing the orientation distribution function (ODF) of primary recrystallized Fe-3%Si steel. The computer models take into account the anisotropy of GB energy and mobility. Using various computer experiments conducted, the importance of initial matrix texture, percentage of mobile boundaries around a growing grain, the mobility differences between GBs and the time of release of various grain boundaries during annealing is discussed. Selection mechanism that operates among various Goss nuclei (which are deviated from the real Goss orientation by few degrees) in the initial primary recrystallized matrix, is discussed in detail using the results obtained.

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APPLICATION OF A STOCHASTIC 3D CELLULAR AUTOMATON FOR THE SIMULATION OF RECRYSTALLIZATION TEXTURES IN BCC STEELS: *Dierk Raabe*¹; ¹CMU, Pittsburgh, PA USA

The paper introduces a 3D stochastic cellular automaton for the spatial, kinetic, and crystallographic simulation of mesoscale transformation phenomena that involve non-conserved structural field variables and the motion of sharp interfaces, such as encountered in recrystallization and grain growth. The local transformation rule that acts on each lattice site consists of the probabilistic analogue of a classical linear symmetric rate equation for interface motion. All possible switches are simultaneously considered using a weighted stochastic sampling integration scheme. The automaton is discrete in time, real space, and orientation space. The required input parameter to such simulations (local texture and a local stored energy measure as a function of x,y,z in conjunction with mobility data for the grain boundaries) can be provided by FE simulations or by coupled local texture and microstructure experiments. The method is very simple to use and allows the user to predict texture and microstructure (and thus anisotropy) under consideration of local effects.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VI: Metal-Matrix Composites - II

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

Wednesday PM

Room: Chicago

October 14, 1998

Location: O'Hare Hilton Hotel

Session Chairs: Dr. R. A. Drew, McGill University, Department of Mining and Metallurgical Engineering, Montreal, Quebec H3A 2B2 Canada; Dr. Z. Xiao Guo, Queen Mary and Westfield College, London University, London United Kingdom

2:00 PM INVITED PAPER

COST-EFFECTIVE MANUFACTURE OF TITANIUM/SILICON CARBIDE (SiC) FIBRE COMPOSITES: *Z. Xiao Guo*¹; ¹Queen Mary and Westfield College, London University, Mile End Road, London United Kingdom

Composites involving continuous SiC fibres and titanium matrices can offer a very desirable combination of weight-specific properties and are ideal for many applications in gas and steam turbine engines. However, large-volume applications are currently hampered by the relatively high costs of the materials. This paper critically examines the characteristics of the manufacturing routes for such composites, including well-studied processes, such as Foil-Fibre-Foil (FFF), Physical Vapour Deposition (PVD), and Vacuum Plasma Spraying (VPS), and new Slurry Powder Metallurgy (SPM) processes currently being developed. For a given manufacturing route, composite properties are enhanced if the material possesses uniform fibre distribution and a low oxygen content and is free from: fibre/matrix interface reactions, residual voids and fibre damage. The capabilities of the above processes in satisfying these requirements are compared and discussed. Possibilities of reducing product costs are analyzed. Several techniques of improving the cost-effectiveness of the composites in relation to manufacture are outlined. Particular emphasis is placed on the study of the SPM routes for Ti/SiC MMCs.

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PROCESSING OF METAL-MATRIX COMPOSITES BY WETTING-ASSISTED INFILTRATION USING METAL-COATED REINFORCEMENTS: Carlos A. Leon¹; Robin A. L. Drew¹; ¹McGill University, Department of Mining and Metallurgical Engineering, 3610 University Street, Montreal, Quebec H3A 2B2 Canada

Metal matrix composites produced via infiltration of a porous preform has been investigated as a viable fabrication process. The combination of properties of SiC/Al and Al₂O₃/Al composites makes of them potentially important as structural materials. Electroless nickel and copper plating techniques were used to enhance the wetting of the solid by the liquid phase in order to promote infiltration. Initially the work focused on establishing the best processing route to achieve effective metal-deposition on the ceramic powders. The quantity and size of deposited material, and the uniformity and nature of the coating upon deposition of Cu and Ni on Al₂O₃ and SiC particles were studied. Microscopy and chemical analysis revealed a continuous and uniform nickel deposition on reinforcements with traces of phosphorus resulting from the electroless solution. Surface morphology of copper-plated substrates consisted of fine adhering particles forming thinner metal coatings. Furthermore, the microstructure, coating distribution and infiltration behaviour of the composites are also being investigated.

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PHASE TRANSITIONS IN THE FORMATION OF Ti₅Si₃/TiAl IN SITU COMPOSITES AND THEIR MULTIPLEX STRUCTURES: Yijun Du¹; K. P. Rao¹; X. D. Han²; K. C. Law²; J. C. Y. Chung²; ¹City University of Hong Kong, Department of Manufacturing Engineering, Tat Chee Avenue, Kowloon, Hong Kong; ²City University of Hong Kong, Department of Applied Physics and Materials Science, Tat Chee Avenue, Kowloon, Hong Kong

A mixture of elemental Ti, Al and Si powders with a stoichiometry of Ti₁₄Al₂₁Si has been ball-milled for producing metastable phases. Differential thermal analysis (DTA) and x-ray diffraction (XRD) have been used to identify the phase reactions and transitions occurring in the metastable phases during heating. It has been found that Ti₅Si₃ is in situ formed through a phase transition chain, i.e., from TiSi₂ to Ti₅Si₄ and finally to Ti₅Si₃. Also, it has been found that formation of Ti₃Al (or TiAl, or TiAl₃, depending on the milling time) competes against TiSi₂ in the metastable phases during heating. In practice, the metastable phases were incorporated into Ti-Al powder matrix, and a final Ti₅Si₃/TiAl composite was obtained when the constituent powders underwent a reactive sintering (RS) process followed by hot isostatic pressing (HIP). Transmission electron microscope (TEM) was employed for revealing the phase structure and distribution of various phases in the composite. A hierarchical structure including ductility-favored phases such as alpha-2 (Ti₃Al) has been observed between the Ti₅Si₃ reinforcement and TiAl matrix. It is thus possible that both room-temperature ductility and high-temperature strength can be enhanced under such a multiplex microstructure. In addition, elemental Si powders of two different sizes (71 μm and 50 nm) were tried to study the respective structure evolutions of the resultant composites.

