10TH INTERNATIONAL SYMPOSIUM ON EXPERIMENTAL METHODS FOR MICROGRAVITY MATERIALS SCIENCE: Session II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Thermodynamics & Phase Equilibria Committee, Jt. Electronic, Magnetic and Photonic Materials Division/Structural Materials Division, Alloy Phases Committee, NASA Microgravity Sciences

Program Organizers: Robert Schiffman, R.S. Research Inc., Crystal Lake, Barton, VT 05822; Carlo Patuelli, Universita di Bologna, Depatrmento di Fisica, Bologna I-40126 Italy

Monday PM Room: Plaza Room A
February 16, 1998 Location: Convention Center

Session Chair: J. B. Andrews, University of Alabama at Birmingham, Dept. of Materials and Mechanical Engineering, Birmingham, AL 35294

2:00 PM

REAL-TIME X-RAY MICROSCOPY OF Al-Cu EUTECTIC SOLIDIFICATION: *Dr. William F. Kaukler*[1]; Dr. Peter A. Curreri[2]; Dr. Subhayu Sen[3]; [1]The University of Alabama in Huntsville, CMMR, Huntsville, AL 35899 USA; [2]NASA/MSFC, ES75, MSFC, AL 35812 USA; [3]Universities Space Research Association, MSFC, AL 35812

Recent improvements in the resolution of the X-ray Transmission Microscope (XTM) for Solidification Studies provide microstructure feature detectability down to 5 micrometers during solidification. This presentation will show the recent results from observations made in real-time of the solid-liquid interfacial morphologies of the Al-$CuAl_2$ eutectic alloy. Lamellar dimensions and spacings, transitions of morphology caused by growth rate changes, and eutectic grain structures are open to measurements. A unique vantage point viewing the face of the interface isotherm is possible for the first time with the XTM due to its infinite depth of field. A video of the solid-liquid interfaces seen in-situ and in real-time will be shown.

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SURFACE DEFORMATION AND MARANGONI CONVECTION IN ELECTROSTATICALLY-LEVITATED DROPLETS: S. P. Sond[1]; *B. Q. Li*[1]; [1]Louisiana State University, Dept. of Mechanical Engineering, Baton Rouge, LA 70803

A mathematical representation of surface deformation and Marangoni convection in electrostatically-levitated droplets in microgravity is developed. The numerical model is based on the boundary element solution of the Maxwell equations, and the finite element solution of the transport equations. Mathematical formulation and computational procedures are discussed. A selection of computed results is given. The effects of the applied electric field and charges on the surface deformation and also the effect of the laser beam arrangement on the internal fluid flow in electrostatically-droplets are analyzed. The results show that the internal flow in a droplet is driven by the Marangoni force and an appropriate arrangement of heating source is required to minimize the convection of the melt in the droplet.

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CRYSTAL GROWTH WITH MAGNETIC LEVITATION IN TRANSPARENT FERRO-FLUIDS: *Felix Huber*[1]; Walter Littke[1]; Ernst Messerschmid[1]; Thomas Rösgen[1]; [1]Steinsbeis TZ Raumfahrtsysteme, Reutlingen D-72766 Germany

Magnetic Fluids have recently been used as a research subject in microgravity fluids science (EuroMIR'95 experiment of ESA). The interactions of these fluids with external magnetic fields and temperature gradients creates forces non-existent in regular fluids. It is foreseen to use these properties of ferro-fluids to create a liquid levitation system and to perform crystal growth experiments. While being of interest by itself the experiment will derive additional impact from the use of, transparent ferro-fluids. Regular, commercially available ferrofluids are deeply opaque and do not permit optical diagnostics in the bulk. In the proposed experiment, sufficient transparency will be available to monitor directly the crystal growth inside the livitation chamber without the need for intrusive probes. The microgravity environment will permit an optimized trade-off between the magnetic fluid strength and transparency which would not be possible of the ground. Furthermore, a high degree of symmetry is desirable in the levitation trap topology which would be destroyed by the directional influence of gravity.

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MAGNETIC DAMPING OF G-JITTER INDUCED OSCILLATING FLOWS IN THE BRIDGMAN-STOCKBARGER GROWTH PROCESSES: B. Pan[1]; *B. Q. Li*[1]; H. C. de Groh[2]; [1]Louisiana State University, Dept of Mechanical Engineering, Baton Rouge, LA 70803; [2]NASA Lewis Research Center, Cleveland, OH 44135

Finite element models are developed for g-jitter induced transient melt flows and energy and species transport in the Bridgman-Stockbarger single crystal growth processes in microgravity with or without the presence of the external magnetic field. The finite element procedures and a selection of computed results from both 2-D and 3-D are presented. The model is applied to study the effects of a wide variety of g-jitter including synthetic single frequency, multi-frequency as well as real g-jitter measured in space craft in the presence of a magnetic field. The results show that an external magnetic field can be effectively applied to further reduce the g-jitter induced flows and hence provide a better control of solute redistribution in microgravity. Numerical simulations further suggest that magnetic damping effects depend on the strength and orientation of the field. For the cases studied, the damping effect becomes more effective when it is oriented parallel to the thermal gradient.

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MEASUREMENTS OF THE SURFACE TENSION OF LIQUID AND UNDERCOOLED METALLIC ALLOYS BY OSCILLATING DROP TECHNIQUE: *Michael Roesner-Kuhn*[1]; G. Kuppermann[1]; M. G. Frohberg[1]; [1]Technical University Of Berlin, Institute of Metallic Materials, General Metallurgy, Berlin 10719 Germany

The surface tension of liquid metallic alloys is an important parameter for the understanding of the material flow at interfaces. Containerless electromagnetic levitation conditions are causing surface oscillations of liquid droplets. The oscillations are detectable with the temperature-time signal of pyrometers and the corresponding frequencies can be obtained by Fast Fourier Transformation of those signals. The performance of an online analysis of these data during the Spaceshuttle Mission MSL 1 provided for specific experimental conditions in the electromagnetic levitation facility TEMPUS. Investigations have been carried out on high viscous metallic glassformers, pure zirconium and stainless steels.

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COMPARISON OF THREE BULK CONTAINERLESS PROCESSING TECHNIQUES: *C. W. Morton*[1]; W. H. Hofmeister[1]; R. J. Bayuzick[1]; [1] Vanderbilt University, Dept. Of Applied and Engineering Sciences, Nashville, TN 37240 USA

Three containerless processing techniques: drop tube processing, electromagnetic levitation, and electrostatic levitation have been compared on the basis of nucleation temperature distributions obtained by processing the same materials in each method. The undercooling experiments were conducted using mm size samples of zirconium in the l05 m drop tube at George C. Marshall Space Flight Center, an electromagnetic levitator at Vanderbilt University, and an electrostatic levitator at Space Systems/Loral. The samples were prepared from three different grades of zirconium with stock purities of 99.8, 99.95, and 99.995% by weight. About 100 consecutive undercooling measurements were obtained for each sample or sample type in each processing method. Histograms of the nucleation temperatures were constructed and revealed distributions with mean temperatures that decreased with sample purity. The distributions were compared using analysis of variance techniques. Statistically significant differences between the mean nucleation temperatures were detected for the same stock material in the different methods. The results will be discussed in terms of the processing characteristics of each method.

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A. C. MODULATION CALORIMETRY ON $Zr_{57}Nb_5$ $Ni_{15.4}$ $Cu_{12.6}Al_{10}$ AND Zr_{11} Ti_{34} Ni_8 Cu_{47}: MSL-1 RESULTS: *Stephen C. Glade*[1]; David S. Lee[1]; William L. Johnson[1]; [1]California Institute of Technology, W. M. Keck Laboratory for Engineering Materials, Pasadena, CA 91125

We present the results of A. C. modulation calorimetry measurements performed with the TEMPUS facility, which flew aboard MSL-1. TEMPUS is an electromagnetic levitation facility that allows containerless processing of metallic samples in microgravity. Using A. C. modulation calorimetry, it is possible to obtain the thermal conductivity and the ratio of heat capacity to total emissivity of stable and undercooled melts of reactive metals. Experiments were performed on two bulk glass-forming metallic alloys, Zr_{57} $Nb_5Ni_{15.4}$ $Cu_{12.6}$ Al_{10} and Zr_{11} Ti_{34} Ni_8 Cu_{47}.

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DIGITAL IMAGE HOLOGRAPHY FOR DIFFUSION MEASUREMENTS OF MOLTEN SALTS IN SPACE: *J. Richter*[1]; E. Marquardt[1]; M. Großer[1]; [1]Center of Physical Chemistry, Aachen D-52056 Germany

The Center has a long experience in measuring mutual diffusion coefficients in binary salt mixtures. lg and µg data of silver/alkali nitrate and chloride systems have been determined. The diffusion boundary is established as a step profile between two salt mixtures with a small difference in composition. The time dependent changes of the concentration profiles are analyzed with a holographic interferometer. We are using a newly developed high resolution Digital Image Holography (DIH). It overcomes the disadvantages of ordinary real time holography and can be used for telescience or automated experiments in space. In DIH the interference pattern of the object beam, passing through the diffusion cell, and a reference beam is stored using a CCD camera. The highest and lowest interference intensity is determined for each pixel. This allows to calculate the phase shift between two pictures and a macroscopic interference pattern. The pattern is analogue to those obtained from real time holography. In difference to real time holography, the reliable DIH creates high contrast interferograms with spatially constant brightness. No adjustment during measurement is necessary. The main advantage is that the interference patterns can be calculated comparing two freely chosen images, whereas in real time interferometry everything is compared to the state of the diffusion cell at the time when the hologram was taken. Also after the experiment run the operator can decide which combination is the most suitable.

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QUELD II - MIR MATERIALS PROCESSING: *R. W. Smith*[1]; T. Smith[2]; [1]Queen's University at Kingston, Department of Materials and Metallurgical Engineering, Ontario K7L 3N6 Canada; [2]Millenium Biologix Inc., Ontario K7M 7G3 Canada

In October 1992, the Shuttle Columbia carried Astronaut Steve McLean into orbit to perform the package of experiments known as CANEX2. One of these experiments was the "Queen's University Experiment in Liquid Diffusion". Some thirty samples were successfully processed in a manually-operated facility designed and built at Queen's (QUELD I). This apparatus consisted of two isothermal furnaces, sample loading / quenching gear, and furnace controls, all packed into a 15 cm cube! When the opportunity arose to fly the Canadian Microgravity Isolation Mount (MIM) on the Russian Space Platform MIR arose, the CSA invited Queen's to consider a reflight of QUELD I, to be used in conjunction with the MIM. This was welcomed, but further review suggested that an automated minimun-crew-activity facility, QUELD II, might better exploit the flight opportunity. Thereupon, the Queen's Group invited Millenium Biologix (MBI), a principal of which had contributed to the Group's design activities since 1982, to collaborate in the preparation of a conceptual design for QUELD II, which would incorporate the essential elements of the successful QUELD I into a more user-friendly automated facility. To this end, MBI was able to tap into its considerable past experience in the design and production of highly reliable, miniaturized, microprocessor-controlled biomedical devices, and so help to produce a number of ground-based units (GBUs) for individual experimenters to use, and also two flight articles (F.A.s) to go to MIR QUELD II has now been used in three successful MIR experimental campaigns, in association with the Canadian Microgravity Isolation Mount. Its ease of use, simplicity of operation, and reliability have attracted much Russian and NASA acclaim. This research facility will be described and the experimental highlights of its use in furthering our understanding of the physical effects of g-jitter will be presented.

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NOVEL ELECTRIC NUCLEATION TECHNIQUE FOR GROWING LARGE SINGLE CRYSTAL IN SPACE: *Hossin Abdeldayem*[1]; Donald O. Frazier[1]; [1]NASA-Marshall Space Flight Center, Space Science Lab/ES 76, Huntsville, AL 35812

We present, herein, an electrical model for growing crystals without a seed which might not be free of defects and thereby still hinder the growth of a perfect crystal in space. The system is designed to confine nucleation to a single site automatically in an under saturated solution to avoid multiple nucleation. The technique is based on the effect of electrostriction, which is the tendency of a material to become more compressed in the presence of an electric field. The system is designed to create an electrical potential well between two hyperboloid electrodes with applied voltage at low frequency. The induced potential well between the electrodes oscillates at low frequency and attracts the solute and condenses it into the region of maximum field intensity. The alternating voltage prevents molecules with intrinsic charge from being attracted to the electrodes. The continuous presence of the electric field during the duration of the experiment, provides a continuous migration of the molecules toward the trapping site. This will eliminate the creation of a depletion region around the nucleation center and will enhance the crystal growth rate. Aside from the above mentioned advantages, the system is compact, safe to operate, and inexpensive to build.

ADSORPTION, ION EXCHANGE, AND SOLVENT EXTRACTION: Adsorption II

Sponsored by: Extraction & Processing Division, Aqueous Processing Committee, Copper, Nickel, Cobalt Committee, Lead, Zinc, Tin Committee, Precious Metals Committee

Program Organizer: Courtney Young, Montana Tech, Metallurgical Engineering, Butte, MT 59701

Monday PM — Room: Plaza Room C
February 16, 1998 — Location: Convention Center

Session Chair: Larry Twidwell, Montana Tech, Butte, MT 59701, Dan Eyde, GSA Resources, Inc., Cortaro, AZ 85652

1:45 PM PLENARY

THE ADSORPTION OF Au(CN)2- ON ACTIVATED CARBON: G. Poinen[1]; S. M. Thurgate[1]; *I. M. Ritchie*[1]; C. Klauber[1]; [1]A J Parker Cooperative Research Centre for Hydrometallurgy, Murdoch, Western Australia 6150

The carbon-in-pulp (CIP) process has revolutionized the extraction of gold from cyanide leach liquors since its introduction some twenty five years ago. However, the mechanism by which the dicyanoaurate(I) complex is so effectively adsorbed onto the activated carbon surface is not well understood and so has been the subject of active debate. In this paper, our present understanding of the adsorption of Au(CN)2- on activated carbon is first reviewed. This review is then followed by some results concerning the adsorption of Au(CN)2- on highly oriented pyrolytic graphite (HOPG). In situ scanning tunnelling microscopy (STM) was used to image the surface of the HOPG on which the Au(CN)2- had been adsorbed. Ex situ images of the exposed surface were made in a scanning electron microscope (SEM) fitted with an X-ray energy dispersive analyser (EDAX) attachment. Some HOPG surfaces, with Au(CN)2- adsorbed onto them, were subjected to x-ray photoelectron spectroscopic (XPS) analysis. From these measurements, it is proposed that the mechanism of adsorption of Au(CN)2- proceeds in two steps. In the first of these, a small positive ion such as Ca2+ is intercalated into the edges of the HOPG lattice. This is then followed by the adsorption of the negatively charged Au(CN)2- onto the topmost layer of the HOPG. The validity of using HOPG as a model for activated carbon is discussed, and how these results relate to industrial practice considered.

2:30 PM INVITED

OBSERVATIONS OF GOLD IODIDE ADSORPTION ON HIGHLY ORIENTED PYROLYTIC GRAPHITE: *J. B. Hiskey*[1]; D. W. Collins[1]; [1]University of Arizona, Materials Science and Engineering, Tucson, AZ 85721

The uptake of gold from halide solutions by activated carbon has been investigated using various techniques. Halide systems typically exhibit combined adsorption - reduction type behavior. In an attempt to gain more information about the nature of these processes, samples of synthetic highly-oriented pyrolytic graphite (HOPG) were contacted with aqueous solutions of gold-iodide. HOPG represents a model structure for activated carbon comprising of basal-plane sites, edge sites, and various defect sites. Two basic instrumental methods were used to examine the uptake of gold by HOPG. The first was standard X-ray photoelectron spectroscopy (XPS). The other involved several scanning probe techniques. XPS clearly indicated a preferred uptake of gold at edge sites. However, iodine appeared to be distributed on both basal-plane and edge sites. At low iodine additions, tapping mode AFM showed that clusters were concentrated along steps and surface defects. At high iodine concentrations, the HOPG surface was uniformly covered by iodine.

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COCONUT SHELL IN REMOVAL OF CADMIUM IONS FROM INDUSTRIAL EFFLUENTS: *Aida Espinola*[1]; Rupen Adamian[1]; Lola M. Braga Gomes[2]; [1]COPPE/UFRJ, Rio de Janeiro 21945-4746 Brazil; [2]Instituto de Quimica/UFRJ, Rio de Janeiro 21945-970 Brazil

Cadmium, even in small doses, is detrimental to the environment and toxic to humans. Minero-metallurgical as well as electroplating industrial effluents often contain cadmium, a serious pollutant of water ways, where it may be absorbed by animals used as food. Several methodologies are usually employed for the aqueous effluents treatment, most usually chemical precipitation, adsorption and electrolytic processes. Precipitation is not effective for cadmium removal because of the high Ksp and the high pH required; only sulfide would be efficient in cases of very small amounts. Some vegetal refuses have shown to be practical for an adsorption process. We have applied coconut (Cocos nucifera) shells which are extremely abundant over most of the extensive Brazilian coast. It has been verified, in our laboratory, that the fiber of this coconut is rich in chemical compounds active in the adsorption of Cd2+. We have established, as relevant variables for the optimization of the process, the previous treatment of the fiber in order to wash out soluble components, the pH and the proportion of mass of fiber/volume of solution. This way we were able to obtain 99% removal of the metallic contaminant, which results in a remaining concentration of only 0.10 ppm Cd2+ which attends to the maximum impurity legally established by the environmental control agency of Rio de Janeiro (FEEMA) for Cd2+ in aqueous effluents to be disposed in rivers and the Guanabara Bay area.

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GOLD RECOVERY FROM BISULFIDE BIOLEACHATE SOLUTIONS: *C. A. Young*[1]; J. Lloyd[1]; R. Ziolkowski[1]; T. Barnett[1]; R. M. Hunter[2]; M. J. Floersch[2]; [1]Montana Tech, Metallurgical Engineering, Butte, MT 59701; [2]YES Technologies, Bozeman, MT 59715

YES Technologies has developed a three-stage bioleaching process for extracting gold from ores using a non-cyanide lixivant. In the first stage, aerobic bacteria are used oxidize sulfidic ores to form acid-rock drainage (ARD). Resulting ARD is then collected and neutralized with sulfate-reducing bacteria to produce bisulfide (HS-). Finally, the generated bisulfide is captured and used to leach gold. Extraction efficiencies as high as 75% have been achieved resulting in solution concentrations high enough for recovery by conventional hydrometallurgical processes. This paper discusses the preliminary results obtained with carbon adsorption and cementation experiments along with the theory and development of the bioleaching process.

4:00 PM INVITED

USING CABSORB ZS500RW FOR REMEDIATION OF THALLIUM IN CONTAMINATED EFFLUENTS: *Dan Eyde*[1]; Tod Johnson[2]; Jerry Hanley[1]; [1]GSA Resources, Inc., Cortaro, AZ 85652; [2]Filter Flow Technology, League City, TX 77573

A western mining company has detected low levels of thallium near 0.05 ppm in its water drainages requiring remediation under new regulatory standards which allow discharges of no more than 0.0017 ppm. GSA Resources, Inc. and Filter Flow Technology have developed an effluent treatment system that satisfied the regulatory standard. The system minimized capital investment in new equipment by converting existing down-flow carbon beds into a series of ion exchange columns and packed with CABSORB ZS500RW, a zeolite compound. Bench tests with solutions containing 0.9 ppm and pilot scale tests with the 0.05 ppm effluent were reduced in a single pass to less than discharge requirements. It is critical to note that there are a number of conventional technologies that can remediate liquid waste discharges to low (>10) ppm economically; however, current or proposed regulatory standards throughout the world mandate to low ppb levels, which can often be below analytical detection limits, as was the case here. Few existing waste treatment schemes can meet this technical challenge. Test work is ongoing to determine optimal flow rates using a pilot scale system treating 15-25 gpm of effluent. Testing is also underway to evaluate different methods of disposal for the spent zeolite. It may be economical to recover the thallium metal which sells for approximately $200/kg.