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THE INFLUENCE OF FABRICATION MODE ON THE PROPERTIES OF HEIGHT DEFORMED Cu-Nb/Ti COMPOSITE WIRES: L. Brandao¹; Peter N. Kalu¹; ¹FAMU-FSU College of Engineering, National High Magnetic Field Laboratory, Department of Mechanical Engineering, Tallahassee, FL 32310 USA

The principal requirements for conductor wires used in high-field pulsed magnets are high mechanical strength and high electrical conductivity. In order not to decrease the conductivity, a combination of fiber strengthening plus work hardening was used to strengthen Cu-Nb/Ti material. Heavy deformation was used to produce the required level of work hardening. Three fabrication methods, swaging, drawing and swaging plus drawing were employed in producing 2.03 mm diameter Cu-Nb/Ti wires. Here, the effect of the mode of fabrication

on the properties and texture development in Cu-Nb/Ti composite is reported.

3:40 PM INVITED PAPER

DEVELOPMENT OF HIGH STRENGTH HIGH CONDUCTIVE COPPER-BASE IN SITU COMPOSITE: H. G. Suzuki¹; K. Mihara¹; K. Adachi¹; Y. Jia¹; T. Takeuchi¹; ¹National Research Institute for Metals, 1-2-1 Sengen, Tsukuba 305 Japan

Cu-15% Cr-C,-Zr in situ composites which have the strength level higher than 1100MPa and electrical conductivity exceeding 70%IACS, have been developed by optimizing the alloy compositions as well as thermomechanical process. Cold drawing is effective to refine lamellar spacing of second phase (Cr fiber) and aging treatment just after solutioning gives precipitation strengthening and recovery of electrical conductivity. The optimization of each process was conducted. The tensile strength follows Hall-Petch type equation with Cr lamellar spacing. The additions of C refines the solidification structure, while Zr increases the precipitation hardening. We will discuss the method for process optimization and the mechanism of strengthening of in situ composite.

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TRIBOLOGICAL PROPERTIES OF SELF-LUBRICATIVE Cu COMPOSITES WITH CLUSTER DIAMOND: Kotaro Hanada¹; ¹Mechanical Engineering Laboratory, Plasticity & Forming Division, Namikil-2, Tsukuba, Ibaraki Japan

Cluster diamond (CD) produced by a detonation method has attracted special interests in various fields because it has unique characterization such as (1) ultra-fine particulate (5nm in average), (2) single crystal, (3) almost a spherical shape, (4) excellent lubrication, (5) excellent abrasion. In this work, CD dispersed self-lubricative Cu composites are fabricated by powder metallurgy method, and the tribological properties are investigated. Pure Cu powder and CD powder are mechanically mixed at 500rpm for 40 hours under Ar atmosphere, and the mixed composite powders are hot pressed at 723°K in vacuum. X-ray analysis shows that CD is not changed to graphite in the compacted Cu/CD composite powders. The coefficient of friction and the linear wear of Cu/1vol%CD composite are superior to pure Cu under all testing condition in this work, and those improvements are 36% and 78% in maximum, respectively. However, as the volume fraction of CD is increased, the diffusion bonding between Cu matrix powders becomes weak because of the agglomeration of CD at the prior particle boundaries of Cu/CD powders, and it results in poor friction properties.

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SIMULATION OF METAL MATRIX COMPOSITE INFILTRATION PROCESSING: Th. Dopler¹; A. Modaressi¹; V. Michaud²; ¹Laboratoire de Mecanique des Sols, Structures et Materialux, CNRS URA 850, Ecole Centrale Paris, Chitenay-Malabry France; ²Laboratoire de Technologie des Composites et Polymers, Department des Materiaux, E-cole Polytechnique Federale de Lausanne Switzerland

Infiltration processing constitutes a simple method to produce metal matrix composites, whereby a liquid metal penetrates into a porous preform of the reinforcing phase. A 3-D finite element software is currently being developed to simulate this process, based on the similarity with drainage phenomena in soil mechanics. Capillarity is taken into account for isothermal infiltration under any increasing applied pressure function. Simulation of preform deformation is also possible. Saffil alumina preforms were infiltrated by molten aluminum. Comparison of simulation results with experimental data shows good agreement, in terms of infiltration kinetics and porosity distribution.

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EFFECT OF FLYASH PARTICLES ON THE WEAR BEHAVIOUR OF ZA-27 ALLOY COMPOSITES: S. C. Sharma¹; B. M. Girish¹; R. Kamath¹; B. M. Satish¹; ¹R. V. College of Engineering, Department

of Mechanical Engineering, Division of Research and Development, Bangalore, Karnataka 560059 India

In the present study, the effect of zircon reinforcement on the unlubricated sliding wear behaviour of ZA-27 alloy composites was investigated using a pin-on-disc sliding wear testing machine. The composite specimens were prepared using the liquid metallurgy technique and the percentage of zircon was varied from 1-5% in steps of 2% by weight. The sliding wear tests were conducted at loads of 3, 4, 5 and 6 Kg (29.4, 39.2, 49.1 and 58.9 N respectively) and rotational speeds of 200, 250 and 300 rpm (i.e., tangential speeds of 1.25, 1.56, 1.87 m/s respectively). The unreinforced alloy was also tested under identical conditions in order to enable comparison. The results of the tests revealed that the zircon reinforced composites exhibited a lower wear rate compared to the unreinforced alloy specimens. Increase in the applied load increased the wear severity by changing the wear mechanism from abrasion to particle cracking induced delamination wear. It was found that with the increase in zircon content, the wear resistance increased monotonically with hardness. SEM micrographs indicated the effect of zircon particles in reducing the wear rate of the composite material.

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SHORT GLASS FIBRES REINFORCED ZA-27 ALLOY COMPOSITES FOR JOURNAL BEARING APPLICATIONS: S. C. Sharma¹; B. M. Girish¹; R. Kamath¹; B. M. Satish¹; ¹R. V. College of Engineering, Division of Research and Development, Department of Mechanical Engineering, Bangalore, Karnataka 560059 India

The bearing performance of cast ZA-27 alloy composite materials reinforced with short glass fibres have been investigated under lubricated, semi-dry and dry conditions. The composite materials were fabricated employing the compocasting technique. The journal bearing in their as-cast conditions were tested using a computer interfaced bearing test rig. The test bearings press against the shaft (EN24) under a radial vertical loading. It followed from the results obtained that the composite bearings exhibited lower friction in comparison with the unreinforced alloy bearings. The composites as well as the alloy bearings tested under lubrication exhibited least coefficient of friction in comparison with those tested under semi-dry and dry tests. In the lubricated tests, the composite bearings were able to run without seizure up to the regimes of boundary lubrication with less friction. In the semi-dry and the dry tests, the composite bearings exhibited lower friction which further reduced with the increase in the fibres content but increased with the increase in load. Under the semi-dry and dry conditions, the unreinforced alloy bearings seized at much lower loads than the composite bearings.

PROCESSING & PROPERTIES OF ADVANCED STRUCTURAL CERAMICS: Advanced Structural Ceramics: Properties

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Rajiv S. Mishra, University of California, Dept of Chemical Engineering & Materials Science; Amiya K. Mukherjee, University of California, Dept of Chemical Engineering & Materials Science

Wednesday PM
October 14, 1998

Room: London
Location: O'Hare Hilton Hotel

Session Chair: Katherine T. Faber, Northwestern University, Materials Science and Engineering, Evanston, IL 60208 USA

2:00 PM KEYNOTE

A SYSTEMS APPROACH TO THE DESIGN OF STRUCTURAL CERAMICS: Rishi Raj¹; Ganesh Subbarayan¹; ¹University of Colorado, Department of Mechanical Engineering, Boulder, CO 80309-0427 USA

A systems approach in the analysis and design of structural ceramics is used to identify the critical parameters in the development of a ceramic for specific applications. In this instance we shall consider ceramic coatings on metallic turbine blades. The presently used zirconia based thermal barrier coatings will be compared with other candidate materials. A rational process for identifying the key issues in each instance will be articulated. Environmental issues and the mechanical chemical properties of the ceramic-metal interface merges as one of the critical areas from the point of view of failure prediction. However, the total performance of the coating also depends on the thermal diffusivity and on the surface roughness of the coating.

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THERMAL AND MECHANICAL PROPERTIES OF NANOSTRUCTURED ZIRCONIA THIN FILMS*: J. A. Eastman¹; G. Soyoz¹; R. J. DiMelfi²; L. J. Thompson¹; ¹Materials Science Division, Argonne National Laboratory, Argonne, IL 60439 USA; ²Reactor Engineering Division, Argonne National Laboratory, Argonne, IL 60439 USA

Stabilized zirconia thermal barrier coatings are used in a number of important applications, such as in protecting gas turbine components in high temperature environments. Improved thermal barrier coatings are required to attain desired higher, and thus more efficient, operating temperatures. Improvements in service lifetimes of components are also desirable and require the development of coatings with improved damage tolerances both at ambient and elevated temperatures. Nanocrystalline coatings are expected to exhibit reduced thermal conductivities compared to coarse-grained coatings because of a reduction in the mean free path of phonons due to presence of grain boundaries. This behavior, combined with expected improved mechanical properties, makes nanostructured zirconia coatings excellent candidates for future applications as thermal barriers. Experiments are underway to produce yttria-stabilized zirconia thin films by metal-organic chemical vapor deposition techniques and to correlate thermal and mechanical properties with film microstructure. *This work is supported by the U. S. Department of Energy, BES-DMS, under Contract W-31-109-Eng-38 and by a grant from Argonne's Coordinating Council for Science and Technology

3:05 PM INVITED PAPER

MECHANICAL PROPERTIES OF SOME OXIDE SINGLE CRYSTALS AT HIGH TEMPERATURES: T. E. Mitchell¹; ¹Los Alamos National Laboratory, Center for Materials Science, MS-K765, Los Alamos, NM 87545 USA

The mechanical properties of sapphire and spinel at high temperatures are described and compared with other oxides. The critical resolved shear stress is found to decrease logarithmically with increasing temperature for both basal and prism plane slip in sapphire and for {110} and {111} slip in spinel. The CRSS of magnesium aluminate spinel ($MgO \cdot nAl_2O_3$) decreases by almost orders of magnitude as n increases from 1 (stoichiometric spinel) to 3.5; at the same time the minimum temperature for compression ductility decreases from $\sim 1800^\circ C$ to $\sim 1300^\circ C$. Deviations from stoichiometry are compensated by the introduction of cation vacancies with a concentration given by $[V_c] = (n-1)/3(3n+1)$. The solution softening effect resulting from increasing values of n and $[V_c]$ is found to be such that the CRSS is proportional to $[V_c]^2$. The temperature dependence and the concentration dependence suggest that the CRSS is controlled by a Peierls stress which is decreased by kink nucleation at cation vacancies. In addition, hydrolytic weakening is observed in sapphire which also may be due to double kink nucleation at point defects, possibly hydrogen. These effects are discussed in terms of their crystal structures.

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THE EFFECT OF HIGH FREQUENCY FATIGUE ON THE INTERFACIAL PROPERTIES OF NICALON FIBER REINFORCED SICON CERAMIC MATRIX COMPOSITES: *Nikhilesh Chawla*¹; Jeffrey Eldridge²; ¹University of Michigan, Department of Materials Science and Engineering, 2300 Hayward Dr., Ann Arbor, MI 48109-2136 USA; ²NASA Lewis Research Center, Cleveland, OH 44135 USA

In a variety of applications, continuous fiber reinforced ceramic matrix composites (CFCMCs) are likely to encounter high frequency fatigue loading. Most CFCMCs have been tailored with relatively weak fiber/matrix interfaces that are conducive to crack branching, crack deflection, and fiber pullout, that substantially enhance the toughness of these materials. During high frequency fatigue, however, it has been shown that wear of the weak interface takes place, contributing to premature failure in these materials. In this work, we have studied a woven Nicalon fiber reinforced SiCON matrix composite, processed by polymer infiltration and pyrolysis (PIP), with a relatively strong fiber/matrix interface to increase the fatigue resistance of the composite. The composite was fatigued at a loading frequency of 100 Hz. The evolution of interfacial shear stresses was measured using fiber pushout technique and related to the macroscopic fatigue behavior of the composite. Finally, modulus reduction, stress-strain hysteresis, and temperature rise in the composites were measured with increasing cycles to provide an estimate of evolution of damage in the composite.

4:00 PM INVITED PAPER

TOUGHENING IN CERAMIC LAMINATES: *K. T. Faber*¹; D. M. Baskin¹; E. R. Fuller²; ¹Northwestern University, Department of Materials Science and Engineering, 2225 North Campus Drive, Evanston, IL 60208 USA; ²National Institute of Standards and Technology, Gaithersburg, MD 20899 USA

In both laminate and fiber-reinforced ceramics, the interface toughness relative to the toughness of the layer or fiber dominates the mechanical behavior and determines whether crack deflection or penetration occurs. We explore, here, laminates of textured and non-textured iron titanate, a highly anisotropic ceramic, in which various combinations of elastic and thermal mismatch can be produced using a single material. Layers as thin as 0.6 mm are made via gel-casting and sintered together to form laminates. Mechanical properties are evaluated using conventional fracture mechanics techniques. Laminate properties are compared to those simulated from microstructures using an object oriented finite element code (OOF). Design concepts for ceramic laminates are confirmed.