4:25 PM INVITED

USE OF NATURAL ZEOLITES AS AN ION-EXCHANGE MEDIA IN RADIOACTIVE WASTE CLEANUP PROGRAMS: *Robert M. Colpitts, Jr.*[1]; [1]American Resource Corporation, Inc., Operations Office, Reno, NV 89502 USA

Over the past 15 years, several groups have tried to use natural zeolites to clean soils, ground and surface waters, and food items such as milk contaminated with various radioactive isotopes. A growing success story is the use of clinoptilolite (high-silica heulandite) in the adsorption of Strontium-90 and Cesium-137, especially in the wake of the Chernobyl disaster. Clinoptilolite was found to preferentially adsorb these isotopes via the exchange of resident cations in the clinoptilolite (such as Na, K, Mg, and Ca). As a soil amendment, the clinoptilolite is plowed into the soil and left to do its work. Studies show that clinoptilolite reduces the uptake of the isotopes from the soil by plants by as much as 90% depending on the species of plant and level of contamination. As a filtration media in a treatment facility, the clinoptilolite is loaded into filtration columns connected in series. Contaminated water is then pumped through the columns. Over time, the exchange process slows down as resident cations are progressively exchanged. The efficiency of the exchange depends on several factors

including clinoptilolite purity, media Cation Exchange Capacity (or CEC), media grain size, and contact time. The purity depends on the deposit as does the CEC. Grain size is controlled in the manufacturing process. Experience shows that a high-purity (>85%) clinoptilolite twice screened in a -20+35 mesh size provides the most reasonable filtration media. Uniformity and purity of the material is controlled with a Quality Assurance/Quality Control program at the manufacturing plant. The CEC can be enhanced by eluting the clinoptilolite with a 3% NaCl solution, although this creates salt water disposal problems.

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SEPARATION PROCESS FOR TREATMENT OF COPPER MINE LEACHATE: *Richard W. Helsel*[1]; Brian T. Park[2]; Richard F. Hammen[3]; [1]IT Corporation, Knoxville, TN 37923; [2]MSE Technology Applications, Inc., Butte, MT 59701; [3]ChelaTech, Inc., Missoula, MT 59802

A technology demonstration project sponsored by the Department of Energy was conducted using a new metal separation/concentration process applied to acidic water in the Berkeley Pit, a former copper mine in Butte, MT. The project involved initial bench-scale media production and screening studies, design and construction of a mobile two l/min pilot plant, operation of the system for several months at MSE's facilities in Butte, data analysis and process modeling, and development of a conceptual design and cost estimate for a full scale process. The technology demonstration goals were to (1) remove all metals to below water quality (Gold Book) standards, (2) recover the major portion of iron, copper, aluminum, zinc and manganese as sulfate brines amenable to use in an existing market, and (3) minimize the production of wastes. The process utilized a sequence of columns containing special porous silica media having selective functional groups attached to the end of hydrophilic spacer molecules. The media characteristics provide a combination of the benefits of solution phase equilibration kinetics and heterogeneous support media. Each column recovered a specific metal through a cycle of loading, rinse, elution, rinse, regeneration and rinse. Lessons learned during the pilot testing led to improvements in the media that were incorporated into a 12,000 l/min conceptual plant.

ALUMINA & BAUXITE: Bauxite and Digestion

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Jean Doucet, Alcan International, PO Box 6090, Montreal, Quebec H3C 3A7 Canada

Monday PM — Room: Fiesta A
February 16, 1998 — Location: Convention Center

Session Chair: Millind Chaubal, Reynolds Metals Co., Aluminum Division, Corpus Christi, Texas 78469 USA

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RIETVELD ANALYSIS OF POOR-SETTLING JAMAICAN BAYER MUDS: *Mr. Wilmon Wallen-Bryan*[1]; [1]Jamaica Bauxite Institute, Kingston 6, Jamaica West Indies

Red mud mineralogy, along with liquor composition, the techniques of polymer preparation and addition to the residue from digestion, and digest temperature constitute the four (4) main factors affecting the settling behavior of Bayer residues. This paper focuses on the mineralogy of residues from three (3) occurrences of plant disruptions caused by scaling-problems spanning a seven (7) year time period and occurring at two different alumina refineries operating in Jamaica. These breakdowns were manifested as poor overflow-liquor clarity and poor compaction of underflow-solids. At the Jamaica Bauxite Institute, X-ray powder diffractograms of these samples were subjected to Rietveld analysis, a software tool which compares measured and calculated scattered intensity profiles in a least-squares sense and adjusts pre-assigned lattice parameters (and other physical variables) to calculate a "good-fit" profile. It appears that aluminum substituted goethite with less than five (5) mole-percent substitution may be a recurring component of these mishaps. This suggests that pure goethitic is more difficult to compact with typical Bayer-mud flocculants and that aluminum substitution in fact makes for more stable flocs. At the other extreme of substitution (~33 mole-percent) the flocs again may become unstable.

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AN EVALUATION OF ALTERNATIVE BAUXITES FOR KAISER'S BAYER PLANT IN GRAMERCY LOUISIANA: *Dr. Tom L. Capron*[1]; [1]Kaiser Aluminium, Kaiser Alumina Technical Services Incorporated, Gramercy, Louisiana 70052 USA

Kaiser's Plant in Gramercy Louisiana has been processing bauxite, from Jamaica's north coast, for 40 years, using Bayer Process technology. The Kaiser Bauxite mining plan with the current Jamaican Government reserve allocation predicts that bauxite quality will not be suitable for sweetening beyond the year 2003. To avoid future production curtailments, Gramercy began a program to search for low monohydrate alternative bauxite sources. One part of the program was to survey and analyze samples from the Defense Logistics Agency (DLA), formally the GSA, stockpiles. Major processing problems with DLA bauxites are high reactive silica, causing high soda losses, and low iron, resulting in poor product quality. This can be compensated for by blending alternative bauxites with Jamaican bauxite. Another problem with DLA bauxites is that they require grinding to prepare them for digestion. This problem can be solved with capital for grinding and handling equipment. Part of the Gramercy Strategic Plan is to secure a supply of low monohydrate bauxite for future production requirements. The DLA is one source that can supply Gramercy's future needs. The Gramercy plant can process DLA bauxites and merchant bauxites while maintaining product quality and production.

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SOME GEOLOGICAL, MINERALOGICAL AND TECHNOLOGICAL FEATURES OF GUJARAT BAUXITES, INDIA: *Dr. Alexander G. Suss*[1]; *Mr. Eugeny P. Kovalenko*[3]; Mr Ashok K. Nandi[2]; [1]VAMI, Russian National Aluminium-Magnesium Institute, Sredny pr., 86 Russia; [2]Jawaharlal Nehru Aluminum RDD Centre, Wadi, Nagpur 440 023 India; [3]Nikolaev Alumina Plant, Nikolaev Ukraine

Bauxite deposits in Gujarat State (India), located on two sides of Rann of Kachchh along the Arabian sea are known for high grade bauxite suitable for refractory and abrasive industries. The overall bauxite reserves in Gujarat are estimated to be 100 million tonnes. Bauxite bearing plateaux are about 50 to 100 meters above mean sea level and high grade white bauxites (Al2O3 ~60%, Fe2O3 >5%) intermixed with laterites/medium grade red bauxites (Al2O3 ~50%, Fe2O3 >10%) occur as irregular and discontinuous orebody. The thickness of orebody varies from 2 to 4 meters and at places, it is covered by Gaj limestone and thin cover of soil. Bauxite is developed on brecciated basalt and traps. Most of bauxite is gibbsitic in nature, however some ore bodies with significant boehmite and diaspore content are also encountered. Textural/structural features, mineralogy and technological properties of medium grade red bauxite of Gujarat for production of alumina were investigated. Given bauxite is characterized by low humidity (<1%) and is fairly hard for crushing and grinding as compared to other lateritic bauxites.

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CONTINUOUS PLUG FLOW PRECIPITATION OF SODALITE SCALE ON HEAT TRANSFER SURFACES: ***Mr. Jonas Addai-Mensah***[1]; [1]Ian Wark Research Institute, University of South Australia, Adelaide 5095 South Australia

Isothermal, continuous, plug flow precipitation of sodalite scale from synthetic, spent Bayer liquor at the surfaces of stainless steel substrates has been investigated. Under non-boiling and solution conditions close to those prevailing in the heat exchanges of Bayer process plants, the sodium aluminosilicate crystalline phase which precipitated within 4 hours at 160° was carbonate-rich sodalite. The rate of scale layer coverage and growth on steel surfaces were observed to increase with increasing solution SiO_2 supersaturation in a distinctly non-linear manner but were independent of the solution laminar, flow velocity and

the degree of surface roughness of the steel substrates. The degree of SiO_2 supersaturation had a marked effect on the mechanism of growth and morphology of the primary particles of scale. Further, increasing solution Na_2CO_3 concentration significantly increased the rate of scale formation.

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MATHEMATICAL MODELING OF THE KINETICS OF GIBBSITE EXTRACTION AND KAOLINITE DISSOLUTION/DESILICATION IN THE BAYER PROCESS: *Dr. Narasimha S. Raghavan*[1]; [1]Alcan International Limited, Kingston Research and Development Centre, Kingston, Ontario K7L 5L9 Canada

An unsteady-state mathematical model incorporating the rates of external mass transfer, intraparticle diffusion and chemical reaction has been developed to describe the extraction of gibbsite from bauxite particles allowing for the competitive reactions - gibbsite and free caustic, and kaolinite and free caustic accompanied by the reprecipitation of the dissolved silica (as sodalite). The governing partial differential equations have been reduced to ordinary differential equations by the method of orthogonal collocation and solved using a standard variable step integration algorithm in terms of two parameters characteristic of the nature of the bauxite. The usefulness of the model is demonstrated by providing examples of cases where it has been applied to elucidate process performance under conditions that are envisaged for the future, or to gain an insight into reasons for certain anomalous trends in process stream quality in terms of silica concentrations.

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DEVELOPMENT OF AN IN-SITU CORROSION MONITORING SYSTEM FOR BAYER LIQUOR OPERATION UP TO 240°C: *Dr. Chris J. Newton*[1]; [1]Alcan International Limited, Kingston Research & Development Centre, Kingston, Ontario K7L 5L9 Canada

Since November 1994, two stand alone in-situ corrosion-monitoring systems have operated on the spent liquor heater train in the digestion section of a high temperature Bayer plant in Aughinish Alumina Ltd., Ireland. A novel design of test probes with samples of different heater tube materials are located at the inlet and outlet of the high temperature spent liquor heaters (170-230°C respectively). Their purpose is to gather on-line corrosion data to study the effects of process variables on different heater tube materials. Corrosion rate can be recorded both "on-line" via electrochemical measurements and by physical examination after exchange of the test samples, e.g. from the apparent loss of mass from each test heater tube section. This paper describes the principle of the monitoring system, what it is able to monitor and some typical electrochemical information. Some of the trigger events for certain corrosion processes have been identified by coupling the corrosion data to other on-line process monitoring sensors, e.g. temperature, flow, etc. In particular, evidence is presented of a peak in the corrosion rate variation following the first 10 to 50 hours after re-start following routine acid cleaning of a heater.

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THE IMPROVED LOW TEMPERATURE DIGESTION (ILTD) PROCESS: AN ECONOMIC AND ENVIRONMENTALLY SUSTAINABLE WAY OF PROCESSING GIBBSITIC BAUXITES: *Dr. Péter Siklosi*[1]; Gyorgy Banvolgyi[2]; [1]Siklosi & Co. Consulting, Engineering and Trading Ltd., ICF Kaiser-Aluterv Ltd., Semmelweis u. 5 Hungary; [2]ICF Kaiser-ALuterv Ltd, 1116 Budapest, Fehervari ut 144.

A short description of the Improved Low Temperature Digestion (ILTD) Process and its experimental background. Presentation of its process flow diagram and its process parameters compared with those of both the conventional low temperature Bayer digestion process and the so-called Sumitomo New Bayer Process. Economical comparison of the above processes. Utilisation potential of the bauxite residue (red mud) and the new by-product: Bayer-sodalite (desilication product).

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IMPROVEMENT OF THE PROCESS OF ALUMINA PRODUCTION AT THE LEASE HOLDERS' ASSOCIATION NIKOLAEV ALUMINA PLANT: *Mr. E. P. Kovalenko*[1]; [1]Nikolaev Alumina Plant, Nikolaev Ukraine

Emergence of Nikolaev Alumina Plant on the map illustrating the aluminium production industry was the result of the improvement of the distortion having taken place in this industry before that is a considerable gap between production of alumina and primary aluminium caused by putting into operation of cheap power supply sourced of big capacity and at the same time raw material (alumina) production base for primary aluminium being practically stagnated. Alumina production lagging behind the far advanced primary aluminium producers could be explained by the lack of deposits of the bauxite suitable for processing. Refineries using non-traditional raw materials such as nepheline, alunite for alumina production, which had been gradually put into operation, appeared to be high power consuming, complicated and costive. Nikolaev Alumina Plant was put into operation in 1980 and very soon reached its design output. At the present moment, Nikolaev Alumina Plant is steady operating due to thoroughly thought-out technology where import bauxite is processed by import equipment of enough high standard. But as the science is progressing and the raw material (bauxite) base does not stick to the constancy, as well as new requirements to the alumina properties are arising, all these resulted in a development of the Programme for improvement of the process and up-dating of the equipment. Implementation of this Programme will ensure solution of the above mentioned matters.

ATOMIC-LEVEL SIMULATION OF MATERIALS: NEW METHODS & NOVEL APPLICATIONS: Novel Methods, Empirical Potentials, and Phase Stability

Sponsored by: ASM International: Materials Science Critical Technology Sector, Computer Simulation Committee

Program Organizers: Jim Adams, Arizona State University, Dept. of Chemical Bio and Materials Dept., Tempe, AZ 85287-6006; Vaclav Vitek, University of Pennsylvania, Dept. of Materials Science & Eng., Philadelphia, PA 19104

Monday PM Room: 203
February 16, 1998 Location: Convention Center

Session Chair: John Moriarty, Lawrence Livermore National Laboratory, Livermore, CA 94551

2:00 PM

ACCELERATED MOLECULAR DYNAMICS OF INFREQUENT EVENTS: *Dr. Arthur F. Voter*[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Molecular dynamics (MD) is a powerful tool for investigating detailed atomic-scale behavior on the time scale of nanoseconds or less. For slower, infrequent-event processes, transition state theory (TST) can be employed, provided the nature of the transition states are known — i.e., if the relevant saddle points can be found. This is the basis of kinetic Monte Carlo methods. However, in many cases, the reactive events that will occur are not known in advance, or the transition states are very complicated. I will discuss a new method for treating this type of case for solid-state systems. A bias potential, constructed from the gradient and Hessian, raises the energy of the system without affecting the TST dividing surfaces. Performing MD on the biased potential leads to accelerated transitions from state to state. In this "hyperdynamics" approach, time is no longer an independent variable; the elapsed time is estimated as the simulation proceeds, converging on the correct time in

the long-time limit. Hyper-MD simulations of metallic surface diffusion on the microsecond time scale will be presented.

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THERMOELASTICITY: A MIXED ATOMISTIC AND CONTINUUM PERSPECTIVE: *Dr. Rob Phillips*[1]; [1]Brown University, Division of Engineering, Providence, RI 02912 USA

Molecular dynamics provides an explicit scheme for characterizing the mechanical and thermal response of large numbers of atoms. This talk will explore an alternative view in which a mixed atomistic and continuum description of the thermal and mechanical properties of a solid is given. Building on earlier work with the quasicontinuum method, the aim is to allow for the explicit consideration of thermal fluctuations where needed while treating regions far away from defects via continuum ideas. Specific reference will be made to the motion of dislocations.

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GREEN'S FUNCTION BOUNDARY CONDITIONS IN ATOMISTIC SIMULATIONS OF 3-D DISLOCATION CONFIGURATIONS: *Dr. Satish I. Rao*[1]; Dr. T.A. Parthasarathy[1]; Dr. C. Woodward[1]; [1]UES Inc., Dayton, OH 45432 USA

A method for dynamically relaxing the boundary forces that develop during atomistic simulations of dislocation configurations is presented. Green's function boundary relaxation method, originally introduced by Sinclair, Gehlen, Hoagland and Hirth (1978), is extended to treat three dimensional simulations. The boundary conditions for three dimensional (3-D) defect cells are evaluated using point force distributions, as opposed to line force distributions for 2-dimensional (2-D) cells. Both, fixed and periodic boundary conditions along the dislocation axis are treated. The method is general and can be incorporated into several interaction potential schemes. Examples of the method using empirical embedded atom method (EAM) potentials are presented for simulations of : a) the bowing of a realistic Frank-Read source in FCC Ni under various applied stresses, b) the energetics of tiny dislocation loops in FCC Ni and c) the structure of a periodic array of kinks in BCC Fe. This work was performed at the Materials Directorate, WL/MLLM under contract #F33615-91-C-5663. Computer time for all of us was provided in the form of a grant of HPC time on the Cray YMP and C90 supercomputers at the DoD CEWES shared resource center.

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CLASSICAL MOLECULAR DYNAMICS WITHOUT PAIR POTENTIALS: *Dr. Sokrates T Pantelides*[1]; Dr. J. S. McCarley[1]; [1]Vanderbilt University, Department of Physics and Astronomy, Nashville, TN 37064 USA

Potentials for classical molecular dynamics typically contain a pair interaction plus other three-body or many-body terms. The analytical forms are usually physically motivated but not derivable from first principles. We report the development of a new family of potentials that are directly derived from density functional theory, the rigorous quantum-mechanical theory for total energies. We achieve "classical" forms through systematic removal of the internal degrees of freedom. The result is an algebraic recipe for the total energy that depends only on the coordinates and the chemical identity of the atoms in the system. The formulation is equally applicable to monatomic or multicomponent systems. In the latter case, each atom carries a dynamical effective charge. We find that the method has full predictive power without any empirical adjustments for simple metals such as aluminum. For covalent semiconductors such as Si or GaAs and for transition metals, a fine-tuning empirical correction is needed. The fitting process is straightforward, robust, easily customizable and yields very accurate potentials. Unlike traditional potentials, the new potentials allow treatment of spin polarization and coupling with electromagnetic radiation. Initial applications to real problems will be discussed. This work was supported in part by ONR Grant N00014-96-1-1042.

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NEW EMBEDDED-ATOM TYPE POTENTIALS FOR Ni, Al AND NiAl: *Dr. Yuri Mishin*[1]; Dr. Michael J. Mehl[2]; Prof. Diana Farkas[1]; Dr. Dimitrios A. Papanonstantopoulos[2]; [1]Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061-0237; [2]Naval Research Laboratory, Complex Systems Theory Branch, Washington, DC 20375-5345

Embedded-atom type potentials are constructed for Ni, Al and NiAl by fitting to both experimental data and the results of first principles calculations. For the pure elements, the database includes the equilibrium values of the lattice parameter, cohesive energy, elastic constants, phonon dispersion curves and vacancy formation and migration energies. Rose's universal equation of state is also included with a certain weight. The potentials are also fit to the energy differences between the ground state structure (fcc) and a few metastable structures with several lattice parameters. The structural energies are obtained by FLAPW calculations. The potentials are tested by comparison with other experimental properties (such as the stacking fault and surface energies) and structural energies which were not included in the fitting database. Other tests, such as the behaviour under strong deformation (e.g. along the Bain and/or similar paths between the stable structures) in comparison with FLAPW calculations are also given to the potentials. For NiAl, the mixed pair interaction is fitted to the lattice parameter, cohesive energy, elastic constants and phonon dispersion curves of B2 NiAl, as well as to the energy differences with alternative structures obtained by FLAPW calculations. Attempts are made to include the angular dependence of atomic interaction in NiAl within the embedded-defect scheme. The potential for NiAl correctly predicts the asymmetric behaviour of constitutional point defects in off-stoichiometric compositions. It also predicts reasonably good values of the antiphase-boundary energies, surface energies and the energies of alternative metastable structures not included in the fitting database. Some atomistic simulation results intended to demonstrate the performance of the new potentials are presented.

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REAL-SPACE APPROACH TO CHEMICAL ORDER IN COMPLEX SYSTEMS: *Dr Patrice E. A. Turchi*[1]; Dr Didier Mayou[2]; [1]Lawrence Livermore National Laboratory, C.&M.S. Department (L-268), Livermore, CA 94551 USA; [2]CNRS, LEPES, Grenoble Cedex 9 F-38042 France

A tight-binding based approach for studying the properties of complex multi-component systems which exhibit both chemical order and topological disorder is presented. The scheme relies upon molecular dynamics and Monte Carlo simulations to solve for the short time-scale of vibrations and relaxations, and for the long time-scale of diffusion, respectively. The energetics which describes the diffusion part of the scheme is obtained within the ECM (embedded cluster method) by solving first the inhomogeneous CPA (coherent potential approximation) problem with a real-space recursion procedure, and by using an orbital peeling technique. Advantages and applications of this new scheme will be discussed together with some preliminary results. Work performed under the auspices of the U. S. Department of Energy by the Lawrence Livermore National Laboratory under contract No. W-7405-ENG-48. Partial support from NATO under contract No. CRG-941028 is gratefully acknowledged.