4:30 PM BREAK

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CERAMIC NANOCOMPOSITES: MECHANISMS FOR STRENGTH ENHANCEMENT: *B. Derby*¹; C. W. Lawrence¹; S. G. Roberts¹; H. Z. Wu¹; ¹University of Oxford, Department of Materials, Parks Road, Oxford OX1 3PH UK

Ceramic nanocomposites consisting of oxide matrices reinforced with submicron intragranular dispersions show an enhanced strength when compared to unreinforced matrix material of the same grain size. This increase in strength cannot be explained by an increase in toughness of these materials. From our understanding of the mechanisms of brittle fracture, this strength increase must be associated with a decrease in the maximum (Griffiths) flaw present in the test pieces. An analysis of surface flaw densities using Hertzian indentation, acoustic measurements and microstructural examination will be presented to demonstrate a lower surface defect density in these materials. This is also evident in a greater resistance to erosive wear shown by these materials. Possible mechanisms for this reduction in surface damage will be discussed.

5:05 PM

ELECTRIC FIELD ENHANCED PLASTIC DEFORMATION KINETICS OF FINE-GRAINED Al_2O_3 : James Campbell¹; *Hans Conrad*¹; Yusef Fahmy¹; ¹North Carolina State University, Materials Science and Engineering Dept., Raleigh, NC 27695-7907 USA

The influence of a contacting, axial electric field ($E=1-1.5$ kV/cm) on the plastic deformation kinetics in tension of fine-grained ($d_0=1.5$ mm) Al_2O_3 was determined at 1450°-1600°C by constant strain rate and stress relaxation tests. The electric current (6-16 mA) and temperature rise (10°-40°C) due to Joule heating were measured. It was found that application of the electric field during the course of a test reduced the flow stress by ~80%, of which approximately one-half was due to Joule heating and the remainder a direct effect of the field and/or current. The electric field had no detectable effect on the stress exponent $n = \ln(\text{strain rate}) / \ln \sigma$, which had a value of ~2 in all tests. The parameters and mechanism by which the electric field and/or current affected the plastic deformation kinetics are discussed.

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MECHANICAL PROPERTIES OF DENSE Al_2O_3 -ZrO₂ NANOCOMPOSITES: *S. Bhaduri*¹; D. F. Bahr²; ¹University of Idaho, Metallurgical and Mining Eng., Moscow, ID 83844-3024 USA; ²Washington State University, Pullman, WA 99164-2920 USA

Nanocrystalline Al_2O_3 -ZrO₂ powders were synthesized using a novel 'Auto Ignition' synthesis process. The respective nitrates and urea were used as precursors. The powders were hot isostatically pressed at 1200C for 2 hours at 175 MPa. Initial mechanical properties were determined by an indentation technique using a Vickers indenter. The results indicated that the hardness value was low and toughness was in the range for toughened ceramics. In order to check if phase transformation of ZrO₂ or ductility was involved, the samples were further tested using a nanoindenter in conjunction with an AFM. Load displacement curves did not indicate any phase transformation in these materials. Considerable pile up of materials was observed around the indentation. This is an evidence of ductility in these materials.

WEDNESDAY PM

Technical Program

THURSDAY AM

DAMAGE PROCESSES IN ADVANCED MATERIALS: Hydrogen and Corrosion Effects

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee, Corrosion and Environmental Effects Committee, Physical Metallurgy Committee

Program Organizers: Russ H. Jones, Pacific Northwest Natl. Lab., Richland, WA 99352 USA; Richard Ricker, NIST, Materials Rm B254, Gaithersburg, MD 20899; Norman S. Stoloff, Rensselaer Polytechnic Inst., MS&E Dept., Troy, NY 12190-3590 USA

Thursday AM Room: Paris C
October 15, 1998 Location: O'Hare Hilton Hotel

Session Chairs: R. H. Jones, Pacific Northwest National Laboratory, Richland, WA 99352 USA; N. S. Stoloff, Rensselaer Polytechnic Institute, Troy, NY 12180-3590 USA

8:30 AM INVITED PAPER

DETERMINATION OF HYDROGEN EFFECTS ON INTERFACIAL FRACTURE USING THIN FILM TESTS: *N. R. Moody*¹; *D. Medlin*¹; *S. Guthrie*¹; ¹Sandia National Laboratories, Livermore, CA 94550 USA

Although it is well-known that exposure to hydrogen and hydrogen producing environments can cause premature failure in many materials, experimental studies are inconclusive as to the mechanism controlling fracture. However, thin film testing provides a promising alternative for determining hydrogen effects on a local level. We have therefore employed nanoindentation and nanoscratch testing to determine how hydrogen affects deformation and fracture of thin tantalum nitride films and thin aluminum films on aluminum oxide substrates. Tantalum nitride films are rigid films that fracture along the film-substrate interface. The aluminum films are ductile and show a great resistance to interfacial fracture. Both films exhibit a strong susceptibility to interfacial fracture and are accompanied by a significant decrease in interfacial fracture energy when exposed to hydrogen or moisture. We will present these results and discuss the relationship that exists between hydrogen and interfacial fracture in these thin film systems.

9:10 AM

MODELING AND QUANTIFICATION OF CORRODED SURFACES: *W. M. Mullins*¹; ¹AFRL/MLLP-TMCI, Wright-Patterson, AFB, OH 45433 USA

The surface damage introduced by corrosion attack of surfaces is considered deleterious to long term structural integrity. As a result, the quantification of this damage represents an interest in the NDE community. Stochastic models for the chemical kinetics of general attack and intergranular/exfoliation corrosion are used to model the morphology of the surface as a function of time of exposure. Experimental results for corroded surfaces are presented which appear to agree with the model predictions. Stress analyses are performed for the surface morphologies in question to elucidate the relationship between fatigue-crack initiation and corrosion damage. Finally, discussions of the relationships between macroscopic measurements and predictions of residual service lifetime predictions are presented.

9:30 AM

THE EFFECT OF MICROSTRUCTURE AND COMPOSITION ON LOCAL CORROSION RESISTANCE OF NEWLY DEVELOPED NICKEL-FREE DUPLEX STAINLESS STEELS: *J. Wang*¹; *M. O. Speidel*¹; *P. J. Uggowitzer*¹; ¹Institute of Metallurgy, ETH, Zentrum, Zurich CH-8092 Switzerland

Duplex stainless steels (DSS) with high nitrogen contents have some advantages, i.e., higher strength and corrosion resistance, more stable austenitic content in high temperatures. Due to high contents of nitrogen which is mostly dissolved in the austenitic phase, the corrosive mechanism of DSS can be different from the low nitrogen containing DSS, which is based on the calculations of pitting resistance equivalents number (PRE) for each phase. This work studied the effect of microstructure and local chemical compositions of newly developed nickel-free DSS on pitting resistance. By applying different heat treatment from 950°C to 1200°C, the phase ratio and partitioning of alloying elements Cr, Mo and N could be changed. The research results showed that austenite has become the stronger phase in regard to pitting potential measurements in artificial seawater in such high nitrogen alloyed DSS and was in agreement with the PRE calculations. The corrosion resistance with nitrides and intermetallic phase precipitation is seriously degraded by local sensitization caused by the depletion Cr and/or Mo. The role of Mo is emphasized on the development of such high nitrogen DSS due to its enrichment in ferrite which increases the PRE of the weaker ferritic phase. The balance of PRE between two phases can be reached through the additions of the proper Mo content.

9:50 AM

EVALUATION OF THE INFLUENCE OF ALLOYING ELEMENTS ON THE PITTING RESISTANCE OF NITROGEN ALLOYED STAINLESS STEELS: *R. E. Ricker*¹; *J. L. Fink*¹; *R. D. Schmidt*¹; *F. Biancaniello*¹; *S. D. Ridder*¹; ¹National Institute of Standards and Technology, Metallurgy Division, Materials Science and Engineering Laboratory, Gaithersburg, MD 30899 USA

The development of improved techniques for the addition of nitrogen to stainless steels is leading to the development of new alloys with superior corrosion resistance. In many cases, these new alloys are so resistant to pitting corrosion that existing standard test methods cannot be used to guide alloy development. To overcome this problem, a new test method was developed and used to identify the influence of different alloying elements on the relative pitting resistance of a series of nitrogen containing alloys. This experimental technique will be presented along with an analysis of the influence of different alloying elements and a discussion of the impact of these results on our understanding of the mechanism of pitting corrosion in these alloys.

10:10 AM

ENVIRONMENTAL EFFECTS ON CREEP AND CRACKING OF ULTRA-HIGH PURITY Ni-16Cr-9Fe ALLOYS AT 360°C: *Denise J. Paraventi*¹; *Tom M. Angeliu*²; *William R. Caitlin*²; *Gary S. Was*³; ¹University of Michigan, Department of Materials Science and Engineering, 2940 Cooley Lab, 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA; ²General Electric Corporate Research and Development Center, Building K-1, Room 3A51, 1 River Road, Schenectady, NY 12309 USA; ³University of Michigan, Department of Nuclear Engineering and Radiological Sciences, Cooley Lab, 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA

Alloy 600 has been used extensively for steam generator tubing and other components in pressurized water reactors. However, these tubes are susceptible to intergranular stress corrosion cracking (IGSCC) in the roll transition areas and the first row U-bends on the primary

water side. Evidence in recent years has indicated that model alloys with lower creep resistance tend to have higher amounts of cracking. Work at the University of Michigan on high purity Ni-16Cr-9Fe has shown that the creep rates can be between a factor of 2 to 10 higher in primary water than in an inert environment. This presentation focuses the effects of the environment on the creep behavior of ultra high purity Ni-16Cr-9Fe. A possible mechanism by which the environment can enhance creep is by hydrogen enhanced plasticity. It is proposed that hydrogen from the corrosion process enters the metal and effectively "shields" dislocation stress fields. The result is easier dislocation motion and thus enhanced plasticity. For Ni, at room temperature, it has been shown that the activation area, a measure of the size of dislocation stress fields, is reduced when hydrogen is present. Load drop tests were conducted at 360°C to evaluate this effect in primary water and argon environments. The effect of the environment on the creep rate as a function of stress and on the activation area were studied. The results showed that the internal stress of the alloy is lower when samples are tested in primary water compared to argon, indicating a reduction in the strength of barriers to dislocation motion. Also, the activation area was smaller by about 21% in primary water than in argon. Both of these results support a hydrogen enhanced plasticity mechanism of enhanced creep for Ni-base alloys at 360°C. To confirm that faster creep rates result from hydrogen enhanced plasticity, constant load tests were conducted in atomic hydrogen generated by dissociating hydrogen gas over a hot tungsten filament. This facilitated constant hydrogen charging at 360°C without using extreme gas pressures or an aqueous environment. Primary and steady state creep rates were obtained in both hydrogen and vacuum environments. Also, stress drops experiments were conducted to determine the activation area in the presence of hydrogen. These results will be discussed in terms of the hydrogen enhanced plasticity model and its relevance in the enhanced creep and intergranular cracking of high purity Ni-16Cr-9Fe alloys.

10:30 AM

DEUTERIUM IRRADIATION EFFECTS IN COPPER COATINGS ON 6061-AL SUBSTRATES: *M. Alam*¹; *M. Y. Inal*¹; ¹New Mexico Tech, Department of Materials Engineering, Socorro, NM 87801 USA

The US DOE is considering production of tritium via the reaction between spallation neutrons and ³He contained inside thin walled 6061-Al tubes [$n(3\text{He},p)3\text{H}$]. The tritium ions will be produced with a high kinetic energy, causing a significant fraction to be embedded into the tube wall. The embedded tritium would not only be effectively lost, but may diffuse through the wall and be released into the surrounding water. This contribution deals with evaluating copper coatings on 6061-Al substrates as potential tritium permeation barriers. The coatings are prepared by the RF magnetron sputtering method. The deposited coatings are characterized for morphology (SEM), surface topography (STM), phase structure (XRD), residual stress (XRD) and bond strength (pull testing) prior to irradiation. Irradiation by deuterium ions is accomplished in a 200 kV accelerator. The variables include ion energy, fluence and irradiation time. The irradiated specimens are re-characterized by the above mentioned techniques. Deuterium depth profiles are obtained by SIMS. The changes induced by irradiation are being studied and the results will be discussed.