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LOCAL ENVIRONMENT EFFECTS IN THE VIBRATIONAL PROPERTIES OF DISORDERED ALLOYS: THE CASE OF Ni3Al: *Didier de Fontaine*[1]; Jeff Althoff[1]; Dane Morgan[1]; Mark Asta[2]; Andrew Quong[2]; Stephen Foiles[2]; [1]University of CA, Berkeley, Department of Materials Science and Mineral Engineering, Berkeley, California 94720-1760 USA; [2]Sandia National Laboratories, Computational Materials Science Department, Livermore, California 94551 USA

Recent experimental and theoretical work has suggested that vibrational entropy could play a more important role than previously believed in determining phase stability. The Embedded-Atom Method is used to study the effects of different local environments on the vibrational spectra and thermodynamic quantities for Ni_3Al. Projections of the density of states onto different local environments are performed and it is found that the contribution to the entropy from a

given atom is primarily determined by its local environment. Approximate methods of treating vibrational properties of disordered systems are discussed, in particular the use of average local environments (virtual crystal) and small supercells designed to mimic disordered local environments (special-quasirandom-structures). The role of substitutional defects in the ordered $L1_2$ structure are examined. Finally, the participation ratios of modes in both the $L1_2$ and disordered phases are calculated.

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ENERGETICS OF CATION VACANCY OCCUPATION IN GAMMA-ALUMINA: *Dr. Frederick H. Streitz*[1]; Dr. John W. Mintmire[2]; [1]Auburn University, Physics Department, Auburn, AL 36849-5311 USA; [2]Naval Research Laboratory, Code 6179, Washington, D.C. 20375 USA

The gamma phase of alumina (Al_2O_3) is a defected spinel structure which allows a well defined number of cation vacancies to occupy at random the available tetrahedral and octahedral sites in the crystal. The nature of this occupation is still under debate — it is unclear whether the vacancies randomly occupy all of the available cation sites, or prefer exclusively either the tetrahedral or octahedral sites. We have investigated the energetics of vacancy occupation in this crystal using the ES+ potentials, which were specifically developed to model the aluminum oxides. We find that although the octahedral site is preferred by 3.1 kcal/mol over the tetrahedral site, a small percentage of tetrahedral vacancies (about 25%) can be incorporated into the structure at very little cost in energy. This work supported by the Office of Naval Research

AUTOMOTIVE ALLOYS II: Session II - Developmental Studies

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Subodh Das, ARCO Aluminum Company, PO Box 32860, Louisville, KY 40232

Monday PM Room: Fiesta B
February 16, 1998 Location: Convention Center

Session Chair: Carl Seidler, ARCO Aluminum Co., Inc., Louisville, KY 40232

2:00 PM

SOLUTE SEGREGATION AND β-PHASE PRECIPITATION AT INTERNAL INTERFACES IN AA5083: *J. S. Vetrano*[1]; R. H. Jones[1]; [1]Pacific Northwest National Laboratory, Richland, WA 99352

The susceptibility of Al-Mg alloys (>3.5 wt% Mg) to stress corrosion cracking (SCC) has limited their use for load bearing structures in automotive applications. Susceptibility to SCC has generally been attributed to grain boundary precipitation of the β-phase (Al_3Mg_2 following exposure at moderate (<200°C) temperatures. High resolution microstuctural evaluations of AA5083 (4.5wt%Mg) following a series of heat treatments have been performed to better understand the nature of Mg segregation and β-phase precipitation at internal interfaces. Compositional measurements by transmission electron microscopy and a scanning auger microprobe revealed that heat treatments as short as one hour at 175° were sufficient to cause significant Mg segregation to grain boundaries and even higher amounts to triple points. Longer heat treatments resulted in β-phase precipitation at internal interfaces but even after 100 hours the phase was not continuous along grain boundaries. Previously reported results have shown that the material is highly susceptible to SCC after 100 hours at 175°C. This indicates that it is possible for Al-Mg alloys to under SCC even if there is not a continuous film of β-phase along the grain boundaries. Methods for mitigation and possible mechanisms for SCC will be discussed. Work supported by the Materials Division, Offices of Basic Energy Sciences, U.S.Department of Energy under Contract DE-AC06-76RLO 1830.

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TEXTURE HETEROGENEITY OF 6010 ALUMINUM ALLOY SHEET WITH AND WITHOUT ANNEALING AFTER HOT ROLLING: Y. Liu[1]; S. Ding[1]; *J. G. Morris*[1]; [1]University of Kentucky, Light Metals Research Laboratories, Department of Chemical and Materials Engineering, Lexington, KY 40506 USA

Aluminum alloys present strong mechanical anisotropy due to preferred orientation after hot rolling and recrystallization. In the present work, textures of hot rolled 6010 aluminum alloy sheets and those annealed at 900°F for 2 hours were determined by pole figure measurement and orientation distribution function (ODF) methods. For each sample, pole figure measurements were performed on different sections parallel to the rolling plane in order to detect the texture heterogeneity in the normal direction of the rolling plane. The typical cold rolling textures in FCC metals indicated by Cu(C)-{112}<111>, Brass (B)-{110}<112> and S-{123}<634> components were observed in the surface layer of the hot rolled sheets. The C, B and S components present similar ODF intensities of about 10. In the center layer, very strong B and S components were developed. The ODF intensities of the B and S components reached a maximum value of 33 and are over 2 times higher than that of the C component. For the annealed samples, the cube {001}<100> and the rotated cube{001}<110> components are predominant in the surface layer. Their ODF intensities increase to maximum values of 15 for the cube and 23 for the rotated cube in the position of 1/8 sheet thickness from the surface, and decrease to about 5 in the center layer. The Goss {110}<001> component becomes dominant in the center layer with an ODF intensity of about 14. The texture transition mechanisms and the effect of texture heterogeneity on properties of the alloy are discussed. It can be concluded that: (1) the development of the strong Goss component in the center layer during annealing is attributed to the present of high intensity B and S components after hot rolling; (2) great texture heterogeneity exists in samples with and without annealing after hot rolling; (3) annealing can effectively remove cold rolling type, C, B and S components but can not eliminate the texture heterogeneity; to a significant degree the cold rolling texture is dependent on the initial texture of the hot band material.

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COOLING EFFECTS ON AGING IN A CAST ALUMINUM ALLOY: *Prof. Rafael Colas*[1]; Ms. Ana Isabel Garcia-Celis[1]; Dr. Eulogio Velasco[2]; Dr. Salvador Valtierra[2]; Dr. Juan Francisco Mojica[2]; [1]FIME, UANL, San Nicolas, N.L. 66451 Mexico; [2]Corporativo Nemak, S.A. de C.V., Garza Garcia, N.L. 66221 Mexico

The effect that cooling rate after solubilization exerts on the aging behavior of a cast aluminum alloy was studied. Samples of the alloy (A319) were prepared with and without addition of modifiers and grain refiners and poured in a steel mould. Bars from these materials were machined and drilled in order to insert a series of type K thermocouples (used to register thermal evolution during heating and cooling). Solubilization (four hours at 480°C) was carried out in a box furnace and the bars were cooled down to room temperature by placing one of their ends in a shallow tank of water. Samples for aging (conducted at 130, 180 and 230°C) were cut from the bars at positions close to the thermocouples. It was found that hardness of the unaged material increased as the cooling rate was reduced, but this behavior reversed (harder material was found in the portions cooled at the faster rates) after aging. These features can be explained in terms of incipient precipitation taking place while cooling.

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DEVELOPMENT OF A MODELING METHODOLOGY FOR THIN WALL ALUMINUM GREEN SAND CASTINGS FOR AUTOMOTIVE APPLICATIONS: *V. K. Suri*[1]; J. Righi[1]; J. R. Fields[1]; [1]Alcoa Technical Center, Alcoa Center, PA 15069-0001

Making 2mm to 3mm thick aluminum alloy structural castings in green sand offers a number of challenges especially in high production

rate automated casting systems. Mold filling in these castings is severely affected by rapid heat removal rates across the flow front aided by moisture evaporation in sand. In addition, alloy and sand characteristics affect casting quality significantly. The goal of the current work is to develop a process model that captures the transport phenomena in green sand casting process for thin wall castings, and utilize it to map process limits and design feeding systems. This paper describes experimental and theoretical work leading to the development of a modeling methodology. The semi-empirical approach adopted in the current work eliminates a large percentage of the thermal field calculation in the mold. Illustrative examples are presented to demonstrate the effectiveness of the model in analyzing feeding design, alloy response and resulting product quality.

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DEVELOPMENT OF MICROSTRUCTURE AND PROPERTIES IN 6XXX SERIES AUTOMOTIVE SHEET ALLOYS DURING PROCESSING THROUGH AN INTEGRATED CONTINUOUS HEAT TREATMENT AND FINISHING LINE: *R. G. Hamerton*[1]; N. C. Parson[1]; S. A. Court[1]; [1]Alcan International Limited, Oxon OX167SP England

A critical stage of the production of automotive sheet for skin panel applications using 6xxx series aluminium alloys such as AA6016A is the solution heat treatment which takes place at final gauge after hot and cold rolling. At Alcan's new facility in Nachterstedt, Germany, this takes place as part of a continuous process in which the aluminium strip can be sequentially solution heat treated, quenched, cleaned, pre-treated and coated. Immediately after quenching the sheet microstructure is in a particularly unstable condition. Because of this, subsequent thermal exposure and mechanical effects occurring during the process steps which immediately follow can have a significant impact upon the final properties of the sheet. This is especially true of the response to heat treatment during automotive manufacture. This paper will report on experimental methods devised to simulate the thermo-mechanical influences acting on the strip during processing through the integrated line. Such methods have been used to isolate and understand the effects of the various process steps on the development of microstructure and properties. Some of these findings will be presented, including, for example, the importance of solution heat treatment conditions and quench rate, as well as the influence of subsequent re-heating during curing of the coating. The significance of alloy composition in relation to this type of processing will also feature in the discussion.

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WARM FORMING BEHAVIOR OF AUTOMOTIVE ALUMINUM ALLOY: *Mr. Zhen Guo*[1]; Dr. Abhijit Dutta[1]; Dr. Amit K. Ghosh[1]; [1]The University of Michigan, Dept. of Materials Science and Engineering, Ann Arbor, Michigan 48109-2136

The excellent combination of low density and high strength provide aluminum alloys with a strong potential for substituting steel as the major material in automotive sheet stamping and forging. Due to its lower formability, however, efforts are in place to examine ways to improve formability of aluminum alloys. The intent of this research was to determine whether warm temperature forming could improve the formability of existing conventional alloy and some kinds of new aluminum alloys. Uniaxial tensile behavior of selected alloys under different thermomechanical processings were determined in the warm temperature range from room temperature to 350°C over a strain rate range of 10^(-2)-10^(0)/sec. Results were discussed based on dynamic microstructure changes during deformation. Some phenomenological and physical models were established to describe the uniform deformation.

CARBON TECHNOLOGY: Cathodes

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Ron Barclay, Alumax, PO Box 1000, Goose Creek, SC 29445

Monday PM — Room: Fiesta D
February 16, 1998 — Location: Convention Center

Session Chair: Larry Boxall, Fluor Daniel, Inc., Greenville, SC 29607-2762

2:00 PM

ALUMINIUM PECHINEY EXPERIENCE WITH GRAPHITIZED CATHODE BLOCKS: *D. Lombard*[1]; T. Beheregaray[1]; B. Feve[1]; J. M. Jolas[1]; [1]Aluminium Pechiney, Cedex France

For a few years, ALUMINIUM PECHINEY has been testing graphitized cathode blocks on different types of pots in various smelters. The theoretical advantages in using graphitized cathode blocks are presented, particularly the effect on potline amperage. Based on different industrial trials, a comparison is drawn with pots using conventional blocks, in terms of cathodic voltage drop, operational results and cathode life duration. The performance of the different graphitized block qualities available on the market and the erosion phenomenon are also discussed. Finally, the economical interest of graphitized blocks is reviewed.

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INCREASED SODIUM EXPANSION IN CRYOLITE-BASED ALUMINA SLURRIES: *Harald A. Oye*[1]; Xian-an Liao[1]; [1]Norwegian University of Science and Technology, Institute of Inorganic Chemistry, Trondheim N-7034 Norway

Sodium expansion of carbon cathode materials was studied in cryolitic melt-alumina slurries. In the acidic cryolitic melt-alumina slurry, a 25% to 50% increase in expansion was observed compared to the expansion of the corresponding slurry-free bath. This is attributed to overvoltage increase for aluminium deposition. The expansion increased 150% to 600% in the basic cryolitic melt-alumina slurry compared to the expansion of the corresponding slurry-free bath. This large expansion increase is attributed to the formation of β-alumina which is a fast ion Na^+ conductor. Factors influencing β-alumina layer formation on the carbon surface were found to have large effect on the expansion. The present finding constitutes a strong argument against basic start-up and muck-up of the cell bottom during this period.

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PHYSICAL AND CHEMICAL WEAR OF CARBON CATHODE MATERIALS: *Xian-an Liao*[1]; Harald A. Oye[1]; [1]Norwegian University of Science and Technology, Institute of Inorganic Chemistry, Trondheim N-7034 Norway

The physical and chemical wear of carbon cathode materials for aluminium electrolysis was studied in mixtures of cryolitic melts, alumina and aluminium at 960-1015°C. The very low wear in aluminium is attributed to poor contact between the metal and the carbon materials due to nonwetting and very low carbide solubility. The much larger wear in cryolitic melt-aluminium system is attributed to the improved wetting and the larger carbide solubility. Under cathodic polarization the samples show even larger wear. Addition of alumina suppresses the wear through physical barrier or poor surface mixing. Different carbon cathode materials have approximately the same resistance to the chemical corrosion due to Al_3C_4 formation. The physical wear in cryolitic melts is parallel to room temperature experiments using alumina slurries in polytungstate solution. The room temperature experiments are recommended for evaluation and ranking of cathode materials.

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PITCH FREE CARBON MATERIALS FOR USE IN ELECTROWINING: *Professor J. A. Sekhar*[1]; Mr. J. Li[1]; Dr. J. Yan[1]; Dr. J. Liu[1]; Professor V. de Nora[1]; [1]University of Cincinnati, International Center for Micropyretics, Department of Materials Science and Engineering, Cincinnati, Ohio 45221 USA

In the past few years, a variety of pitch free carbon materials have been developed for use as anodes, cathodes, sidewalls and other cell components including pastes. A review of these materials and a description of processing, composition, and properties for each application will be discussed. A listing of the presently available best compositions will be presented. An estimate of the total environmental impact of these pitch free carbon materials will also be presented. A discussion of the significantly higher oxidation resistance and sodium resistance of the pitch free materials when compared with conventional side walls and cathode materials will also presented.

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POTLINE-SCALE APPLICATION OF TiB_2 COATING IN HEFEL ALUMINIUM & CARBON PLANT: *Xian-An Liao*[1]; Prof. Yong-Zhong Huang[1]; Ye-Xiang Liu[1]; Jin-Ru Chan[2]; Shui-Hua Zhong[2]; [1]Central South University of Technology, Changsha, Hunan 410083 P R China; [2]Hefel Aluminium & Carbon Plant, Hefel, Anhul

A potline-scale test of TiB2 coating has been under way in Hefel Aluminium & Carbon Plant since the last quarter of 1995. More than 2% higher current efficiency and more than 300 kwh per ton of aluminium less power consumption are observed in the test cells than in the reference cells. The results are attributed to the easier operation of the test cells due to cleaner carbon cathode surface and more even current distribution. So far the TiB2 coating technology has been tested in 12 aluminium smelters in China. Low cost, simple preparation procedure and improved cell performance are the main reasons of the popularity of this new technology.

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SOME EXPERIMENTS IN CATHODE CARBON: *S. Wilkening*[1]; [1]VAW Aluminium-Technologie GmbH, 53114 Bonn Germany

In laboratory and industrial-scale investigations, answers were sought to the following questions: To what extent can cathode blocks be improved through non-carbonaceous additives? How does the impregnation rate of cathode carbon depend on the composition of the electrolytic melt? What happens to the cathode block during an anode effect, particularly during pot start-up? How can the penetration of bath constituents through the carbon bottom be stopped or retarded? Can different concepts and material combinations minimize spent pot lining? Our attempts and results will be presented and some conclusive answers will be given to the above-listed problems.

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DEVELOPMENT IN THE FIELD OF GRAPHITE CATHODES: *Dr. Karl-Theodor Piel*[1]; [1]VAW CARBON GmbH, Grevenbroich 41515 Germany

Graphite cathode blocks from our company are supplied to aluminium smelters since 1988. During the last years more and more amorphous cathodes were substituted by graphite. Due to this fact an investigation program was started in our laboratory. The target of this program was to test the influence of various raw materials. Tested were two petrol cokes, one pitch coke and one isotropic coke. The influence on physical properties of the finished product such as: *Density *Mechanical strength *Rapoport expansion *Thermal expansion *Thermal shock resistance *Young's modulus* Mechanical abrasion were tested and compared with our standard quality grade.

CAST SHOP TECHNOLOGY: Session IB - Molten Metal Processing

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Diran Apelian, Worcester Polytechnic Inst., 100 Institute Rd., Worcester, MA 01609-2280

Monday PM Room: River Room B
February 16, 1998 Location: Convention Center

Session Chair: Raj Mutharasan, Drexel University, Philadelphia, PA 19104

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REFINING OF POTROOM METAL USING THE HYDRO RAM CRUCIBLE FLUXING SYSTEM: *Bjorn Rasch*[1]; Erling Myrbostad[2]; Kjell Hafsaas[1]; [1]Hydro Aluminium R & D Materials Technology; [2]Hycast A/S

The Hydro RAM process involves the introduction of aluminium fluoride through a patented spinning rotor head into the aluminum melt. Extensive full scale industrial experiments have been performed at two plants in Norway with metal amounts in the crucibles of approximately 1,8 and 3,6 metric tonnes. In addition to a rapid reduction in alkaline content (Na, Ca and Li) in the metal during this process, a substantial decrease in inclusion content is observed.

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PREDICTION OF AGGLOMERATION AND BREAK-UP OF INCLUSIONS DURING METAL REFINING: *Dr. Stein Tore Johansen*[1]; Dr. Shoji Taniguchi[1]; [1]SINTEF, Materials Technology, N-7034 Trondheim Norway

Agglomeration of SiC and other particles are studied in a water-based system. From theoretical considerations expressions for Hamaker constants are extracted from the data and further translated to the metallurgical system. In addition a theoretical model which can describe combined agglomeration and break-up is derived. The model is used to reproduce data from the water system and further used to extrapolate into the metallurgical system. The results are applied in a discussion about how stirring affects the different particle sizes during metal treatment.

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A CONTRIBUTION TO INCLUSION MEASUREMENT AFTER IN-LINE DEGASSERS WITH PODFA AND LIMCA: W. Schneider[1]; *H. P. Krug*[1]; [1]VAW Aluminium AG, Research & Development, Bonn D-53117 Germany

The reduction of particulate impurities by in-line degassing units is claimed by all major suppliers of this equipment. The mechanism is suggested to be a flotation like process. Proofs to demonstrate the validity of this beneficial effect have up to now been tried by PoDFA-evaluations. A statistical survey over a period of four years on different in-line degassing units reveals as well acceptable reductions as well as failures. In case of using LiMCA-measurements downstream of degassers to check the assumed removal of inclusions the detrimental disturbance by ultrafine gas bubbles on the test procedure must be avoided. VAW aluminium AG R&D has developed a method to perform the acquisition process of the LiMCA-Instrument in pressure mode instead of vacuum mode. It will be proved that this method allows a trouble-free acquisition after degassers alone and in combination with filtration systems - within certain limits - and that the obtained values are close to the real inclusion rates. The results of these LiMCA-measurements show that the selected method does not influence the normal evaluation within the range of the usual precision and that the ultrafine bubbles created and expelled by the degassing units range in their majority between 10 and 30 microns. The evaluation shows further that the reduction of separate in-line degassers with respect to inclusions in

AA10xx, 11xx and 12xx-alloys made mainly from potroom metal is lower than that of most filters.

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INCLUSION REMOVAL DURING CHLORINE FLUXING OF ALUMINIUM ALLOYS: *Raja R. Roy*[1]; Torstein A. Utigard[1]; Claude Dupius[2]; [1]University of Toronto, Metallurgy and Materials Science, Toronto, Ontario M5S 2E4 Canada; [2]Alcan International Limited, Arvida Research and Development Center, Jonquiere, Quebec Canada

Although chlorine fluxing is widely used in the aluminium industry to improve molten metal cleanliness, there is a very limited understanding of the mechanisms governing inclusion removal. This paper describes the fundamental approach used to evaluate the role of the key parameters (nitrogen and chlorine flowrates, stirring energy and time) affecting inclusion separation. The use of diluted MMCs combined with image analysis has proven to be a good model to investigage inclusion removal mechanisms. The presence of a minimum concentration of chlorine in the fluxing gas is essential to achieve positive inclusion removal. The mechanical stirring energy can successfully be used to reduce the chemical energy requirements to maintain inclusion removal rates.