MECHANISMS AND MECHANICS OF COMPOSITE FRACTURE: Particle 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, UES, Inc., Dayton, OH 45432-1894 USA; Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

Thursday AM Room: Paris A
October 15, 1998 Location: O'Hare Hilton Hotel

Session Chair: Sunil G. Warrier, UES, Dayton, OH 45432

8:30 AM

QUANTITATIVE MEASUREMENTS OF CLEAVAGE AND DIMPLE FORMATION IN THE FRACTURE OF Al-SiC COMPOSITES:

*Edward Y. Chen*¹; *Lawrence R. Lawson*²; *M. Meshii*³; ¹GE Corporate Research & Development, Physical Metallurgy Laboratory, P. O. Box 8, Schenectady, NY 12301 USA; ²Metallurgist, 226 Interstate Parkway, Bradford, PA 16701-0286 USA; ³Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

This study examines cleavage and dimple formation in the fracture toughness testing of two composites having similar aluminum alloy matrices but different forms of silicon carbide reinforcement, particulate and whisker. Cleavage, not often observed in FCC metals without the presence of some form of chemical embrittlement (e.g. liquid metal or hydrogen), is seen in the whisker reinforced but not in the particulate reinforced composite. Whisker reinforcement is seen to induce a greater degree of constraint and to provide fractured whisker sites which could provide high-modulus crack nuclei. Fractured particulate in the particle-reinforced composite, although less common than whisker fracture, also provides high modulus nuclei. However, these nuclei are not associated with cleavage facets in the matrix. Analysis of the shapes and spacing of reinforcement suggests that, in addition to a nucleus, there must be a sufficient shadow of constraint provided by the second phase to allow a cleavage crack to grow. The cleavage crack would appear to grow from a sharp nucleus within the high-modulus phase into the shadow. The analysis obtaining these results was performed by application of stereological methods to fractography using models of fracture toughness for correlation. It is suggested that for the same volume fraction of reinforcement, whisker reinforcement is likely to result in lower fracture toughness than particle reinforcement through its capacity to induce cleavage.

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FATIGUE CRACK GROWTH IN A BIMATERIAL Al 2124-(SiCp+Al2124): *N. Yang*¹; *P. Bowen*¹; ¹The University of Birmingham, School of Metallurgy and Materials, Edgbaston, Birmingham B15 2TT UK

The fatigue crack growth resistance of a bimaterial Al 2124-(SiCp+Al 2124) has been investigated for crack growth perpendicular to the bimaterial interface. The bimaterial is an Al 2124 alloy and Al 2124 matrix composite reinforced with 22.5vol.% silicon carbide particles and was processed using powder metallurgy. These two components have different elastic moduli and thermal expansion coefficients. The study shows that the fatigue crack growth of the bimaterial is influenced markedly by residual stresses due to mismatch of thermal coefficient of the two components in the as-received bimaterial. The crack can penetrate the bimaterial interface without much diffi-

culty when approaching the interface from the Al 2124 alloy side, but it can arrest before the interface when approaching the interface from the composite side, and depending on the value of the effective stress intensify factor range. Weight functions are used to quantify these observations and finite element methods are also used to quantify behaviour close to the bimaterial interface.

9:20 AM INVITED PAPER

CREEP OF MOLYDISILICIDE COMPOSITES: *K. Sadananda*¹; C. R. Feng¹; M. G. Hebsur²; ¹Naval Research Laboratory, Washington, DC 20375 USA; ²NASA Lewis Research Center, Cleveland, OH 44135 USA

Creep behavior Molydisilicide with Si₃N₄ reinforcements has been investigated as a function of stress at 1200 C under tension and compression. Creep rates are lower in compression and are related to the possible suppression of void formation. Effect of reinforcement, whiskers versus particulates, were also investigated. Theoretical analysis indicated that alloying element, such as Al is likely to go into Si position and increase the metallic bond between Mo and Al at the expense of covalent bond. This is expected to increase the metallic nature and reduce their brittle nature. Experiments are done therefore to evaluate the creep rates with the addition of Al. Since there is always some glassy phase (SiO₂) at grain boundaries, Al can reduce silica by forming Al₂O₃ at the boundaries. To evaluate the relative effects of Al versus Al₂O₃ on creep, effect of Al₂O₃ additions were also investigated. The trends in the behavior and the mechanisms underlying the creep will be elucidated.

9:45 AM INVITED PAPER

SUBCRITICAL CRACK GROWTH PROCESSES IN SiC/SiC COMPOSITES: *R. H. Jones*¹; C. A. Lewinsohn¹; C. H. Henager¹; ¹Pacific Northwest National Laboratory, Richland, WA USA

Time-dependent cracking can occur in SiC/SiC composites by a fiber relaxation mechanism (FRM), interphase removal mechanism (IRM) or oxidation embrittlement mechanism (OEM). These processes occur at different environmental conditions and exhibit different effects on the material. FRM occurs over a range of temperatures but at very low O₂ concentrations while both OEM and IRM require the presence of O₂. IRM occurs at T > T_g, the glass transition temperature for SiO₂, and pO₂ less than atmospheric pressure while OEM occurs at T < T_g and atmospheric pressure of O₂. Other possible crack growth mechanisms include a viscous interphase mechanism (VIM), where the fiber/matrix interphase is viscous, and a fiber embrittlement mechanism (FEM). Neither of these latter two processes have been demonstrated in SiC/SiC composites, however, they are feasible in these materials. Evidence for the FRM, IRM and OEM processes will be given and the conditions at which each occurs will be described. * Research supported by the office of Basic Energy Sciences of the U.S. Department of Energy.

10:10 AM

THERMAL STRESSES IN IN SITU Cr-Cr₂Nb COMPOSITES: PART I, THEORY: *Sanboh Lee*¹; P. K. Liaw²; C. T. Liu³; Y. T. Chou¹; ¹National Tsing Hua University, Department of Materials Science, Hsinchu, Taiwan Republic of China; ²The University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; ³Oak Ridge National Laboratory, Metal and Ceramic Division, Oak Ridge, TN 37831-6115 USA

In Part I of this paper, thermal stresses due to a spheroidal inclusion were investigated using an equivalent inclusion approach proposed by Eshelby. The temperature inside the spheroid is maintained constant and different from that of the surrounding matrix of an infinite extent. A new relationship between the Cartesian coordinates and spheroidal coordinates was established. Based on this relationship, the solution for a prolate spheroidal inclusion was readily obtained from that for an oblate spheroidal inclusion. However, it is difficult to simplify the relationship between the solutions for a prolate spheroidal inclusion and for an oblate spheroidal inclusion using the conventional coordinate transformation. The principal stresses

inside the spheroid increase with decreasing m, the ratio of shear moduli of the spheroid and matrix. Among all principal stresses, the maximum tensile stress is located at the interface between the inclusion and matrix. The numerical results are in agreement with those reported in the literature. The present theory was used to predict the cracking condition in dual-phase in-situ Cr-Cr₂Nb composites, as shown in Part II of this paper. This research is sponsored by the Fossil Energy AR & TD Program (R. R. Judkins program manager), U.S. DOE, and the National Science Council, Taiwan, Republic of China.

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THERMAL STRESSES IN IN SITU Cr-Cr₂Nb COMPOSITES: PART II, APPLICATION: *Sanboh Lee*¹; P. K. Liaw²; C. T. Liu³; Y. T. Chou¹; ¹National Tsing Hua University, Department of Materials Science, Hsinchu, Taiwan Republic of China; ²The University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; ³Oak Ridge National Laboratory, Metal and Ceramic Division, Oak Ridge, TN 37831-6115 USA

The influence of thermal stresses on the microstructures of in situ Cr-Cr₂Nb composites has been investigated. Three cases were studied: (1) Cr-rich particles surrounded by the eutectic network, (2) Cr₂Nb particles embedded in the Cr-rich matrix, and (3) Cr-rich particles inside the Cr₂Nb matrix. The micrographs show that cracks appear on the interface between the particle and matrix. These phenomena are explained by the thermal stresses due to spheroidal inclusions. The maximum tensile stress and the absolute maximum shear stress were found to be located at the interface for the particles with all aspect ratios. The theoretical analyses support the formation of crack embryos at the interface, as the tensile stress exceeds a critical value. In a similar situation, interface decohesion takes place as a result of dislocation pile-ups. The thermal-stress calculation predicts the crack nucleus formed by dislocation pile-ups.

11:00 AM

THE CYCLIC FATIGUE AND FRACTURE BEHAVIOR OF Al₂O₃ PARTICULATE-REINFORCED 2014 ALUMINUM ALLOY METAL-MATRIX COMPOSITES: *T. S. Srivatsan*¹; M. Manoharan²; ¹The University of Akron, Department of Mechanical Engineering, Akron, OH 44325 USA; ²Nanyang Technological University, School of Applied Sciences, Nanyang Avenue 639-798 Singapore

A study was undertaken with the objective of rationalizing the influence of the discontinuous ceramic particulate reinforcements on the mechanical response of 2014 aluminum alloy metal-matrix composites. The cyclic strain amplitude-controlled fatigue properties and fracture characteristics of the 2014 aluminum alloy metal matrix discontinuously-reinforced with varying amounts of Al₂O₃ particulate reinforcements will be highlighted. Specimens of the discontinuously-reinforced aluminum alloy composite were cyclically deformed over a range of cyclic strain amplitudes, using fully-reversed tension-compression loading, giving cyclic fatigue lives of less than 10⁴ cycles. The influence of test temperature on cyclic strain resistance and stress response was critically examined. In this presentation, the intrinsic mechanisms and micromechanisms controlling the strain resistance and stress response characteristics will be highlighted and discussed in light of composite microstructural effects, deformation characteristics of the matrix, cyclic strain amplitude, response stress and test temperature

11:25 AM

CREEP CAVITATION IN DISPERSION STRENGTHENED ALUMINUM WITH HIGH VOLUME FRACTION OF Al₂O₃ PARTICLES: *B. Q. Han*¹; D. C. Dunand¹; ¹Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Creep cavitation was studied by density measurements in pure aluminum containing high volume fractions of submicron alumina dispersoids. In all experiments, the cavity fraction increases linearly with strain, i.e. the ratio of cavity fraction to strain is constant. This cavitation ratio ($\dot{\epsilon}$) decreases with increasing stress at constant tem-

perature; (ii) is weekly dependent of temperature at constant stress; (iii) increases with increasing volume fraction of dispersoids at constant stress and temperature. The cavitation ratio was similar for materials with micron- and millimeter-size grains, indicating that cavitation occurs at dispersoid/matrix interfaces, not at matrix grain boundaries. Assuming that cavities nucleate continuously at dispersoid/matrix interfaces and that the rate of cavity growth is controlled by coupled diffusional flow and power-law creep, a model for the cavity volume as a function of strain, stress, temperature and volume fraction of dispersoids is developed for composites with high volume fraction of dispersoids. The theoretical predictions are in a reasonable agreement with the experimental results.

11:50 AM

THE TENSILE STRENGTH OF 339 ALUMINUM REINFORCED WITH KAOWOOL FIBERS: A COMPARISON OF T5 AND T6 HEAT TREATMENTS: *William J. Baxter*¹; Anil K. Sachdev¹; ¹GM Research & Development Center, 30500 Mound Road, Warren, MI 48090 USA

This study compares the effects of T5 and T6 heat treatment on the tensile strengths of Kaowool fiber reinforced and unreinforced 339 aluminum. The 339 Al-T6 is stronger than 339 Al-T5, but for a Kaowool/339 Al composite the T5 condition is stronger than the T6. The controlling parameter is the strength of the aluminum dendrites, which is proportional to the concentration of magnesium in the dendrites. In the T5 condition, more than half of the magnesium is in large intermetallics in both the unreinforced alloy and the composite. During a T6 heat treatment, magnesium in the intermetallics is redissolved. In the unreinforced T6 alloy this additional magnesium is retained in the dendrites. But in the T6 composite, the magnesium segregates to the Kaowool/aluminum interfaces softening the dendrites. This factor alone accounts for the low strength of the T6 composites. The tensile strengths of the T5 and T6 composites correspond to the calculated values for a perfectly bonded system.

12:15 PM

OBSERVATIONS OF THE FRACTURE PROCESSES IN A Nb-Si IN-SITU COMPOSITE USING CROSS-SECTIONAL ELECTRON MICROSCOPY: *Richard Grylls*¹; B P Bewlay²; H A Lipsitt¹; H L Fraser¹; ¹The Ohio State University, Department of Materials Science and Engineering, 2041 College Road, Columbus, OH 43210 USA; ²G.E. Corporate Research and Development, P.O. Box 8, Schenectady, New York 12301 USA

Composites based on Nb-Si are attractive candidates for use as structural materials at the very high temperatures required for future generation aircraft engines. Binary Nb-Si alloys typically possess very low room-temperature fracture toughness, but recently a more highly alloyed material has been developed which has fracture toughness of $> 20 \text{ MPam}^{1/2}$. In this paper the fracture behavior of Nb-19Si is compared to that of the more highly alloyed material using cross-sectional transmission electron microscopy (TEM) of the fracture surface of specimens broken in four-point bending. The composites studied in this work were produced by directional solidification, which gives an in-situ composite microstructure consisting of niobium silicide dendrites surrounded by a eutectic mixture of metallic niobium solid solution and niobium silicide. Using cross-sectional TEM it is possible to view directly the micromechanisms of composite fracture, including the details of slip transmission, microcracking, crack bridging, ductile-phase pull-out and interfacial debonding. In this paper the micromechanisms of fracture of the two composites will be compared, and differences in the fracture toughness will be related to the observed deformation microstructures.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VII: Nanostructured and Graded Materials