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ELECTROMAGNETIC CONTROL OF HIGH TEMPERATURE LIQUID METAL JETS: *D. J. Short*[1]; P.A. Davidson[1]; [1]Cambridge University, Engineering Dept., Cambridge, England, UK

Abstract not available.

3:40 PM
MEASUREMENT OF CELL AND WINDOW SIZE IN CERAMIC FOAM FILTER MANUFACTURE: *S. Ray*[1]; N. J. Keegan[1]; [1]Foseco International, Staffordshire B78 3XQ England

An image analysis system for measuring polyurethane foam and ceramic foam filter cell and window size has been developed. A program of work was undertaken to gather production data for nominally 20 to 80 ppi filters. This data is used to show how careful process control is fundamental to the successful production of reliable fine pore filters. The paper discusses how this information has been used to characterize the filter structure and develop specifications for cell and pore size. These are compared to the traditional ppi and air permeability techniques. The paper concludes by discussing and comparing practical casting experience and results with the measurements made using image analysis.

4:00 PM
LIMCA II EVALUATION OF THE PERFORMANCE CHARACTERISTICS OF SINGLE ELEMENT AND STAGED CERAMIC FOAM FILTRATION: *Dawid D. Smith*[1]; Leonard S. Aubrey[1]; [1]Selee Corporation, Hendersonville, NC 28739 USA

Numerous filter performance models are published in the literature, based on trajectory flow modeling, intercept-collector theory, probabilistic modeling etc., where capture efficiency is predicted based on the characteristics of the filter media, metal flow rate and the characteristics of the inclusion system. Although there is a wealth of experimental modeling and laboratory data to support these modeling results, there is very limited definitive information which was obtained under industrial conditions. Therefore, Selee Corporation and VAW evaluated under production conditions in the VAW-Rheinwerek Cast House the effect of filter pore size flow rate and filter thickness on inclusion capture efficiency using both LiMCA II and pressure filtration tests (PoDFA and LAIS). Various combinations of single and staged filters from 30 to 70 ppi were evaluated under variable flow rates (0.55 to 3.16 lb/inch^2/min). As predicted by filter performance models, pore size turned out to be a major variable in determining metal quality.

4:20 PM
PERFORMANCE OF A STAGED FILTRATION SYSTEM INSTALLED AT NORANDAL USA, INC. TO FILTER CONTINUOUS TWIN ROLL CAST CONVERTER STOCK: *Dan Barbis*[1]; Dawid D. Smith[2]; [1]Norandal USA, Inc., Salisbury, NC 28145 USA; [2]SELEE Corporation, Henderson, NC 29739

Continuous twin roll casting of 11XX series aluminum for thin foil converter stock applications requires filtration systems capable of delivering metal with excellent microcleanliness over extended campaigns. In an ongoing effort to improve their process, Norandal USA, Inc. embarked on a joint venture with Selee Corporation to install and test a staged filtration system at Norandal's Salisbury plant. Metal cleanliness at Salisbury was characterized via PoDFA, LAIS, and Alscan at the beginning and end of production runs. Rolling performance and foil quality compared well against rigid media systems. The overall user friendliness and small footprint combined with a comparable cost of operation made the staged filtration system ideal for Salisbury's requirements.

DEFECTS IN CRYSTALS: A SYMPOSIUM HONORING THE CONTRIBUTIONS OF JOHN P. HIRTH: Crystal Plasticity

Sponsored by: Jt. Electronic, Magnetic & Photonic Materials Division/Structural Materials Division , Chemistry & Physics of Materials Committee, Physical Metallurgy Committee, Shaping and Forming Committee

Program Organizers: Craig S. Hartley, National Science Foundation, 4201 Wilson Blvd Room 545, Arlington, VA; Brent L. Adams, Carnegie Mellon University, Dept. of Materials Sci & Eng., Pittsburgh, PA 15213-3890; Richard G. Hoagland, Washington State University, Dept. of Materials Sci & Eng., Pullman, WA 99164-2920; Rob Wagoner, The Ohio State University, Dept. of Materials Sci & Eng., Columbus, OH 43210

Monday PM Room: 102
February 16, 1998 Location: Convention Center

Session Chair: TBA

2:00 PM INVITED
JPH: THE REST OF THE STORY: *Robert A. Rapp*[1]; [1]The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

John Hirth has contributed importantly to Defects in Crystals, the subject of this symposium, but he has also participated in advances and understanding in many other areas of research. For example, his research helped to clarify the effect of surface structure and surface charge on the sublimation of ionic crystals. Diffusion-controlled reactions such as the growth of multi-layered scales, the growth of intermediate phases in alloy interdiffusion, displacement reactions in the solid state, phase suppression in interdiffusion, and diffusion barriers have also gained his attention. Most recently, he has helped to clarify the behavior of defects "between solids", namely the role of interphase interfacial line defects in vacancy creation and annihilation in scaling and interdiffusion reactions.

2:30 PM INVITED
AN EXPLICIT SOLUTE DRAG LAW AS AN UPPER BOUND TO CREEP STRENGTH: *U. Fred Kocks*[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

When solute drag controls dislocation mobility, one expects essentially straight edge dislocations to carry the deformation. Their interaction leads to a steady-state strain rate proportional to the third power of the stress. The details of the solute/dislocation interaction determine the activation energy and pre-factors. When the dislocations are saturated with solutes, there should be no concentration dependence and the simple Dorn form of the creep law should hold. Indeed, when independently measured activation energies are used, as well as temperature dependent elastic moduli, a third-power dependence, with the proper pre-exponential factor, has been observed. It is

postulated that this law provides an upper bound to the strength of solution hardened alloys at high temperature-or, conversely, an upper bound to the service temperature for a given strength.

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EXPLOSIVE STRAIN RATE EFFECTS ON THE DISLOCATION STRUCTURE OF TANTALUM: *F. Robert Frasier*[1]; Michael Kaufman[1]; [1]Northwest Technical Industries, Inc., Sequim, WA 98382 USA

At conventional strain rates, tantalum is generally considered to be almost an elastic-perfectly plastic material. However, when subjected to explosive strain rates, it has been found that tantalum can exhibit significant work hardening. A series of experiments has been performed to determine if explosive straining creates immobile or tangled dislocation structures. Both electron beam melted and powder processed samples were investigated to determine the effect of interstitial impurity concentration on the dynamic dislocation structures. Dislocation structures were examined using transmission electron microscopy (TEM). Specimens were taken from annealed, explosively strained and explosively strained followed by conventionally strained material. The results of the TEM evaluation will be correlated with observed changes in mechanical properties, and implications for the mechanisms of dislocation behavior in tantalum will be discussed.

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DISLOCATION LOCKING AND STRENGTH ENHANCEMENT IN A SOLID-SOLUTION NiAl-0.3Hf SINGLE CRYSTAL ALLOY: *Dr. Anita Garg*[1]; Dr. Ronald D. Noebe[1]; [1]NASA Lewis Research Center, Materials Division, Cleveland, OH 44135 USA

Mechanical testing followed by detailed transmission electron microscopy of deformed samples has been performed in a [001]-oriented solid solution strengthened NiAl-0.3Hf single crystal alloy in the temperature range 300-1400 K. Yield stress-temperature data indicates that this alloy is slightly weaker than a precipitate strengthened NiAl-0.5Hf alloy at temperatures up to 800 K, but beyond this temperature strength of the 0.3Hf alloy approaches or exceeds that of the 0.5Hf alloy. Dislocation contrast analysis at 1100 K indicates that the strength enhancement in the solid solution NiAl-0.3Hf alloy is due to the formation of [110] and [-110] sessile locks which are likely promoted by a high concentration of vacancies that exist in the alloy at these intermediate temperatures. Precipitate-dislocation interaction prevents the formation of such locks in the NiAl-0.5Hf alloy. It is proposed that these sessile locks are formed by reactions of the <101> glide dislocations that are responsible for deforming the alloy and are stabilized by Hf segregation to the dislocation cores.

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PRECIPITATE-INDUCED PLASTICITY IN BCC METALS: *Dr. Ronald Gibala*[1]; Mr. Raul Fournier[1]; [1]University of Michigan, Materials Science & Engineering, Ann Arbor, MI 48109-2136 USA

Under applied or thermal stresses, precipitate-matrix interfaces can act as an efficient source of dislocations in metals. If the generated dislocations are glissile, enhanced plasticity can result. This effect can be particularly large in materials for which plastic flow is dislocation-mobility and/or dislocation-density limited, such as body-centered cubic metals deformed at relatively low homologous temperatures. In the present paper, the mechanical behavior of high-purity niobium-hydrogen alloys deformed at 77 K is used to demonstrate such precipitate-induced plasticity enhancement. Examination of the dislocation substructures of the deformed Nb-H alloys discloses that a model by Hirth and co-workers, in which prismatic dislocations generated by the large matrix-precipitate misfit act as sources for glissile dislocations, accounts well for observed behavior.

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COMPUTER SIMULATION OF A [110] EDGE DISLOCATION INTERSECTING A Σ 11 <101>{131} GRAIN BOUNDARY IN ALUMINUM: *Howard L. Heinisch*[1]; Richard G. Hoagland[1]; Richard J. Kurtz[1]; John P. Hirth[1]; [1]Pacific Northwest National Laboratory, Richland, WA 99352 USA

There have been numerous, atomistic studies of dislocations and grain boundaries (GBs) in metals, but relatively few efforts have focused on the interaction of matrix dislocations with GBs. In general, atomic-scale investigations of dislocation-GB interactions have suffered from the restriction of periodic boundary conditions along the direction of the dislocation line. Typical problems include simulation of extrinsic GB dislocations or interactions of dislocations lying parallel to a GB. In the present work we examine an edge dislocation intersecting a high-angle GB out of the boundary plane, a problem that requires 3D geometry. As a first problem we assumed that the dislocation line intersects the GB, existing in both grains simultaneously, In our model no atomic arrangement is assumed for the GB dislocations required by such a situation. An embedded atom method potential for aluminum is used to describe the atomic interactions. The computational cell is cylindrical, with a Σ11 <101>{131} GB at its center plane and normal to the cylinder axis. 1/2[110] edge dislocations (split into partials) are introduced about ten degrees off normal in each grain by imposing the appropriate anisotropic elastic displacement field. The relaxed atom configuration is determined by energy minimization with flexible border conditions. Several combinations of relative orientations of the dislocations in the two grains are examined. A dominant feature is that the separation of the partials changes significantly near the intersections with the GB.

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THE EFFECTS OF DISLOCATION INTERACTIONS ON THE MECHANICAL BEHAVIOR OF AN IRON-BASED SUPERALLOY: *Neville R. Moody*[1]; Michael I. Baskes[1]; Darcy A. Hughes[1]; [1]Sandia National Laboratories, Livermore, CA 94551-0969 USA

Coplanar slip is the dominant mode of deformation in most iron-based superalloys. However, the effects of dislocation interactions on coplanar slip band formation and properties are not well-defined, appearing to vary with test technique and sample geometry. As a consequence, large sample and small volume testing were used to study dislocation behavior in the superalloy IN903. Strain rate cycling and stress relaxation were used on relatively thick tensile samples where interior slip processes dominate behavior. Nanoindentation was used where surface effects control the onset of dislocation motion. Behavior differed markedly, with properties exhibiting a high strain rate sensitivity at the onset of deformation to being almost strain rate insensitive near failure. These results will be presented and used to show how dislocation and microstructure interactions control deformation in this class of alloys. This work supported by U.S. DOE Contract DE-AC04-94AL85000.

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DISLOCATION PROCESSES IN HARD-ORIENTED NiAl-BASED INTERMETALLIC COMPOUNDS: *Rajagopalan Srinivasan*[1]; M. F. Savage[1]; M. S. Daw[1]; R. D. Noebe[1]; Michael J. Mills[1]; [1]The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

The mechanical properties of NiAl and its alloys are dictated by a number of complex, dislocation-level processes. This paper concentrates on the dislocation behavior that influences the observed yield strength vs. temperature behavior in both stoichiomctric and off-stoichiometric, hard-oriented NiAl. The dramatic influence of limited additions of Hf (0.3 at.%) to NiAl on the observed dislocation microstructure (beyond the slip transition temperature) will also be discussed. Specifically, the mechanism of decomposition of a<111> dislocations leading to the slip transition (around 600 K for stoichiometric NiAl), and subsequent dislocation activity beyond the slip transition temperature will be revisited. In addition, the core structures of a<101> dislocations, and the influence of stoichiometry on the observed core structures will be discussed. This research has been supported by the U.S. Department of Energy under Contract No. DE-FG02-96ER45550 (for RS, MFS and MJM) and by The National Science Foundation under grant no. 95-10259 (for MSD).

GENERAL ABSTRACTS: II - Innovations in Aluminum II

Sponsored by: Light Metals Division, Aluminum Committee, U.S. Department of Energy

Monday PM Room: Patio A
February 16, 1998 Location: Convention Center

Session Chair: Toni Grobstein Marechaux, U.S. Department of Energy, Office of Industrial Technologies, Washington, DC 20585

2:00 PM
OVERVIEW OF DOE'S PROGRAMS IN ALUMINUM METAL CASTING FOR IMPROVED ENERGY EFFICIENCY: *Ms. Toni Grobstein Marechaux*[1]; [1]U.S. Department of Energy, Washington, DC 20585

The Department of Energy funds a range of projects in aluminum metalcasting for applications in industries and transportation. The ultimate goal of these programs is improved understanding leading to more valuable materials applications and better energy efficiency. Programmatic coordination and synergism in DOE's Office of Energy Efficiency and Renewable Energy will be discussed.

2:30 PM
COMPUTER AIDED ENGINEERING SOFTWARE FOR THE SEMI SOLID METAL MANUFACTURING PROCESS: *Mr. Gerald Backer*[1]; [1]Flow Logic, Southfield, MI 48075 USA

This project is aimed at providing an improved capability for process simulation and analysis for the rapidly growing manufacturing method of Semi-Solid Metal (SSM) forming. For the high volume production of aluminum parts, die casting has historically been the method of choice due to its rapid cycle times and relatively low cost. SSM is becoming the preferred manufacturing method when high mechanical properties and improved structural integrity are important. A computer simulation model, similar to that employed for die-casting, is being developed for an SSM mold-filling simulation package.

3:00 PM
ADVANCED UNIFORM DROPLET SPRAYS FOR ALUMINUM PRODUCTION AND PROCESSING: *Dr. Tae-Woo Lee*[1]; Dr. Ampere A. Tseng[1]; [1]Arizona State University, Tempe, AZ 85287-6106 USA

The use of uniform droplet sprays has emerged as an advanced manufacturing technology for metal production and powder metallurgy. A variable diameter jet and a controllable planar jet will be developed and used for applications to rapid production and processing of aluminum with precise control of the metal internal microstructure. The associated spray dynamics and deposition characteristics which are critical in the understanding and successful implementation of these new designs will be investigated.

3:30 PM Break

4:00 PM
MANUFACTURING OF MACRO-COMPOSITE ALUMINUM BRAKE ROTORS FOR REDUCING WEIGHT AND IMPROVING PERFORMANCE OF AUTOMOBILES: *Dr. Xiaodi Huang*[1]; [1]Institute of Materials Processing, Michigan Technological University, Houghton, MI 49931-1295 USA

This concept is based on building an aluminum alloy brake rotor with aluminum composite cladding (silicon carbide and graphite particles) on the friction surfaces. This engineering design can be cost-effectively produced with a newly developed commercially viable CastCon process. CastCon is a manufacturing approach which combines casting, forging, P/M, bonding and coating in one process to produce high performance components. The resulting rotors will be lightweight, strong, and ductile, and will have superior wear resistance and excellent braking efficiency.

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HIGH PRESSURE PULSE BONDING OF ALUMINUM: *Dr. Jack Kolle*[1]; [1]Tempress Technologies, Inc., Kent, WA 98032 USA

This concept uses high-pressure jet pulses to join aluminum components. The process could be deployed using robot manipulators and would be well suited to high volume and flexible manufacturing of aluminum structures.

5:00 PM
DEVELOPMENT OF ALUMINUM RECLAIMER FOR SMALL FOUNDRY APPLICATIONS: *Mr. Dan Groteke*[1]; [1]Q.C. Designs, Inc., St. Joseph, MI 49085 USA

Q.C. Designs is developing a low-cost aluminum "reclaimer," which would be ideally suited to support improved melting recovery in small to medium sized foundries or die casters. The concept has been demonstrated to permit recovery at the generating furnace of between 30% and 80% of the metal units contained in the dross, depending upon the condition and composition of the dross. Thus it is also expected to significantly reduce the volume and toxicity of the residual product.

GENERAL ABSTRACTS: IX - Recycling and Primary Production

Sponsored by: TMS

Monday PM Room: Patio B
February 16, 1998 Location: Convention Center

Session Chair: Courtney Young, Montana Tech, Dept. of Metallurgical Eng., Butte, Montana 59701-8997; Viola Acoff, University of Alabama,

2:00 PM
EFFICIENT CLEANING OF DUST LADEN GASES WITH WET TUBULAR ELECTROSTATIC PRECIPITATORS: *C. Wadenpohl*[1]; W. Peukert[1]; [1]Hosokawa MikroPul GmbH, Köln 51149 Germany

In many industrial processes the efficient removal of fine aerosol particles from dust laden gas streams is required. Especially when the gas contains a high concentration of water vapour or other condensible components, clogging may cause problems. Thus, dry operating dust collectors like bag filters for instance, can generally not be used. As an alternative, wet tubular electrostatic precipitators may be employed, which show outstanding collection efficiencies and moderate power consumption. A special feature of the electrostatic precipitator described in this paper is the continuously irrigated collection electrode by means of a liquid film. The advantages of this technology are as follows: Formation of a dust layer on the collection electrodes is avoided and thus no problems with reentrainment or back corona occur. There is no breakdown of the electric field during flushing as it occurs for spray irrigated electrostatic precipitators which are regenerated intermittently. The danger of clogging is low because contact between particles and the collection electrode is prevented by the liquid film. The liquid film can be grounded and therefore conducts the corona current. This makes it possible to manufacture the electrostatic precipitator from nonconductive materials (e.g. polypropylene, reinforced plastics) which allows cost effective design even when the gas streams contain corrosive components. The investigations under variation of the most important parameters showed that collection efficiency is much better than predicted by theory. In order to develop rules for scale up, dimensional analysis was used to define non-dimensional numbers that describe collection performance of tubular ESPs with different size.

2:20 PM

BENEFICIATION PERMITS RECYCLING OF TITANIA-RICH PIGMENT SLUDGES: *G. Belardi*[1]; L. Piga[1]; [1]Istituto Trattamento Minerali, Rome Italy

The main minerals employed for the manufacture of titanium products are ilmenite (FeTiO3) and rutile (TiO2). Owing to the progressive depletion of high-grade ilmenite deposits and to reduce the quantity of plant effluent, many TiO2 producers utilize Ti-enriched slags deriving from reductive electrosmelting of low-grade ilmenite ores. However, a considerable amount of TiO2-containing waste is generated during this process. A study has been conducted to assess a new wet beneficiation treatment for the recovery of the sulphuric-acid-soluble titanium oxide contained in fine wastes from an Italian titanium pigment facility. The main components of these wastes are TiO2 (54%, of which 42% acid-soluble and 12% insoluble), SiO2 (30%) and Al2O3 (6%). Eighty percent of the material is finer than 45 micron. The first step was to ascertain the possibilities offered by size classification, wet gravity separation, froth flotation and wet high-intensity magnetic separation to improve the grade and recovery of TiO2 and reduce the SiO2 content. The next step was to optimize the operating conditions of the various separations and assemble them into a complete flowsheet. Beneficiation ensures 70% recovery of the TiO2 with a concentrate grade of 80%.

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"CORDIAM" PROCESS: A NEW TREATMENT FOR ASBESTOS WASTES: *C. Abbruzzese*[1]; A. M. Marabini[1]; Federica Paglietti[1]; Paolo Plescia[1]; [1]CNR - Istituto Trattamento Minerali, Roma 00138 ITALY

In this work we present the Cordiam Process, a new patented method for detoxication of asbestos-containing wastes (ACW) and for subsequent recycling of inerts for production of ceramic materials, like cordierites and mullite-cordierites ceramics. The cordiam process work on all types of ACW: asbestos cement, insulation, asbestos board, brake and textiles. The proposed process involves mixing the refuse containing chrysotile or amphiboles with clays rich in kaolinites or kaolinite - illites to obtain a sintered refractory materials through a thermal solid state reaction. The reaction through chrysotile or amphiboles with Kaolinite clays occurs between 550 deg C and 950 deg C; this reaction involving a structural change of asbestos structures and the crystallization of olivine and pyroxene structures. Materials so obtained can be used in ceramic industries, to prepare catalytic supports or high temperature refractories or filters. With cordiam process it can be obtained to main results: - complete detoxication of ACW- complete recycling of ACW in a secondary raw materials

3:00 PM

SEXI: A NEW PORTABLE, IN SITU AND IN REAL TIME SPECTROMETER FOR CHEMICAL AND MINERALOGICAL CHARACTERIZATION OF MATERIALS: *Federica Paglietti*[1]; Paolo Plescia[1]; [1]CNR-National Council of Research, Institute for Mineral Processing, Rome Italy

This paper describes the characteristics of a new portable spectrometer, SEXI (Structural Elemental X-ray Instrument), developed for chemical and phases characterization of materials in situ. This spectrometer adopt a new geometry that permits a contemporary analysis of X-ray diffraction (XRD) and X-ray fluorescence (XRF). The system works with a new type of X-ray micro-tube with a power of 6 watt and an energy dispersive detector cooled with Peltier element. Weight of SEXI is around ten kilograms and it can operate in non-distructive mode virtually on all surfaces and samples, because it has a movimentation system that it can rotate on 4 axis. All movements are assured by step motors, controlled with a portable PC, that is also used for evaluation of data. The reveleability limit for XRF is around 50 ppm for metals and better for heavy elements. Another characteristics of SEXI is the possibility of great chemical and mineralogical mapping of surfaces: for example on great metal structures, walls or other wide surfaces. Some application of SEXI to materials characterization (metals, composites and ceramics) are also reported.

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THE INFORMATION SYSTEM "MOSSBAUER SPECTRA OF METALS AND MINERALS " : *Dr. Nikolay Ablesimov*[1]; Mrs. Lyuba Ablesimova[1]; [1]Institute of Materials FEB RAS, Khabarovsk Russia

The data base contains the information about the form sand parameters of the minerals, rocks and binary alloys Mossbauer spectra which were published in 1961-1995 years. It is necessary for qualitative and quantitative analysis of the iron-, tin- and antimony-contain minerals in the mixtures and iron-contain binary alloys in surface. STN data bases (such as GEOREF) are very extensive and non-concrete. It is uncomfortable for the investigator. Access to them is very expensive. The Mossbauer data are presented as the relational data base. It contains the Mossbauer parameters for the identification of compounds, for the graphic illustrations, as well as the experiment description and commentary. 1620 original science papers were put on the data base. It ensures the spectra search according to the composition and the crystalline structure of substances.

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SIMULATION OF METAL CYCLES FOR THE SUSTAINABLE METAL PRODUCTION ON PLAN(E)T EARTH: *M. A. Reuter*[1]; [1]Delft University of Technology, Department of Raw Materials Processing, Delft 2628 RX The Netherlands

The production of metals forms a complex web of metal flows. In recent papers the flow of zinc was mapped and optimal process routes synthesized. This approach basically advocates using the available processes and "optimizes" the link between unit operations to achieve an environmental as well economical sustainable zinc operation. In other words, the world is considered to be a larger plant in which as much as possible zinc producing processes are inter-linked to ensure sustainable zinc production. This synthesis approach did not take cognizance of the linked and associated metal producing industries. This paper will discuss an attempt to model this interaction, which visualizes the web of inter-linked and interrelated processes. This modelling can arguably be done considering the world as a processing plant and factories therein as unit operations, and simulating this scenario with plant simulation software. It is obvious that it is extremely difficult to model and parametrize all metal flows and interactions in the way suggested here. However, the poorly defined structure of eco-software for metal production and processing makes such an approach imperative to ensure that these software do not supply an overly simplified view of metallurgy. Therefore, rather than simplifying the problem, the proposed model attempts to supply economic and ecological detail even to the level of specific plants with their respective data. In addition to the above, this visualization of metal flows can assist in designing/selecting better global flow sheets for the future, especially in view of closing the metal cycles. In addition this approach can play a valuable part in education as well as assist politicians to make decisions.

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TESTING OF NEW ADDITIVES IN CONJUNCTION WITH THE REMOVAL OF COBALT FROM ZINC ELECTROLYTES BY CEMENTATION: *Amy Nelson*[1]*;* George P. Demopoulos[1]; George Houlachi[2]; [1]McGill University, Mining and Metallurgical Engineering, Montreal, Quebec H3A 2B2 Canada; [2]Noranda Technology Centre, Pointe Claire, Quebec H9R 1G5 Canada

Cobalt impurities are usually removed from zinc electrolyte solution by cementation with zinc dust prior to electrowinning. Although the thermodynamics for this reaction are favourable, kinetic barriers to cobalt reduction render the method almost useless in practice unless activators such as copper and antimony or arsenic are added. There is a large body of work studying the effect of these additives, yet the mechanism by which they act is still poorly understood. Moreover, regardless of the beneficial effect of the additives, from time to time the process fails to meet the target level of 0.1 ppm cobalt in the purified electrolyte, with negative consequences in the electrowinning operation. Even when the target conditions are met, zinc dust consumption is excessively high; satisfactory operation requires up to 150 times the stoichiometric amount of zinc dust. The objective of this work is three-fold: (1) to examine the effects of activators and eluci-

date the characteristics that make a good activator; (2) to identify new additives that could lead to a more efficient purification process; and (3) to test the new additives with synthetic and real electrolyte solutions. The present paper gives a status report on this project.

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HYDROTHERMAL REDUCTION OF COBALT HYDROXIDE TO COBALT POWDER PREPARATION: *Dr. Hun S. Chung*[1]; Dr. Dong J. Kim[1]; Dr. Kening Yu[2]; [1]Korea Institute of Geology, Mining & Materials, Materials Processing, Taejon 305-350 S. Korea; [2]Institute of Chemical Metallurgy/CAS , Hydrometallurgy, Beijing 100080 China

The hydrogen reduction was investigated to prepare fine cobalt powder from cobalt hydroxide slurry under hydrothermal conditions. The reduction rate was mainly the functions of the end pH, the amount of catalyst, hydrogen pressure and temperature. The induction period was affected highly by the hydrogen pressure. The cobalt powder obtained was spherical in shape with a particle size of less than 400nm with the solution pH of over approximately 6. The reduction kinetics in the temperature range of 145C to 195C were well fitted to a core model equation. It was found that the activation energy from the Arrhenius plot is 34.4KJ/mol and the process is controlled mainly by chemical reaction rather than diffusion in the temperature range of 145C to 195C. In addition, the cobalt ion reduction rate was closely related to the initial hydrogen pressure by assuming a gas chemisorption reaction type equation.

HIGH TEMPERATURE SUPERCONDUCTORS: Applications and Characterization

Sponsored by: Jt. Electronic, Magnetic & Photonic Materials Division/ Structural Materials Division, Superconducting Materials Committee

Program Organizers: U.Balu Balachandran, Argonne National Laboratory, 9700 S. Cass Ave. Bldg. 212, Argonne, IL 60439; Pradeep Haldar, Intermagnetics General Corp., 450 Old Niskayuna Rd., Latham, NY 12110; Paul McGinn, University of Notre Dame, Center for Materials Science, Notre Dame, IN 46556

Monday PM Room: Fiesta C
February 16, 1998 Location: Convention Center

Session Chair: D. U. Gubser,Naval Research Laboratory, Materials Physics Branch, Washington DC 20375

2:00 PM INVITED

SUPERCONDUCTING CABLES FOR ELECTRIC POWER TRANSMISSION: *Dr. Paul M. Grant*[1]; [1]EPRI, SR&D, Palo Alto, CA 94304 USA

Ever since its discovery in 1911, the application of superconductivity to electric power transmission has been a long-sought goal. It was not until the arrival of Type II superconductors and their development into practical wire that fulfillment of this dream seemed possible. Projects in the United States and Europe were undertaken in the 1970s and 80s to explore the realization of superconducting transmission cables, the most famous perhaps being the 115 m, 3 phase, 1000 MVA ac system comprised of Nb_3Sn tapes constructed and tested at the Brookhaven National Laboratory in the decade 1975-85. This project, although a technical success, was before its time both economically and in the role it could play in the changing utility technology of that era. The discovery of high-temperature superconductivity in 1986 gave new impetus to developing superconducting cable systems and such projects are currently underway in the US, Japan and Europe. It is expected that 1998 will see demonstration installations of ac underground superconducting cables at several locations in the US and Japan. In developing regions of the world, the transmission of enormous amounts of electrical power from widely-distanced generation sites (fossil, hydro, nuclear) and large urban load centers may require use of dc superconducting cables of completely new design. We will address the design issues, socio-economic as well as technical, of superconducting cable systems presently under consideration to meet both local distribution and long-distance transmission needs.

2:20 PM INVITED

HIGH FIELD HTS MAGNET TESTS: *Dr. Donald U. Gubser*[1]; Dr. Thomas L. Francavilla[1]; Dr. Robert J. Soulen, Jr.[1]; [1]Naval Research Laboratory, Materials Physics Branch, Washington, DC 20375-5343 USA

Operating characteristics of a laboratory magnet producing a field greater than 6 tesla and a gyrotron amplifier magnet producing a field of 3.5 tesla, both constructed with high temperature superconducting bismuth cuprate conductors, will be described. The laboratory magnet, built by American Superconductor Corporation, is conduction cooled with an operating temperature near 25K. It has a 5cm room temperature bore and a ramp time to full field of 4 minutes. At full field, the system will dissipate 10 watts of power in the steady state. The gyrotron magnet, built by Intermagnetics General, Inc. is also conduction cooled with an operating temperature near 10K. Size limitations and field homogeneity requirements, due to incorporation into an existing radar system, put severe demands on the magnet design and the superconductor performance which necessitated the low operating temperature. This system will be ultimately used in a 94 Ghz Navy radar demonstration.

2:40 PM

HIGH-TC SUPERCONDUCTORS FOR ELECTRIC POWER APPLICATIONS: *Dr. U. Balachandran*[1]; [1]Argonne National Laboratory, Energy Technology Division, Argonne, IL 60439 USA

Because of the expected large impact of high-T_c superconductors on the power industry and end users of electricity, the U.S. Department of Energy set up the Superconductivity Partnership Initiative (SPI) program, through which it has established alliances with national laboratories, universities, and private industries. SPI, a flagship program in the nation's effort to commercialize high-T_c technologies, was started in fiscal year 1994. Activities of the Initiative focus on development of a prototype generator coil, fault-current limiter, transmission cable, and motor. Long lengths of BSCCO superconductor tapes are used for the prototype products and each prototype has either set a world record or far exceeded its original design objectives. Because of the flux creep problem, use of BSCCO tape rules out an operating temperature above 30 K. New superconductor prospects have emerged with the development of coated YBCO conductors that can operate at higher temperatures. The current status of the development of various prototype devices, together with recent progress in the development of high-T_c conductors, will be presented. Work is supported by the U.S. Department of Energy (DOE), Energy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, under Contract W-31-109-Eng-38.

3:00 PM INVITED

APPLICATIONS OF HIGH TEMPERATURE SUPERCONDUCTORS FOR MAGNETO-MECHANICAL DEVICES IN SPACE : *Dr. Ki Bui Ma*[1]; Dr. Eunjeong Lee[1]; Dr. Jang-Horng Yu[1]; Dr. Wei-Kan Chu[1]; [1]University of Houston, Texas Center for Superconductivity, Houston, Texas 77204-5932 USA

In the practical application of high temperature superconductors (HTSs) in mechanical devices such as magnetic levitation bearings on earth, enough thrust has to be provided to support the weight of the rotor, and the superconductor has to be kept sufficiently cold. Both of these requirements are easier to comply with in low earth orbit or the lunar surface, and naturally satisfied in deep space. This renders mechanical devices made with magnets and HTSs attractive candidates to replace mechanical components of space equipment that find it harsh to operate under the cold vacuum of space. Here, we will discuss some potential extraterrestrial applications of HTSs such as low loss bearings for reaction wheels on mini-satellites, wide gap bearings for telescope mounts on the moon, vibration isolation of noise sources onboard the

space station and vibration damping of truss structure in the next generation of deep space infra-red observatories.

3:20 PM INVITED
CHARACTERIZATION OF HIGH CRITICAL TEMPERATURE SUPERCONDUCTING WIRES FOR SUPERCONDUCTING MOTORS: *Kathryn L. Zeisler-Mashl*[1]; Thomas L. Francavilla[1]; Robert A. Masumura[1]; Chandra S. Pande[1]; [1]Naval Research Laboratory, Matls. Science & Tech. Div., Washington, DC 20375-5343 USA

Superconducting motor performance depends critically on the magnitude of the current density and its stability with time. These properties are strongly influenced by the high Tc superconductor microstructure. The goal of this study is to characterize both current transport and microstructures in high Tc superconductors in order to optimize material performance. Initial work has focused on BSCCO tapes. Superconducting properties were measured as a function of temperature and applied magnetic field for sections of this material. Additionally, the microstructures were characterized using optical and electron microscopy and x-ray diffraction. These microstructural parameters were then correlated with the superconducting properties in order to rank their relative importance and to predict the combinations which should maximize superconductor performance.

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ELECTROMECHANICAL CHARACTERIZATION OF SILVER-CLAD BSCCO SUPERCONDUCTORS: *Dr. Sherif Salib*[1]; Dr. Anand Iyer[1]; Dr. C. Vipulanandan[1]; Dr. K. Salama[1]; Dr. U. Balachandran[1]; [1]University of Houston, Texas Center for Superconductivity, Houston, TX 77204-4792

Significant progress has been made in the development of high-temperature superconductors (HTS) for possible electric power and high field magnet applications. However, practical applications of HTS will be governed by their electrical and mechanical characteristics. During fabrication and service the conductors are subjected to stresses which could lead to degradation in their electrical properties. In the present investigation, effort was expended to evaluate the electro-mechanical characteristics of the silver-clad BSCCO conductors. Extensive studies, at 77 K, of the monofilament and composite BSCCO conductors were evaluated by subjecting the tapes to in-situ tensile test. The irreversible strain limit of the monofilament and composite tapes were 0.15 and 0.6%, respectively. Detailed phase and microstructural analysis have been conducted using XRD and SEM. A model developed based on the constituent properties of the tapes was used to verify the performance of the tapes.

4:10 PM INVITED
AC LOSS AND MATRIX RESISTIVITY IN MULTIFILAMENTARY HTSC TAPES: *Dr. M. D. Sumption*[1]; Dr. E. W. Collings[1]; [1]The Ohio State University, Columbus, OH 43210 USA

Measurements of magnetization, M, versus magnetic field strength, H (and hence AC loss), have been made on multifilamentary Bi2223/Ag tape fabricated partly by the "continuous tube forming/filling" (CTFF) process. Transverse matrix resistivities, $\rho_{\perp}$, were extracted from the inverse slopes of the loop-height, $\Delta M(H)$, versus dH/dt curves and compared with the semiempirically determined H-dependent resistivity of the matrix material, $\rho_{Ag}(H,T)$. Careful examination of normalized 4.2 K and 30 K $\rho_{Ag}(H,T)$ data revealed the existence of conventional eddy current loss. However, the absolute values of the losses were much lower than expected, leading to actual values of $\rho_{\perp}$ (H,T) that were two orders of magnitude larger than the filling-factor-corrected $\rho_{Ag}(T)$. Then at 60 K a new phenomenon was observed. Already enhanced at zero field, $\rho_{\perp 60\ K}$ increased still further with increasing field strength, a phenomenon which we tentatively attribute to an extra resistive contribution to the eddy current path arising from flow-like vortex motion within the filaments.

4:30 PM INVITED
REDUCING AC LOSSES OF Bi(2223) TAPE BY OXIDE BARRIER: *Dr Yibing Huang*[1]; Prof. Rene Flukiger[1]; Mr. G Grasso[1]; [1]Univ. Geneva, Group of Applied Physics, Geneva 1211-ch Switzerland

A significant reduction of ac coupling losses has been achieved in Bi(2223) multifilamentary tapes by introducing a thin layer of high resistive BaZrO3 barrier around each filament, thus increasing the transverse resistivity by a factor of 10. The electric decoupling effect of these barrier layers is clearly demonstrated by 'magnetic length scale'- and Hall sensor magnetization experiments. These tapes can also be twisted with a twist pitch of 2 cm, which yields an even higher decoupling effect without deterioration of the transport properties. The uniformity of filament size and barrier layer thickness have been improved by a modified Four Roll PIT method. With this new technique jc values of the tapes with barriers reaches so far 15000 A/cm2 (77K, 0T).

4:50 PM INVITED
MAGNETO-OPTICAL OBSERVATION OF MAGNETIC FLUX BEHAVIOR AND CURRENT DISTRIBUTION IN HIGH-T_C SUPERCONDUCTORS: *Dr. Anatolii A. Polyanskii*[1]; *Prof. David C. Larbalestier*[1]; [1]University of Wisconsin, Applied Superconductivity Center, Madison, WI 53706 USA

Very important question of high temperature superconductors (HTS) is a knowledge of the mechanisms controlling the critical current density in particular conductor forms. Existence of various defects such as some high angle grain boundaries (HAGB), cracks and large second phase particles define the current carrying capability of HTS rather than the pinning of vortices in conventional superconductors. Magneto-optical technique, based on the Faraday rotation in a ferrimagnetic Bi-doped garnet indicator films with in-plane anisotropy, was used to study flux penetration and current distribution in both BSCCO tapes and polycrystalline films based on Tl and Y components. In BSCCO tapes a magnetization current flow preferably at the silver sheath and percolative, granular current patterns in the central part of the tape were observed. By applying transport current this granular behavior was replaced by a more uniform current flow. The correlation of MOI with the microstructure data taken on polycrystalline films revealed that the flux preferentially penetrated into the film along colonies HAGB, which causing a significant distortion of current stream lines and resulting in percolative current flow in the film. Analysis of magneto-optical images suggests that current carrying capability of particular conductor forms can be significantly improved by eliminating various defects in HTS.

HUME ROTHERY AWARD FOR PROF. RYOICHI KIKUCHI: Hume-Rothery Symposium II

Sponsored by: Jt. Electronic, Magnetic & Photonic Materials Division/ Structural Materials Division, Alloy Phases Committee
Program Organizers: Juan M. Sanchez, The University of Texas, Center for Materials Science, Austin, TX 78712; Lawrence Anthony, The University of Toledo, Dept. of Physics and Astronomy, Toledo, OH 43606

Monday PM Room: 104
February 16, 1998 Location: Convention Center

Session Chair: Didier de Fontaine, University of California, Materials Science, Berkeley, CA 94720-1760

2:00 PM INVITED
CHEMICAL SELF-ORGANIZATION DURING CRYSTAL GROWTH: *Dr. Francois Ducastelle*[1]; Dr. Pascal Quemerais[2]; [1]ONERA, Materials Department, 92322 Chatillon Cedex, France; [2]CNRS/LEPES, 38042 Grenoble Cedex, France

A chemical growth model for binary alloys is presented. At each step, using a simple energetic criterion, an A or a B atom is aggregated to the growing cluster. Two one-dimensional models are considered.

The first one is a lattice-gas model with short-range decreasing and convex chemical interactions. It is proved that the growing structures are the so-called uniform structures where the atoms are arranged as regularly as possible, and which are known to be the ground state equilibrium structures of the same model. When they are not convex, other ordered structures can grow for appropriate initial conditions. The second model uses an electronic tight-binding hamiltonian for which the growth of uniform structures is also observed.

2:30 PM INVITED

CONFIGURATIONAL KINETICS AND ELECTRICAL RESISTIVITY STUDIED BY PATH PROBABILITY METHOD: *Dr. Tetsuo Mohri*[1]; [1]Hokkaido University, Division of Materials Science and Engineering, 060 Sapporo, Hokkaido, Japan

Cluster Variation Method(CVM) devised by Kikuchi has been recognized as a powerful means to study configurational thermodynamics of a given alloy system. Recently, by combining with electronic structure calculations, even first-principles calculation of alloy thermodynamics has been attempted. Path Probability Method is a natural extension of the CVM to time domain, and, therefore, is best suited to trace the time evolution behavior of an alloy configuration during the relaxation process. We have attempted several types of calculations for fcc-based systems. Among them are, time evolution of order parameters and diffuse intensity spectrum, fluctuation spectrum around the kinetic path etc. The main focus of the present study is placed on the simulation of electrical resistivity in the ordered phase field. Experimentally, they detected not only the single relaxation time but also two kinds of relaxation times, indicating the coexistence of more than two relaxation mechanisms. Also interesting is the pseudo critical slowing down behavior observed in the vicinity of transition temperature. Based on the phase diagram calculated by CVM, attempts are made to simulate above intriguing phenomena of electrical resistivity by Path Probability Method.