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

Thursday AM Room: Chicago
October 15, 1998 Location: O'Hare Hilton Hotel

Session Chair: Dr. C. Koch, North Carolina State University, Department of Materials Science and Engineering, Raleigh, NC 27695-7907 USA

8:30 AM INVITED PAPER

NANOSTRUCTURED MATERIALS FOR STRUCTURAL APPLICATIONS: PROMISE AND PROGRESS: *Carl C. Koch*¹; ¹North Carolina State University, Department of Materials Science and Engineering, Raleigh, NC 27695-7907 USA

There has been growing interest in nanostructured materials—materials with microstructural dimensions such as grain size \bar{y} 100 nm—for potential structural applications. This interest is due to both the experimental results which indicate high strength and hardness for nanostructured materials as well as predictions of ductility and toughness in normally brittle materials or even superplasticity at relatively low homologous temperatures. The many methods for synthesis of nanostructured materials can be classified into two groups. One involves particulates which must be compacted into bulk form. The other, such as devitrification, electrodeposition, or physical vapor deposition, is a “one-step” process. The artifacts/structural defects which can occur during particulate compaction can influence certain mechanical properties dramatically. This review will contrast the mechanical behavior of nanostructured materials made by “one-step” vs “two step” processes as well as review our present understanding of mechanical properties of this new class of materials.

9:00 AM INVITED PAPER

BULK MECHANICAL ALLOYING FOR FABRICATION OF NANO-STRUCTURED MATERIALS: *Tatsuhiko Aizawa*¹; ¹University of Tokyo, Department of Metallurgy, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113 Japan

The milling type and attriting type mechanical alloying methods have been widely utilized to yield various non-equilibrium phase materials in the order of grams. In addition to the small yield on the route of those mechanical alloying methods, their fatal demerits to industrial production are: 1) Long time processing, 2) Little capability for precise process control, and 3) Significant contamination from vial wall and balls. In order to overcome the above difficulties and to promote the intrinsic features to the mechanical alloying, author has been developing new mechanical alloying process: bulk mechanical alloying. Completely different from the conventional mechanical alloying methods, no vials nor balls are necessary in processing; the constituent element powder or granule mixture, or, solidified alloy bulk solids or agglomerates can be pored into a die cavity and subjected to the specified cyclic loading at Ar atmosphere in ambient temperature. With increase of the number of cycles in loading, the starting materials are uniformly mixed, homogenized, refined and alloyed. The systematic studies have been done during several years to have demonstrated that 1) 500 to 1000 cycles (4 to 8 ks) are sufficient to yield the final stage phase for each targeting material system, 2) little

or no contamination is expected in principle of this processing, 3) solid or fluid paraffin-wax are sometimes necessary to reduce the friction between samples and die material without oxidation or other reactions, or 4) warm pressing is also effective to consolidate the mechanically alloyed green samples. In the present study, Cu-Co and Al-Si systems are employed to produce nano-granular and nano-precipitate materials in the order of 100 grams and to control the magneto-resistance and the mechanical strength by tuning the nano-size of the secondary phase.

9:30 AM INVITED PAPER

PROCESSING OF METALLIC NANOSTRUCTURES BY ELECTRODEPOSITION TECHNIQUES: *F. Ebrahimi*¹; D. Kong¹; T. Mathews¹; Q. Zhai¹; ¹University of Florida, Materials Science & Engineering Department, Gainesville, FL USA

Electrodeposition techniques have been employed in processing nanocrystalline metals and laminated nanocomposites. These materials exhibit very high strength levels and have diverse applications from manufacturing tools for die casting to giant magneto resistive films for magnetic data recording. While thin films (<10 μm) can be processed relatively easily by electrodeposition, processing of bulk materials (electroforming) has remained to be a challenge. This paper discusses the effects of processing parameters, such as pulse profile, composition, pH, temperature and agitation on macroscopic defect structure, microstructure, and tensile properties of pure copper and nickel metals and laminated copper/nickel and copper/silver nanostructures.

10:10 AM

PROCESSING AND MECHANICAL PROPERTY OF FULL-DENSITY IN SITU Cu-Fe NANOCOMPOSITES CONSOLIDATED FROM MECHANICALLY ALLOYED POWDERS: *Li He*¹; Larry F. Allard²; Evan Ma¹; ¹Louisiana State University, Mechanical Engineering, RM 2420, CEBA, Baton Rouge, LA 70803 USA; ²Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831 USA

A new route has been developed to produce full-density, bulk, two-phase nanocomposites. Nanocrystalline supersaturated solid solution Cu_{100-x}Fe_x (x=40, 85, 100) powders were produced by mechanically alloying of Cu and Fe at room temperature or liquid nitrogen temperature. These supersaturated solid solutions were decomposed on nanoscale in situ upon hot consolidation, forming Cu-Fe two-phase nanocomposites. Fully dense composite samples have been obtained using constrained sinter forging, which features near-net-shape manufacturing. Their microstructures were characterized using transmission electron microscopy. The Cu and Fe phase domains and their distributions were analyzed using energy dispersive X-ray spectroscopy with a focused electron beam. The average domain/grain sizes of Cu and Fe observed were well below 100 nm, confirming the formation of nanocomposites. The mechanical property of these nanocomposites will be studied using compression test. The strengthening mechanism will be examined in terms of interphase boundary strengthening, solid solution hardening, precipitation hardening, and dispersion hardening.

10:30 AM

MECHANICAL PROPERTIES OF GRADED MATERIALS: *Franz Josef Kahlen*¹; ¹University of Central Florida, CREOL, 4000 Central Florida Blvd, Orlando, FL 32816-2700 USA

The authors present a one-step manufacturing tool to create graded materials suitable for industrial applications. Material powders are melted and form planar, two-dimensional geometries as the substrate is moved under the laser beam in the XY-direction. After completing the geometry in the plane, the substrate is displaced in the Z-direction, and a new layer of material is placed on top of the just-completed deposit. By continuous repetition of this process, 3D parts were created. The material composition was altered by mixing different material powders prior to their delivery to the laser beam - substrate interaction region. A high power CO₂ laser was used as the energy source, material powders under investigation were Stainless

Steel SS304L, Copper, and Aluminum. Helium was used as the shield gas at a flow rate of 15 l/min. The incident CO₂ laser beam power was varied between 300 W and 400 W, with the laser beam intensity distributed in a donut mode. The laser beam was focused to a focal diameter of 600 μm . The local material composition was investigated, to judge the effectiveness of the mixing just before deposition. Strength and hardness of the created components were measured and phases identified.