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KWW TYPE RELAXATION PROCESS OF IONIC CONDUCTION IN LATTICE GAS MODELS: *Dr. Hiroshi Sato*[1]; [1]Purdue University, School of Materials Engineering, W. Lafayette, IN 47907-1289 USA

The relaxation process of ionic conductivity in solid electrolytes is characterized as Kohlrausch-Williams-Watts(KWW) type. This behavior is generally considered to be an anomaly created by complicated interactions among constituent particles and disorder in their distribution. Earlier, we showed that by an heuristic, but analytical calculation in the format of the linear response theory, by starting from the pair approximation of the path Probability method (PPM) in lattice gas models, that the relaxation motion of a single particle in a lattice gas is intrinsically KWW without interaction and disorder [1]. The relaxation process is represented by the time dependent correlation factor for a single tracer ion when a constant external field which acts only on tracer particles is switched on at t=0. The major interest of the presentation is to show how the format of the linear response theory which deals with following the motion of a single tracer ion analytically. The PPM leads to the Debye relaxation process, while converting it to the format of the linear response theory leads to the KWW relaxation process. From this, possible reasons why the appearance of the KWW behavior is considered to be an anomaly are speculated. [1] H.Sato, A, Datta and T. Ishikawa, Solid State Ionics 86-88(1996)1319; J. Noncrystalline Solids 203 (1996) 306.

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GLOBAL ANALOGUES OF THE PATH PROBABILITY METHOD WITH APPLICATIONS TO WEAKLY NONLINEAR ISOTHERMAL MATERIALS PATTERN FORMATION: *Dr. J. S. Kirkaldy*[1]; [1]McMaster University, Brockhouse Institute for Materials Research, Hamilton, Ontario L8S 4M1 Canada

Under the circumstances that a patterning subsystem and its heat bath are near isothermal, and that the topological defects such as phase boundaries and dislocations which comprise the pattern are sufficiently dilute, implying weak non-linearity, the free boundary degeneracies can be removed via appropriate variants of the Onsager-Kikuchi Path Probability Method. This structure emerges globally as obeying a time-dependent, steady-state or equilibrium Ginzburg-Landau (G-L) equation or as a principle of dissipation stationarity in patterning order parameter space at the steady state. The steady state G-L version describes spinodal decomposition in discontinuous form as observe in Au-Ni. With Kikuchi we take the norm for verification of this statistical procedure as Klein's two-level quantum model (which is a special case of the Master Equation Method) and illustrate how the explicit solutions as a function of virtual initial conditions varying both towards equilibrium with the heat bath and saturation of the free energy source about any given stable steady state map out a saddle surface in both the dissipation rate and the Helmholtz Free Energy. We further indicate how Hamilton's Principle for multiparticle frictional systems, force to compatibility with the first and second laws, and correspondingly to an order parameter space, leads to an equivalent and more general result.

4:10 PM INVITED

VIBRATIONAL ENTROPY DIFFERENCE BETWEEN ORDERED AND DISORDERED PD_3V: *Michael E. Manley*[1]; Laura J. Nagel[2]; Brent Fultz[1]; [1]Calif. Inst. of Technology, Eng. and Applied Science, Pasadena, CA 91125 USA [2]West Texas A&M University, Engineering Technology, Canyon, TX USA

We are investigating the difference in vibrational entropy between DO_{22} ordered and fcc disordered Pd_3V. Differential thermal expansion measurements were performed at cryogenic temperatures on the ordered and disordered phases. Two metal strips of the same dimensions—one ordered and one disordered—were combined to make a bi-layer. The difference in thermal expansion was determined by cantilevering the bi-layer and optically measuring the change in slope at the free end. Heat capacity was extracted from the thermal expansion data to determine vibrational entropy. We used cryogenic calorimetry to measure directly the difference in heat capacity between the ordered and disordered phases. Results from both techniques will be presented. This work was supported by the U. S. Department of Energy under contract DE-FG03-96ER45572.

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SUPERHEATING, SUPERCOOLING AND TRANSIENT METASTABLE PHASES ASSOCIATED WITH THE FIRST ORDER MAGNETIC TRANSITION IN THE HEAVY LANTHANIDES — Tb, Dy, Ho AND Er: *Dr. Karl A. Gschneidner, Jr.*[1]; Dr. Vitalij K. Pecharsky[1]; Dr. David Fort[2]; [1]Iowa State University, Ames Laboratory, Ames, Iowa 50011-3020 USA; [2]University of Birmingham, School of Metallurgy and Materials, Birmingham B15 2TT, UK

A study of the low temperature properties of ultrapure heavy lanthanide metals (Tb, Dy, Ho and Er) revealed some unusual behaviors associated with the first order magnetic transformations, which occur at 220, 90, 20 and 20K, respectively. Both superheating and supercooling were observed in these metals during heat capacity and cooling/heating curve measurements. In some cases metastable intermediate states were observed during the transformation from the ferromagnetic (F) to antiferromagnetic (A) state and the reverse. For example, a total of four such transient states are observed in Er on heating (F->A) and two on cooling (A->F), while no intermediates are observed in Dy. This work was supported by U.S. Dept. of Energy, Office of Basic Energy Sciences, Division of Material Sciences, under Contract No. W-7405-ENG-82.

INTERNATIONAL SYMPOSIUM ON IRON ALUMINIDES: ALLOY DESIGN, PROCESSING, PROPERTIES & APPLICATIONS: Physical Properties and Point Defects

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee, Jt. Electronic, Magnetic and Photonic Materials Division/ Structural Materials Division, Alloy Phases Committee, Materials Design and Manufacturing Division, Powder Metallurgy Committee

Program Organizers: S.C. Deevi, Philip Morris USA, Research & Development Center, Richmond, VA 23234; David G. Morris, University of Neuchatel, Inst of Structural Metallurgy, Neuchatel Switzerland; J.H. Schneibel, Oak Ridge National Laboratory, Metals & Ceramics Division, Oak Ridge, TN 37831; Vinod K. Sikka, Oak Ridge National Laboratory, Metals & Ceramics Division, Oak Ridge, TN 37831

Monday PM Room: 108
February 16, 1998 Location: Convention Center

Session Chairs: R. R. Judkins, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831; P.R. Munroe, University of New South Wales, School of Materials Science and Engineering, Sydney, NSW 2052 Australia

2:00 PM INVITED

ELECTRICAL PROPERTIES OF IRON ALUMINIDES: *A. C. Lilly*[1]; S. C. Deevi[1]; [1]Philip Morris, Richmond, VA 23234 USA

Intermetallics based on iron aluminides are being developed as structural materials due to their superior strengths, and oxidation and corrosion resistances over many of the conventional steels, and alloys. In addition, iron aluminides exhibit interesting electrical properties with increase of Al content. For example, the electrical resistivities of Fe-Al alloys, and Fe_3Al increase with increase of Al content, and the electrical resistivity of iron aluminides can be as high as 160 micro Ohm-cm in the vicinity of Fe_3Al phase field. Interestingly, the electrical resistivities of iron aluminides based on FeAl (with a B2 structure) exhibit a decrease with increase of Al content in the FeAl phase field. Also, the electrical resistivity of iron aluminides increases with increase of temperature, and exhibits a positive temperature coefficient of resistance. In this paper, we present the electrical properties of iron aluminides based on Fe-Al alloys, Fe_3Al, and FeAl alloys. In addition, we will discuss the influence of defect structure/vacancies (generated by the composition and heat treatment) on the electrical properties of FeAl. The results will be discussed based on the electronic band structure of the alloys.

2:30 PM INVITED

ELECTRICAL RESISTANCE ANOMALY IN Fe_3Al-BASED ALLOYS WITH THE SUBSTITUTION OF TRANSITION ELEMENTS: *Yoichi Nishino*[1]; [1]Nagoya Institute of Technology, Dept. Materials Science & Engineering, Nagoya 466 Japan

The electrical resistivity of $D0_3$-type $(Fe_{1-x}M_x)_3Al$ alloys with transition elements M = Ti, V, Cr, Mn and Mo shows an abnormal temperature dependence: a resistance maximum near the Curie point and a negative resistivity slope at higher temperatures. In particular, the Heusler-type Fe_2VAl exhibits a semiconductor-like behavior with a large resistivity of about 3000 $\mu\Omega$ cm at 4.2 K, in spite of the possession of a sharp Fermi edge. A substantial mass enhancement deduced from specific-heat studies is believed to be responsible for the anomalous electron transport. At a temperature above 800 K, the resistivity curves show an inflection as a sign of the $D0_3$-B2 transformation. The substitution of M = Ti, V and Mo is particularly effective for raising the transformation temperature T_0. The solute effect on T_0 can be related to the variation of electron concentration, which is an important factor for the site preference of the substituents in the $D0_3$ superlattice.

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EFFECT OF TEMPERATURE ON ELASTIC CONSTANTS AND SLIP BEHAVIOR OF Fe-30% AlSINGLE CRYSTALS: *C. Hartig*[1]; M. H. Yoo[2]; M. Koeppe[1]; H. Mecking[1]; [1]Technical University Hamburg-Harburg, Physics and Technology of Materials, 21071 Hamburg Germany; [2]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

The elastic constants of Fe-30% Al single crystals were determined using a resonance technique over the temperature range of 293 to 1173 K, including the observed peak of the yield strength at 750 K. Simple shear tests of Fe-30% Al single crystals were performed in order to measure the critical resolved shear stresses for {110}<111> slip and {112}<111> slip independently and without superimposed normal stresses on the slip planes. From the elastic stiffnesses and compliances, the elastic shear anisotropy A, Zener's factor, and the factor M related to a shear in a <111> direction were determined. Zener's factor is high, A = 4.6 to 6.8, whereas M is rather low, M = 1.2 to 1.3, over the measured temperature range. The values of the elastic anisotropy factors for the dislocation width calculated for the {110}<111> slip dislocations suggest that the mobility is significantly higher for edge than for screw character in the temperature range of the peak stress. For a more detailed study of dislocation micromechanics to explain the plastic deformation behavior of Fe-30% Al, discussion is given on the anisotropy of APB energies, the relationship of non-Schmid effect with the anisotropic coupling coefficients, and possible glide resistance associated with the unstable orientation of dislocation line tension.

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THERMODYNAMIC FACTOR IN INTERDIFFUSION OF FeAl ALLOYS FROM THE DIFFUSE X-RAY SCATTERING: *Dr. Simon Dorfman*[1]; Prof. David Fuks[2]; Mr. Vlad Liubich[2]; [1]Technion, Dept. of Physics, Haifa 32000 Israel; [2]Ben-Gurion University of the Negev, Materials Enginering Dept., Beer Sheva 84105 Israel

The diffusion behaviour can be studied in binary systems for which appropriate radioisotopes for both constituents of the alloy are available. With respect to the Fe-Al system appropriate and inexpensive radioisotopes are available for Fe component but not for Al. In the framework of the Boltzmann-Matano method the diffusion behaviour of Fe-Al intermetallics can be gained and interdiffusion coefficients can be determined. The interdiffusion coefficient is related via the modified Darken equation to the tracer diffusivities of the constituents. The thermodynamic factor entering the Darken equation is proportional to the second derivative of the Gibbs free energy of the alloy on the molar fraction of one of components. These relations are used to deduce the tracer diffusivity of Al provided that the tracer diffusivity of Fe, the interdiffusion coefficient and the thermodynamic factor are known. Calculations are done on the basis of the X-ray diffuse scattering data for B2 FeAl phase.

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VACANCY CONCENTRATIONS AND HARDENING IN FeAl ALLOYS: L. M. Pike[1]; *Y. A. Chang*[1]; C. T. Liu[2]; [1]University of Wisconsin-Madison, Department of Materials Science and Engineering, Madison, WI 53706 USA; [2]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

The intermetallic compound FeAl with the B2 crystal structure is well known to have significant numbers of thermal vacancies at elevated temperatures. These vacancies often remain after slowly cooling or even after long-term low-temperature heat treatments. Moreover, these vacancies are responsible for significant solid solution hardening and concomitant loss of ductility in these alloys. The relationship between hardness and vacancy concentration in FeAl will be reviewed. This discussion will initially focus on binary alloys, but will be extended to include the effects of ternary alloy additions. Recent work to measure vacancy concentrations at temperature will also be discussed.

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THE EFFECT OF PROLONGED LOW-TEMPERATURE ANNEALING ON THE HARDNESS OF (Fe,Ni)Al ALLOYS: *P. R. Munroe*[1]; C. H. Kong[1]; [1]University of New South Wales, School of Materials Science and Engineering, Sydney, NSW 2052 Australia

It is now well-established that thermal vacancies severely affect the mechanical properties of FeAl, and an understanding of their formation and removal from the lattice is required in order to control the mechanical properties of FeAl-based materials. It has also been shown that nickel additions which replace iron severely harden FeAl, and subsequently it has been shown that one effect of the nickel atoms is to slow the removal of thermal vacancies from the lattice. As a consequence, this study has examined the hardness and microstructure of (Fe,Ni)Al alloys following very long anneals (up to a month). It is shown that although such heat treatments are sufficient to remove retained thermal vacancies, the nickel still produces hardening effects in excess of those expected from solute strengthening effect alone. The hardening effects of nickel are believed to be associated with the formation complex defects in the lattice and changes in slip system.

4:50 PM INVITED

NEUTRON DIFFRACTION STUDY ON SITE OCCUPATION OF SUBSTITUTIONAL ELEMENTS AT SUBLATTICES IN Fe3Al INTERMETALLICS: *Prof. Zuqing Sun*[1]; Wangyue Yang[1]; Lizhen Shen[1]; Yuanzing Huang[1]; Baisheng Zhang[2]; Jilian Yang[2]; [1]University of Science and Technology Beijing, Department of Materials Science and Engineering, Beijing 100083 China; [2]China Institute of Atomic Energy, Beijing 102413 China

The site occupation of substitutional elements Cr, Mo, Ti, Mn, Si, at sublattices in DO_3-type stoicheometric Fe_3Al intermetallics, and effect of the elements on magnetic momentum of unit cell and DO_3-B2 transformation behavior have been determined by means of diffractometry. The experimental results indicate that Cr, Mo, and Ti atoms all occupy the next nearest neighbour of Al atoms, Ni, and Mn atoms the nearest neighbour, while Si atoms occupy the positions of Al atoms. All the alloying elements investigated in the present work modify the magnetic momentums of two type Fe atoms in the unit cell. Based on the above results and the corresponding mechanical measurements, the effects of substitutional alloying elements on the mechanical properties are discussed.

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TENSILE PROPERTIES OF Fe-Al SINGLE CRYSTALS STRENGTHENED BY EXCESS VACANCIES: *Kyosuke Yoshimi*[1]; Yoshiyasu Saeki[2]; Man H. Yoo[3]; Shuji Hanada[1]; [1]Tohoku University, Institute for Materials Research, Sendai 980-77 Japan; [2]Tohoku University, Department of Materials Processing, Sendai 980-77 Japan; [3]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115

In this study, to investigate the effect of excess vacancies on plastic flow of Fe-Al, tensile properties of single crystals containing 33, 41 and 44 mol.% aluminum were examined. Vacancy elimination heat treatment slightly diminishes the critical resolved shear stress (c.r.s.s.) of as-homogenized specimens for all the alloys. The work hardening rate (w.h.r) is similar between as-homogenized and vacancy-eliminated specimens. Air-cooling drastically enhances c.r.s.s. and reduces elongation for all the alloys, and the c.r.s.s. increases and the elongation decreases as aluminum concentration increases. The w.h.r. of Fe-41mol.%Al is apparently reduced by air-cooling, while that of Fe-33mol.% Al is not almost changed. Interestingly, air-cooled specimens exhibit serrated flow or Luders yielding for all the alloys. Numerous nano-loops of dislocations having the same Burgers vector as glide dislocations were observed in strained, air-cooled specimens. Based on the obtained results, an excess vacancy strengthening model will be considered.

INTERNATIONAL SYMPOSIUM ON PROCESSING OF METALS & ADVANCED MATERIALS: Design and Microstructures

Sponsored by: Extraction & Processing Division, Synthesis, Control, and Analysis in Materials Processing Committee

Program Organizer: Ben Q. Li, Washington State University, School of Mechanical and Materials Engineering, Pullman, WA 99164-2920

Monday PM — Room: 201
February 16, 1998 — Location: Convention Center

Session Chair: Yimin Ruan, Aluminum Company of America, Alcoa Center, PA 15069 USA, Ben Q. Li, Washington State University, Pullman, WA 99163 USA

2:00 PM

INVERSE CONTINUOUS SHEET QUENCHING PROCESS DESIGNS: *Yimin Ruan*[1]; [1]Aluminum Group of America, Alcoa Technical Center, New Kingston, PA 15069 USA

An inverse design method is developed to determine optimal cooling conditions for the continuous quenching of precipitation hardenable sheet alloys to achieve a required yield strength. The design objective is to optimize the quenching process so that the required yield strength can be achieved. With inverse design method, the required yield strength is specified. The cooling condition during quenching is optimized with the conjugate gradient method. The adjoint system is developed to compute the gradient of the objective functional required by the conjugate gradient method. An Aluminum sheet quenching problem is presented to demonstrate the inverse design method.

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MICROSTRUCTURES IN COMPOSITES OF AGE HARDENABLE ALUMINUM ALLOYS DEFORMED BY ROOM TEMPERATURE ROLLING AND TENSILE TESTING: *S. K. Varma*[1]; Daniel Salas[1]; Erica Corral[1]; Erika Esquivel[1]; Miriam Regalado[1]; [1]The University of Texas El Paso, Department of Metallurgical and Materials Engineering, El Paso, TX 79968-0520 USA

The composites of 2014 aluminum alloys reinforced with AI, O, particles have been deformed at room temperature tensile testing and rolling. The composites were solutionized for various times at 540°C. The influence of dislocations generated by the coefficient of thermal expansion (CTE) effect and the grain sizes evolved during solutionizing on the microstructural development during the two types of deformation will be investigated. An attempt has been to determine the bonding characteristics, between the particles and the matrix, in the composites as influenced by the solutionizing time with the help of fractured samples from the tensile testing. The details of microstructural features will be correlated to either the flow stress or hardness values. The results will be compared with those in the monolithic alloy heat treated under similar conditions. This research has been supported by the National Science Foundation through the grant number HRD-9353547.

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THE TECHNOLOGY OF QUENCH; HARDENING OF ROLLED PRODUCTS BASED ON CYCLICAL HEAT TREATMENT: *Alexey Lozhko*[1]; Ludmila Sidorenkova[1]; [1]The State Metallurgical Academy of Ukraine, Dniepropetrovsk UA-320635 Ukraine

During heat treatment for metals, the product surface is exposed to multiple fast heating and cooling. To ensure product quality, cooling effects must be carefully controlled. It is known that turbulence enhances heat exchange and increases cooling effect without increasing of volume flow rate of water. To maintain the regularity of heating and cooling, the technology for the cyclic heat treatment of rolling prod-

ucts, which involves forming mobile zones of turbulent heat exchange on a surface of a product, was developed. Two face spray systems are installed, each having six long-range injectors and being located circularly. Two flows of water move towards each other along the surface of the workpiece. When they collide, a ring zone of turbulent heat ex change is created and heat transfer in this zone is of 10-15 time more intensive than in other regions. The turbulent zone is moved on the workpiece by adjusting volume flow rate. The speed of turbulence zone movement is chosen such that cooling penetrate s a small depth only. Thus, after the turbulent zone passes a predetermined portion of a surface, the temperature can be recovered quickly. Mathematical simulations are conducted to determine the process parameters, which among others include the movement of collision point of the flows. Experiments are also conducted. Results show that the frequency of movement of turbulent zone is an important technological parameter. It defines the mechanical properties of the surface of the workpiece and should be chosen based on the profile of workpiece and the metal grade.

3:00 PM

EFFECTS OF SOLID/MELT INTERFACE SHAPES ON MICROSTRUCTURAL DEVELOPMENT DURING CONTINUOUS CASTING PROCESS: *Jemin Park*[1]; Yong J. Kim[1]; [1]Pohang University of Science and Technology, Graduate School of Iron and Steel Technology, Center for Advanced Aerospace Materials, Pohang 790-784 Korea

Solid/liquid interface shape during continuous casting process has a strong influence on the resulting microstructure of the ingot. The interface shape is primarily governed by the heat flow condition between the molten metal and a mold (or container). The role of the microstructural transition resulting from various solid/liquid interface shapes will be discussed using pure tin and Pb-75wt%Sn alloy. Three different types of mold such as cold mold, resistant heated mold, and electromagnetically heated mold were used to generate those interfaces having different curvatures. Experimental measurements of the complete temperature profiles along the axis and edge of the ingot are used to determine the solid/liquid interface geometry. The results are presented along with their implications to the ingot property and microstructures.

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COMPUTER MODELING OF THE FABRIC STACKING SEQUENCE EFFECTS ON MECHANICAL PROPERTIES OF A SIC CERAMIC COMPOSITE WITH PLAIN-WEAVE SIC FIBER REINFORCEMENTS: *Mr. Wei Zhao*[1]; Dr. N. Allen Yu[1]; Dr. Peter K. Liaw[1]; [1]University of Tennessee at Knoxville, Dept. of Materials Sci. & Engr., Knoxville, Tennessee 37996-2200 USA

For a plain-weave SiC fiber-fabric reinforced silicon carbide (SiC) ceramic composite, little attention has been paid to the lamina stacking sequence effect. In this paper, the fiber-fabric stacking sequence effects on the elastic stress distribution in laminated flexural testing bars, and the elastic properties of the laminate are quantified. A computation model is established based on the classical laminated plate theories, and a fiber undulation model developed by Chou et al is employed to estimate the reduced stiffness matrix of the composite fabric. A FORTRAN program is compiled to implement the modeling. The mechanical behavior of continuous fiber reinforced ceramic matrix composites with different combination of fiber lamina orientation and their stacking sequence are studied. Acknowledgment The research is sponsored by a National Science Foundation project on ceramic matrix composites (under contract number: NSF EEC-9527527) to The University of Tennessee at Knoxville with Mrs. Mary Poats as the contract monitor.

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AN INTEGRATED MODEL FOR THERMOMECHANICAL DEFORMATION AND MICROSTRUCTURE EVOLUTION DURING METAL FORGING: S. Illendula[1]; R. A. Mirsham[2]; B. Q. Li[1]; [1]Washington State University, Dept. of Mechanical Engineering, Pullman, WA 99163 USA

A mathematical model is developed to represent the thermomechanical behavior of the materials and microstructure evolution during metal forging. The model development is based on the finite element solution of the thermoviscoplastic deformation of metals coupled with the internal state variable model describing the microstructure evolution involving work hardening, recovery, recrystallization and grain size distribution. The procedures for the integration of the microstructure model into the macro finite element model are described. Computed results for both macro and micro phenomena during metal forging, such thermomechanical deformation and temperature distribution, grain size evolution, recrystallization volume fraction and stress-strain relations will be presented.

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RELATIONS OF MICROSTRUCTURE AND FLEXURAL STRENGTH OF A 2-D SiC/SiC CERAMIC COMPOSITE BEFORE AND AFTER OXIDATION: *Dr. Wei Zhao*[1]; Dr. David C. Joy[1]; Dr. Peter K. Liaw[1], [1]University of Tennessee at Knoxville, Dept. of Material Sci. & Engr., Knoxville, Tennessee 37996-2200 USA

In this paper, the mechanical properties of ceramic matrix composites (CMCs) were examined by correlating the microstructure features and flexural strengths of the CMCs. As-received and oxidized specimens were studied. Failure mechanisms were investigated using scanning electron microscopy (SEM). The chemical change after oxidation such as Si, C, and O, and their distributions were detected by SEM linescan chemical analysis technique. The effects of oxidation and porosity on the mechanical properties of the CMC, such as flexural strength and toughness were also discussed. Acknowledgment:The research is sponsored by a National Science Foundation project on ceramic matrix composites (under contract number: NSF EEC-9527527) to The University of Tennessee at Knoxville with Mrs. Mary Poats as the contract monitor.

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INFLUENCE OF REINFORCEMENT MORPHOLOGY ON THE MECHANICAL PROPERTIES OF SHORT-FIBER COMPOSITES: *Y. T. Zhu*[1]; S. Zhou[2]; N. Shi[3]; J. A. Valdez[1]; A. L.. Lovatol[1]; M. G. Stout[1]; B. R. Blumenthal[1]; T. C. Lowe[1], [1]Los Alamos National Laboratory, Division of Materials Science and Technology, MS G755, Los Alamos, NM 87545 USA

A major problem of short-fiber composites is the interfaces between the fiber and matrix, which play a critical role and, in many cases, become a limiting factor in improving such mechanical properties as strength of the composites. For a short fiber, a strong interface is desired to effectively transfer load from matrix to fiber, thus reducing the ineffective fiber length. However, a strong interface will make it difficult to relieve fiber stress concentration in front of an approaching crack; and such stress concentration can result in fiber breakage. We report in this paper an innovative approach to overcome this problem: reinforcement morphology design. Short-fibers with enlarged ends are processed and used to reinforce polyester matrix. The initial results show that the bone-shaped short-fiber composite has a significantly higher strength than the conventional short straight fiber composite.

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RESISTANT TMT BARS USING SMAW PROCESS: Ramen Datta[1]; D. Mukerjee[1]; Sanak Mishra[1]; R. Veeraraghavan[2]; K.L. Rohira[2]; [1]R&D Centre for Iron and Steel, SAIL, Ranchi-834002, India; [2]Welding Research Institute, Tiruchirapally-620014, India

Recent efforts at Steel Authority of India Limited (SAIL) has led to successful development of atmospheric and marine corrosion resistant high tensile Thermo Mechanically Treated (TMT) bars with minimum specified yield strength of 50 kg. mm-2. These rebars are increasingly being used for a wide range of end-applications, namely, concrete reinforcement structures, bridges, flyovers, dams etc. Welding is an important processing tool for each of the above applications. In view of the importance of welding, a comprehensive evaluation of the weldability properties has been carried for the two grades of steels using Submerged Metal Arc Welding (SMAW) process; the most popularly used technique of welding of reinforcement bars (rebars). Implant tests were conducted to assess the cold cracking susceptibility of the steels. The static fatigue limit (SFL) value, determined for the atmospheric corro-

sion resistant (Cu-TMT) rebars under no preheat and no rebaking conditions was found to be 63.8 kg mm-2, which was significantly higher than the Minimum Specified Yield Strength (MSYS) (50 kg.mm-2). This indicates adequate resistance to hydrogen induced cracking (HIC). On the other hand, the SFL value obtained for the marine corrosion resistant rebars (Cu-Cr TMT) under no preheat, no rebaking conditions was found to be inconsistent. Repeat of the test with fully rebaked electrodes (350°C-2h) resulted in a high SFL value (60.3 kg mm-2). The rigidity of the structure or restraint influences the susceptibility of the weld joint to cracking. The cracking tendency increases with restraint intensity (K) of an assembly. The critical restraint intensities determined for the two steels were found to be in excess of 1680 kg/mm.mm, indicating good welding behaviour with the following welding conditions; for Cu-TMT rebars with no preheat and no rebaking and for Cu-Cr TMT rebars with no preheat and fully baked electrodes; that is under low hydrogen levels. Based on the weldability tests, the optimized conditions for welding were formulated. Extensive tests carried out on the welded joints indicated adequate strength, toughness and internal soundness for both the atmospheric and marine corrosion resistant TMT bars. Based on the results of the above tests, it was concluded that the weld joints ensure a high integrity of the fabricated structures.

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MATRIX MICROSTRUCTURAL EFFECTS ON THE DUCTILITY AND FRACTURE TOUGHNESS OF PARTICULATE-REINFORCED ALUMINUM ALLOY COMPOSITE: *A.B. Pandey*[1]; B.S. Majumdar[2]; D.B. Miracle[3]; [1]Systran Corporation, Dayton, 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. However, lower ductility and fracture toughness of DRAs impose constraints for using these materials in aerospace structures. Despite significant studies available on the fracture aspects of DRA, a clear understanding of fracture micromechanism is still lacking. The purpose of this study is to evaluate the influence of matrix microstructure on the ductility and fracture toughness of a DRA, 15 vol.% SiCp/7093 Al, processed using a powder metallurgy technique. In this study, tensile and fracture toughness, Jlc, tests using ASTM B-813 standard were performed on DRA in different heat treatment conditions. The ductility and toughness varied inversely as the strength being highest in the solution treated and highly-overaged conditions, and the lowest in the peak—aged condition. Damage in the tensile and CT specimens a head of crack tip were analyzed to provide an insight into the damage micromechanisms. Damages in the form of particle fracture and interface and bonding were observed in all the heat treatment conditions. Existing models are unable to explain the observed dependence of ductility and fracture toughness on the strength. Modeling efforts to explain such a behavior will be discussed.

INTERNATIONAL SYMPOSIUM ON SULFIDE SMELTING '98: CURRENT AND FUTURE PRACTICES: Session II - Copper Smelting - Smelter Operations

Sponsored by: Extraction & Processing Division, Copper, Nickel, Cobalt Committee

Program Organizers: Tony Eltringham, BMP Copper, 550 California St., San Francisco, CA 94104-1020; Jussi Asteljoki, Outokumpu Oy, Lansituulentie 7, Espoo Fin-02101 Finland; Pete W.J. Chen, Phelps-Dodge Mining Co., P.O. Box 2860, Silver City, NM 88062; Osamu Ishikawa, Bechtel Corp, Mining & Metals, San Francisco, CA 94119-3965; Robert L. Stephens, Ausmelt Technology Corp., 1331 17th St., Denver, CO 80202; Courtney Young, Montana Tech, Metallurgical Engineering, Butte, MT 59701

Monday PM Room: 207
February 16, 1998 Location: Convention Center

Session Chairs: W. J. (Pete) Chen, Phelps Dodge Mining Company, Silver City, NM 88062; Markku Kytö, Outokumpu Technology, Espoo FIN-02210 Finland

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FLASH SMELTING BEHAVIOR OF VARIOUS COPPER CONCENTRATES IN A PILOT SCALE FURNACE: Nobumasa Kemori[1]; *Yasuhiro Kondo*[1]; Keiji Fujita[1]; [1]Pyrometallurgical Research Center (P.R.C.), Niihama Research Laboratories, Sumitomo Metal Mining Co., Ltd., Ehime 793 Japan

Six kinds of copper concentrates were treated individually in a pilot scale Outokumpu furnace in order to study their flash smelting behavior such as oxygen efficiency and dust generation. The tested copper concentrates were selected from more than twenty sources which are treated at the Sumitomo Toyo copper smelter, so as to have a wide range of chemical compositions in terms of Cu, S, Fe, Pb + Zn and SiO2 contents. Oxygen efficiency and dust generation obtained in the experiments ranged from 80 to 108% and from 12 to 20% respectively. Relation between these results and such experimental conditions as chemical and mineral composition of the copper concentrates, flux ratio, fuel consumption and so on was also discussed quantitatively in this paper.

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THERMODYNAMIC INVESTIGATIONS OF PYROMETALLURGY OF SULFIDE CONCENTRATES: *Dr. Vladimir Y Mindin*[1]; Dr. Natela Kiknadze[2]; Mr. Yakov V. Mindin[3]; [1]AFP, Manhasset, NY 11030 USA; [2]Georgian Technical University, Tbilisi, Georgian Republic; [3]J. F. Jelenko & Co., Armonk, NY 11030

The report contains results of thermodynamic investigations (TI) and direct experimental studies (ES) of some essential for pyrometallurgy of sulfide concentrates (SC) topics. Among them: 1. TI and ES of SC roasting process 2. TI of sulfur dioxide off-gases treatment technologies 3. TI and ES of "Mindin-Kiknadze" effect of suppression of sulfur oxide generation during SC roasting in the vapor containing atmosphere 4. TI and ES of SC co-roasting processes with manganese containing materials

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RECENT OPERATIONS AT THE ATLANTIC COPPER SMELTER IN HUELVA: Patricio Barrios[1]; Jesus Contreras[1]; *Miguel Palacios*[1]; [1]Atlantic Copper S.A.; Huelva Spain

In February 1996, Atlantic Copper completed the construction of a Project for Expansion and Environmental Improvements in its Smelter and Electrolytic Refinery located in Huelva. Following the expansion, the new operating concept is based on the simultaneous blowing of two

converters, increasing throughput in the Flash Furnace up to the level required to deliver matte to both converters. The production level planned with the Project (270,000 mtpa of copper from concentrates) was reached the following June, and since then work has been carried out to optimize the new equipment and to coordinate the new operating schedule. The present paper describes operational experience from the start-up of the Expansion Project, difficulties encountered and operating improvements introduced as well as the debottlenecking project that will be completed by mid-1997 and will allow an increase of production levels to over 300,000 mtpa of copper.

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CURRENT TENIENTE CONVERTER PRACTICE AT SOUTHERN PERU'S ILO SMELTER: William E. Torres[1]; [1]Southern Peru Limited Ilo Smelter, Miami, FL 33126-1032

The Southern Peru Limited smelter in Ilo, Peru, operates two reverberatory furnaces, seven Peirce Smith converters, one Teniente Converter, two casting wheels and a single contact acid plant. The Teniente Converter was commissioned in August 1995 and was designed to smelt 744 dry metric tonnes (820 dry short tons) of concentrate per day. The process off-gas is collected in a water-cooled hood, cooled in a spray chamber and cleaned in a Joy electrostatic precipitator for further treatment in the Acid Plant. This paper presents the current Teniente Converter operating practices and control philosophy. Relevant improvements and modifications to achieve a 379 day campaign without tuyere replacement and a daily average smelting rate of 862 dry metric tonnes (950 dry short tons) of concentrate will be discussed.

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OPERATION OF THE CONTOP PROCESS AT THE ASARCO EL PASO SMELTER: *Martin Brueggemann*[1]; Eric Caba[1]; [1]ASARCO Inc., El Paso, TX 79999

In 1993, the El Paso Smelter began operation of the first commercial CONTOP copper smelting furnace. The CONTOP process was chosen for the plant modernization due to its low capital cost and the ease of incorporating the process into a 100-year-old existing smelter. After four years of operation, CONTOP has proved itself to be a reliable copper smelting technology. This paper will discuss a brief start-up history, current operations, some advantages and disadvantages, and future modifications of the CONTOP process at El Paso.

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GAS RECIRCULATION AND ENDOTHERMIC DISSOCIATION OF SULPHUR TRIOXIDE FOR SMELTIG HIGH ENERGY SULPHIDES WITH TECHNICALLY PURE OXYGEN: *N. A. Warner*[1]; [1]The University of Birmingham, School of Chemical Engineering, Birmingham D 15 2TT United Kingdom

To fully secure the environmental and social acceptability benefits of making a sulphide smelting facility and its ancillary sulphuric acid plant or other means for fixing sulphur, a virtually zero gas emission operation, it will be necessary to utilize exclusively technically pure oxygen. The strategy proposed for preventing the smelter from overheating involves highly efficient energy transfer by promoting sulphur trioxide formation at a relatively higher temperature than normal. A closed-loop gas circuit uses an educator system actuated by the process oxygen to recirculate sulphur trioxide and unconverted sulphur dioxide back to the smelter, the endothermic dissociation of sulphur trioxide becoming a major contributor to the removal of excess heat. The only gases leaving the system relate to the bleed of nitrogen impurity introduced in the tonnage oxygen.

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LOW TEMPERATURE CHLORINATION OF CHALCOPYRITE CONCENTRATES AND RECOVERY OF ELEMENTAL SULPHUR CHLORIDES: *I. Gaballah*[1]; N. Kanari[1]; B. Allain[1]; J. C. Mugica[2]; [1]Mineral Processing and Environmental Engineering, Cedex 54501 France; [2]Inasmet, San Sebastian 20009 Spain

Chalcopyrite concentrates and their pure constituents were chlorinated by Cl2 + N2 between 25 and 750ºC. The reaction products were analyzed by SEM, XRD and chemical analysis. The effects of gas flow rate, chlorine content of the gas mixture and time on the reaction rate were investigated. At room temperature, these concentrates start to react with chlorine giving metal and sulfur chlorides. Complete reaction is achieved at about 300ºC and the iron and sulfur chlorides were fully volatilized. The chlorination residue is composed of valuable metal's chlorides. A simplified flow-sheet for the treatment of chalcopyrite concentrates by chlorine is suggested.

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INTRODUCTION OF RELIABILITY CENTERED MAINTENANCE IN A COPPER SMELTER: *Rey B. Marquez*[1]; [1]Phelps Dodge Hidaldo, Inc, Hidalgo Smelter, Playas, NM 88009

This presentation introduces and outlines the implementation of Reliability Centered Maintenance (RCM) at Phelps Dodge-Hidalgo. By using a disciplined approach to the everyday maintenance functions - a proactive strategy is being developed which "raises the bar" to plant maintenance. Main objectives are: integration of Operations and Maintenance partnerships and transformation of maintenance practises from Reactive to Proactive made through progressive Predictive /Preventive Maintenance program (PDM/PM) paradigm changes increased management support of maintenance function, introduction of new indices and root cause analysis, updated training, definition of RCM components. RCM is the vehicle being utilized to reach the higher level of equipment availability and utilization, however, the ultimate goal is to venture beyond RCM and into the new frontier of Total Maintenance Management (TMM).

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KENNECOTT UTAH COPPER SMELTER MODERNIZATION: *Chris J. Newman*[1]; Tony G. Weddick[1]; [1]Kennecott Utah Copper Corporation, Magna, Utah 84044-6001

Prior to 1995, the Kennecott Utah Copper Corporation (KUCC) Smelter incorporated Noranda reactors and Peirce-Smith converter. To meet expansion opportunities and conform to sulfur fixation requirements imposed by the State of Utah, a number of smelting options were evaluated. The final selection was the Outokumpu flash smelting process, coupled with Kennecottt-Outokumpu flash converting technology. The facility was designed to process 1.1 million tons per year of copper concentrate wit possible expansion to 1.3 million tons. The plant was commissioned in the summer of 1995. This paper outlines the design, construction, start-up and first two years of operation of the modernized smelter.

INTERNATIONAL SYMPOSIUM ON VALUE ADDITION METALLURGY: Session II - Specialty Materials

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee

Program Organizers: W.D. Cho, University of Utah, Dept. of Metallurgical Eng., Salt Lake City, UT 84112; H.Y. Sohn, University of Utah, Dept. of Metallurgical Eng., Salt Lake City, UT 84112

Monday PM Room: 208
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Session Chairs: C. Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya Japan; Michael B. Mooiman, Metalor USA Refining Corporation, North Attleboro, MA USA

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REMOVAL OF OXYGEN FROM NEODYMIUM BY HALIDE FLUX TREATMENT: *Hiroyuki Sano*[1]; Masami Tashiro[1]; Toshiharu Fujisawa[2]; Chikabumi Yamauchi[1]; [1]Nagoya University, Dept. of Materials Science and Engineering, Graduate School of Engineering, Nagoya 464-01 Japan; [2]Nagoya University, Research Center for Advanced Waste and Emission Management, Nagoya Japan

Rare earth metals usually contain large amount of oxygen because of their reactiveness, and therefore oxygen removal is one of the major problems on the purification of rare earth metals. In the present research work, halide flux treatment at high temperature under inert gas atmosphere was applied to the oxygen removal from neodymium. NdF_3 was used as halide flux. Deoxidization reaction and its mechanism were discussed based on the experimental results. Halide flux treatment is very effective for oxygen removal from rare earth metals.

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MICROSTRUCTURAL CONTROL AND MAGNETIC PROPERTIES OF Nd-Fe-B PERMANENT MAGNETS: *Toshiro Kuji*[1]; Masakazu Fujita[1]; Tetsuji Saito[2]; [1]Mitsui Mininng and Smelting Co., Ltd., Corporate R&D Center, Saitama 362 Japan; [2]Chiba Institute of Technoloy, Chiba 275 Japan

The magnetic properties of the permanent magnets are strongly dependent on the microstructure. In this study, we are demonstrating a novel process for microstructural control of Nd-Fe-B alloys to obtain excellently high-energy products, as shown below: 1)Nd-Fe-B amorphous powders, obtained by rapid solidification were consolidated into bulk shape without loosing the amorphous state by dynamic compaction. 2) The bulk amorphous alloys were crystallized under the existence of plastic deformation (die-upsetting). The resultant Nd-Fe-B magnets had an unique morphology, i.e., crystallographically aligned Nd2Fel4B grains with polygonal shape. The compositionally modified Nd-Fe-Co-Ga-B magnets had an excellently high energy-products, (BH)max-54 8MGOe.

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THERMODYNAMICS OF THE Si-B-N SYSTEM: *Mitsuru Tanahashi*[1]; Toshiharu Fujisawa[2]; Chikabumi Yamauchi[1]; [1]Nagoya University, Department of Materials Science and Engineering, Graduate School of Engineering, Nagoya, 464-01 Japan; [2]Nagoya University, Research Center for Advanced Waste and Emission, Nayoga Japan

Thermodynamic properties of impurities in, molten silicon are of prime importance, in developing economical production methods of solar grade silicon. In the present research work, thermodynamic properties of boron, the most difficult element to be removed, in molten silicon were measured by chemical equilibration techniques in the temperature range of 1723 to 1773 K. Activity coefficients and self-interaction parameters of boron in molten silicon were determined by equilibrating the melt with solid BN under controlled nitrogen partial pressures. Interaction parameters between boron and nitrogen in molten silicon were also determined hy equilibrating the molten Si-B alloys with solid Si_3N_4. Based on the data obtained, the stability diagram of the SiB-N system was established.

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COST EFFECTIVE CLEANER TECHNOLOGY FOR UPGRADING ZINC QUALITY: *P. Tait*[1]; N. Warner[1]; [1]University of Birmingham, School of Chemical Engineering, Edgbaston B15 2TT UK

The refining of thermally produced zinc is currently performed using a highly energy intensive and antiquated process, distilling zinc under atmospheric pressure at temperatures of 900°C to 1000°C. In order to produce refined zinc more profitably and with less environmental impact, a new process has been proposed. This process is generic to all thermal zinc smelters and aims to match the purity of zinc from current technology (99.999%). The objective is to perform all separation processes using only the heat contained in the smelter product stream. Vacuum conditions are extensively used lowering operating temperatures and enhancing separations. As part of this process zinc is distilled under vacuum from high concentration obtaining a refined product with respect to lead content. A mathematical model has been developed from fundamental principles to describe this stage, the distillation of zinc across a temperature difference between the surfaces of two circulating zinc streams under vacuum. Heat for the distillation is supplied by the liquid phase, necessitating a large recycle to maintain high operating temperatures, and keep zinc concentrations high. In determining this model and the operating conditions required, relationships between temperature, pressure and composition are considered.

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STOICHIOMETRIC ANALYSIS OF MgSiO4 AND Y2SiO5 SINGLE CRYSTALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY: *Kayoko Shinzawa*[1]; Katsunori Takemur[1]; Sakiko Taenaka[1]; Mitsuyoshi Ide[1]; [1]Mitsui Mining and Smelting Co., Ltd., Corporate R&D Center, Saitama 362 Japan

The laser characteristic is strongly dependent upon the stoichometry of MgSiO4 and Y2SiO5 single crystals, because of change in the valency and occupied site of doped Cr in the lattice so that mole ratios of MgO/SiO2 and Y2O3/SiO2 in the above single crystals must be precisely analyzed. In this study ICP-AES was applied for analyzing the mole ratios. Emission intensities corresponding to Si and Mg or Si and Y were measured at the same time witninner standard method. Preparation of sample solutions, measurement conditions of ICP-AES and interferences by coexisting components were discussed, resulting in excellently precise determination of stoichiometry of the above single crystals.

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SEPARATION OF CERIUM FROM SULFURIC ACID LEACHED SOLUTION OF BASTNASITE BY ION-SIEVE METHOD: *Ho-Sung Yoon*[1]; Sung-Don Kim[1]; Chul-Joo Kim[1]; Joon-Soo Kim[1]; [1]Korea Institute of Geology, Mining and Materials, Division of Minerals Utilization and Materials, Doejon Korea

This study was performed to establish the optimum leaching condition through the experiments for the oxidized roasting followed by leaching of fluorocarbonate rare earth mineral (bastnasite) by using sulfuric acid, and to establish the optimum condition for the recovery of cerium by ion-sieve method from the leached solution of bastnasite. The optimum conditions for the sulfuric acid leaching by using the oxidizing roasting of bastnasite concentrates are that the roasting temperature is 550~600°C, the roasting time is 2 hrs, the concentration of sulfuric acid is 6N, the pulp density is 20%, the leaching temperature is 50°C and the leaching time is 3 hrs. At this time, the leaching yield of total rare earth oxides in the leaching of bastnasite concentrates is about 91%. When cerium is recovered by ion-sieve method from the solution obtained by the sulfuric acid leaching of the roasted bastnasite, the optimum conditions are that the TREO of leaching solution is 70~80 g / γ, the flow rate of solution is 0.49 cm/min and the ratio of height to diameter of column is above 9. In these conditions, the recovery of cerium of yield 85% and quality 96% is possible.

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THE PRODUCTION AND USE OF ULTRA HIGH PURITY PRECIOUS METALS - A REVIEW: *Michael B. Mooiman*[1]; David J. Kinneberg[1]; Leo Simpson[1]; [1]Metalor USA Refining Corporation, North Attleboro, MA USA

Ultra high purity precious metals are those that have a purity greater than 99.99%. The various refining processes used for the production of ultra high purity silver, gold, platinum and palladium are reviewed. These processes are largely hydrometallurgical in nature although combinations of hydrometallurgy and pyrometallurgy are sometimes used. The applications and necessity for these very high purity metals are discussed. Some the challenges involved in working with these materials while maintaining purity are outlined.

LITHIUM: Properties of Lithium and Its Compounds

Sponsored by: Light Metals Division, Refractory Metals Committee
Program Organizers: Donald R. Sadoway, MIT 8-109, Cambridge, MA 02139-4307; Georges J. Kipouros, University of Nova Scotia, Dept. of Mining & Met. Eng., Halifax, NS B3J2X4 Canada

Monday PM Room: 209
February 16, 1998 Location: Convention Center

Session Chairs: Donald R. Sadoway, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307; George J. Kipouros, University of Nova Scotia, Halifax, NS B3J2X4 Canada

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THERMODYNAMIC MODEL FOR CHLORIDE MELTS AND APPLICATION IN THE PRODUCTION OF LITHIUM: *Prof. William T. Thompson*[1]; Dr. Boyd R. Davis[1]; Dr. Olivo Sivilotti[2]; [1]Royal Military College of Canada, Centre for Research in Computational Thermochemistry, Kingston, ON K7K 5L0 Canada; [2]Sigma Tau Technologies Inc., Kingston, ON K7M 1P1 Canada

Extensive experimental thermodynamic investigations and data treatment for molten chloride electrolytes originally aimed at magnesium electrolysis have resulted in thermodynamic properties for all components in the molten phase of the LiCl-KCl-NaCl-CaCl2 system. These data when used with related properties for other species and phases permit a detailed thermodynamic analysis of such matters as Li contamination with Na and Ca, crystallization of the melt, loss of vapour species, hydrolysis of calcium chloride, chlorination of contaminant oxides, possible purification processes, etc. The presentation will illustrate the use of the data in conjunction with F*A*C*T (Facility for the Analysis of Chemical Thermodynamics). This system co-developed by the first author is a user friendly software suite with databases enabling a wide range of metallurgically important computations to be performed with ease.

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LITHIUM-METAL OXIDES FOR RECHARGEABLE Li BATTERIES: THE ROLE OF STRUCTURE AND CHEMISTRY: *Prof. Gerbrand Ceder*[1]; Dr. Kadri M. Aydinol[1]; Mr. Anton Van der Ven[1]; [1]MIT, Dept. of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Lithium-metal oxides have been tested with almost any 3d metal for potential application in rechargeable Li batteries. Although the properties of these materials are now well characterized, a clear relation between the structure, chemistry and resulting properties has not yet been established. We show how computational modeling can be used to investigate this relation. Using first-principles methods to calculate the electrochemical potential at which Li intercalates, we can systematically investigate which effect structure and chemistry each have on the battery voltage. These results have led to a set of design criteria for new, higher energy density materials. Experiments on these materials will also be shown.

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STABILIZATION OF LiMnO2 IN THE ALPHA-NaFeO2: Y-I. Jang, B. Huang, Y-M. Chiang, D. R. Sadoway; Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139-4307

LiMnO2 of the alpha-NaFeO2 structure type has previously been obtained only by the ion-exchange of lithium salts with alpha-NaMnO2. We have found that LiAl (x) Mn (1-x) O2 solid solutions crystallize in this structure under conditions where neither end member does. The compounds were synthesized by firing homogeneous hydroxide precursors in a reducing atmosphere to control the manganese valence state. These powders show excellent reversible capacity (148) mAh/g) when cycled over both the 4 V and 3 V plateaus.

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THE EXTRACTION OF LITHIUM CARBONATE FROM A PEGMATITE DEPOSIT IN MANITOBA, CANADA: *Prof. Werner Dresler*[1]; Mr. B. C. Jena[1]; Mr. I. G. Reilly[1]; [1]Laurentian University, School of Engineering, Sudbury, ON P3E 2C6 Canada

An ore sample from a pegmatite deposit at Wekusko Lake in northern Manitoba contained 1.7% Li2O, Scanning electron microscope analysis showed that lithium is present as spodumene at a concentration of 7.4% Li2O with 0.9 to 1.6% Fe2O3. Potassium and sodium oxide concentrations in the spodumene are relatively low, 0.01% K2O and 0.21% Na2O. Major minerals associated with the spodumene are Na-feldspar, K-feldspar, and quartzite. Muscovite, apatite, and garnet are present at low concentration. Because of the relatively high iron content, the spodumene can be utilized in the glass and chemical industries. The extraction of Li2CO3 from the ore is described, comprising froth flotation, heat treatment at 1100°C of the spodumene concentrate, sulphuric acid roasting, and solution purification. The final product contained above 98% Li2CO3.

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REVIEW OF LIQUID LITHIUM CORROSION OF CONTAINMENT MATERIALS: *Prof. David L. Olson*[1]; Prof. Brajendra Mishra[2]; [1]Colorado School of Mines, Department of Metallurgical & Materials Engineering, Golden, CO 80401-1887 USA; [2]Colorado School of Mines, Kroll Institute for Extractive Metallurgy, Golden, CO 80401

An overview of experimental observations and results of liquid lithium corrosion of engineering materials will be presented. Liquid lithium corrosion behavior in both stationary and flowing streams will be described. The nature of the degradation and its mechanism will be explained. The influence of temperature, microstructure, stress, impurities and service time on the corrosion behavior for various engineering alloys will be discussed. The state of the mechanistic understanding of the corrosion process will be given. Selection rules for materials of containment for liquid lithium shall be suggested.

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A STUDY OF THE PRESSURE LEACHING PROCESS FOR THE CALCINE OF LEPIDOLITE BY AMMONIUM CHLORIDE: *Dr. Shengming Xu*[1]; [1]Hunan University, College of Chemistry and Chemical Engineering, Changsha, Hunan 410082 China

The process for ammonium chloride pressure leaching of the calcine of lepidolite has been studied. The influences of temperature, time, ratio of liquid to solid, the amount of NH4Cl, etc., on the leaching recovery of lithium were investigated, and a mathematical model was constructed. The optimal conditions were chosen, and verification experiments of the model demonstrated that leaching recovery could exceed 91%. The model has practical significance for the choice of technologies control and predict of the process.

MATERIALS PROCESSING FUNDAMENTALS: Materials

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee

Program Organizers: Patrick R. Taylor, University of Idaho, Dept. of Met & Mining Eng., Moscow, ID 83843-3024; Srinath Viswanathan, Oak Ridge National Lab, PO Box 2008 Bldg. 4508, Oak Ridge, TN 37831-6083

Monday PM Room: 206
February 16, 1998 Location: Convention Center

Session Chair: Dr. Patrick R. Taylor, McClure 203, University of Idaho, Moscow, ID 83844-3024

2:00 PM

MICROSTRUCTURAL REFINEMENT/CONTROL AND STRENGTH ENHANCEMENT OF Cu-Cr-Nb (Cu-Cr2Nb) ALLOYS BY MECHANICAL MILLING: K. R. Anderson[1]; Don G. Ulmer[2]; Beverly J.M. Aikin[3]; D. L. Ellis[3]; *J. R. Groza*[1]; [1]University of California-Davis, Chem Engrg & Matls Sci Dept, Davis, CA 95616 USA; [2]Boeing North American, Rocketdyne Division, Canoga Park, CA 91303 USA; [3]NASA-Lewis Research Center, Cleveland, OH 44135 USA

Recently, Cu-Cr-Nb alloys with the Cr:Nb ratio of 2:1, developed by NASA, have received special attention as the next generation high strength-high conductivity dispersion-strengthened (DS) alloys. The present study deals with processing steps to further enhance mechanical properties of two such alloys: Cu-8 Cr-4 Nb and Cu-4 Cr-2 Nb. Current experimental work involves the mechanically milling/alloying (MM/MA) of as-atomized pre-alloyed Cu-Cr-Nb powders; copper, chromium and niobium starting powders; and finally, just copper. The milled powders are consolidated by hot pressing and extrusion. Behind this approach is the need and expectation for providing further insight into second-phase intermetallic particle breakdown/redissolution, incorporation and formation in a Cu metal matrix utilizing severe plastic deformation via MA/MM; and their relative effects on strength, (electrical) conductivity and thermal stability. Microscopy (optical, SEM and TEM) should show that there is not only a definite refinement in Cu grain size, but also a refinement in both size and distribution of the large Cr2Nb precipitates. Strength (hardness) and conductivity will be correlated with microstructural refinement due to MM/MA. Electrical conductivity measurements are also used to determine the effects of MM on electrical conductivity as compared to non-milled/hot pressed, as well as as-extruded Cu-Cr-Nb. Electrical conductivity has shown a better correlation with extent of deformation, or microstructural refinement, than impurity pickup, at least for longer milling times. (Results of the current experiments, which are carried out with an attritor mill may be compared with earlier similar experiments where a SPEX mill was used.) Strength/conductivity combination optimization via microstructural control and analysis is an essential part of the overall goal of this present study.

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COMPUTATIONAL SIMULATIONS AND EXPERIMENTAL VALIDATION OF A FURNACE BRAZING PROCESS: *F. M. Hosking*[1]; S. E. Gianoulakis[1]; [1]Sandia National Labs, Albuquerque, NM 87185 USA

Modeling of a furnace brazing process is described. The computational tools are being used to predict the thermal response of loaded hardware in a hydrogen brazing furnace to a programmed furnace profile. Experiments were conducted to validate the model and resolve computational uncertainties. The results from selected furnace simulations and measured thermal responses are compared. Critical boundary conditions that affect the materials and processing response to the furnace environment are identified. “Global” and local issues (i.e., at the furnace/hardware and joint levels, respectively) are discussed. The ability to accurately simulate and control furnace conditions is considered. 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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FORMATION OF TiB BY MECHANICAL ALLOYING: *Sedat Özbilen*[1]; [1]Gazi University, Metal Dept., Ankara Turkey

A mixture of TiH_2 and B powders in stoichiometric proportion were mechanically alloyed under Ar to synthesize TiB compound in the form of powder. Milled powder were examined by X-Ray Diffraction and Scanning Electron Microscopy to determine the feasibility of MA in the production of intermetallic compounds such as TiB as investigated in the present study.

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THE COMPUTER ANALYSIS THERMAL-MECHANICAL-CHEMICAL PROCESSES IN PRODUCTS OF THE IRREGULAR SHAPE BASED ON ORTHOGONAL CONFORMAL TRANSFORMATIONS OF COORDINATES: *Alexey Lozhko*[1]; [1]State Metallurgical Academy of Ukraine, Dept of Heat Tech & Enviro Engrg, Dniepropetrovsk UA-320635 Ukraine

It is necessary to take into account thermal stresses at intensive processes of heat treatment of products. It is especially important for products of the irregular form, when identical rate of cooling in all volume of a product is impossible, because its separate parts have different thermal masses. There are some undesirable consequences of high level thermal stresses. The product can change the form during the heat treatment and has residual stresses after it. The residual stresses can create problems at the subsequent machining of a product. An original mathematical apparatus based on orthogonal conformal transformations has been used for the simulation of the non-stationary temperature field. This enabled to solve the problem with higher speed and accuracy in comparison with such methods as finite or bottleneck elements. Experimental part contains examples of successful choice of technology of heat treatment, which minimize residual stresses.

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GAS PHASE REACTIONS BY ELECTRON BEAM PROCESSING: *Patrick R. Taylor*[1]; [1]University of Idaho, Moscow, ID 83844-3024 USA

This paper describes several new areas of application for electron beam processing of materials. The technology and use of electron beam systems in gas phase synthesis, activated sintering and surface modifications are described. Gas phase reactions may be used to generate ultrafine metal or ceramic powder that are very pure and crystalline. Among the examples presented are: the use of electron beams for activated sintering of pressed metal or ceramic parts; the gas phase synthesis of silicon carbide for silica and methane; and vacuum deposition of thin film coatings. The experimental system is described and future theoretical and experimental objectives are identified.

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RHEOLOGY OF POWDER INJECTION MOULDED PARTS: *Dr. Sedat Özbilen*[1]; [1]Gazi University, Metal Dept., Ankara Turkey

The determination of the variation of pressure against time for PIN feed-stocks during their stay in the mould has gained importance in PIN technology, a hot research area of recent years. The possibility of this will case the control of properties of the parts that will be produced with this technology, such that the conditions to increase the productivity of the process can be realized. In the present work, the rheology of 316L stainless steel powder-variable binder mixes were therefore studied. Results of this investigation will be presented.

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SYNTHESIS OF TITANIUM NITRIDE IN A THERMAL PLASMA REACTOR: *Dr. Patrick R. Taylor*[1]; Wenxian Zhu[1]; Edgar E. Vidal[1]; P. V. Ananthapadmanabhan[2]; [1]University of Idaho, Dept of Met & Min Engrg, Moscow, ID 83844-3024 USA; [2]Bhabha Atomic Research Centre, Laser & Plasma Technology Division, Mumbai 400 085 India

Titanium nitride has been synthesized in a thermal plasma reactor from ilmenite ore concentrate using methane and ammonia as the reactive gases. The product has been characterized by x-ray diffraction and SEM. Results show that a high concentration of methane favors the formation of iron, titanium nitride and titanium carbonitride. The formation of titanium carbonitride can be minimized by controlling the concentration of methane. The formation of TiN proceeds by the progressive reduction of the oxides of titanium to titanium, which is converted to titanium nitride.

MICROSTRUCTOLOGY CONNECTING PHASE DIAGRAMS KINETICS & STEREOLOGY TO MICROSTRUCTURAL EVOLUTIONS: F.N. RHINES: Stereology and Microstructural State

Sponsored by: ASM International: Materials Science Critical Technology Sector, Atomic Transport Committee
Program Organizers: Robert T. DeHoff, University of Florida, Dept. of Materials Sci & Eng., Gainesville, FL 32611-6400; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Monday PM Room: 103
February 16, 1998 Location: Convention Center

Session Chair: J. C. Zhao, GE Corporate R&D Laboratory, Schenectady, NY 12301

2:00 PM INVITED
MOTION BY WEIGHED MEAN CURVATURE AS SEEN FROM ANY ANGLE: *Dr. Jean E. Taylor*[1]; [1]Rutgers University, Mathematics Department, Piscataway, NJ 08855 USA

Suppose one observes isotropic thin film grain boundary motion (motion by curvature multiplied by a mobility factor), but one looks at the plane of the film from an angle so that circles appear to be ellipses. In this case, apparent ellipses would shrink as ellipses to points, and the surface energy would appear to be anisotropic as would the mobility. It doesn't matter: if one uses the apparent surface energy and the apparent mobility, then the motion by weighted curvature with mobility is the same as if one used isotropic energy and mobility in the original plane but then viewed the result from the angle. The same is true for surfaces in 3-space and for initially arbitrary surface energy functions and mobilities. In case that the surface energy function is at least twice differentiable, it even applies to the case of motion viewed through distorted lenses (i.e., diffeomorphisms). This result is to be contrasted with the result which states that for area-preserving affine transformations of the plane where the energy and mobility are NOT also changed, motion by curvature to the power 1/3 (rather than 1) is invariant.

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EXTRACTION OF FREE ENERGIES AND MOBILITIES OF GRAIN BOUNDARIES FROM THE GEOMETRY OF MICROSTRUCTURES: Prof. Brent L Adams[1]; Prof. David Kinderlehrer[2]; Prof. William W Mullins[1]; *Prof. Anthony D Rollett*[1]; Prof. Shlomo Ta'asan[2]; [1]Carnegie Mellon University, Materials Science & Engineering, Pittsburgh, PA 15213-3890 USA; [2]Carnegie Mellon University, Dept. of Mathematical Sciences, 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. 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 uses a novel multiscale statistical approach and provides relative energies (mobilities) over the entire fundamental zone. Calibration of the energies requires at least one absolute measurement of boundary energy for a known boundary type. 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.

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QUANTITATIVE CHARACTERIZATION OF SPATIAL ARRANGEMENT OF MICROSTRUCTURAL FEATURES: Mr. Asim Tewari[1]; *Dr. Arun M Gokhale*[1]; [1]Georgia Institute of Technology, Materials Science and Engineering, Atlanta, GA 30332 USA

Most of the previous research on quantitative metallography concerns estimation of metric and topological properties of microstructural features. In the past, very little attention has been paid to quantification of spatial arrangement of features in microstructures, in terms of the descriptors such as nearest neighbor distribution and higher order neighbor distributions, radial distribution function, etc. This contribution presents application of digital image analysis and processing techniques for characterization of spatial arrangement of microstructural features. The methodology will be illustrated through experimental data on porosity in cast microstructures, and tungsten grains in liquid phase sintered WHA alloy.

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THREE DIMENSIONAL RECONSTRUCTION OF PROEUTECTOID PRECIPITATES IN ALLOY STEELS: *Dr. Milo Kral*[1]; Dr. G. Spanos[1]; [1]Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375-5000 USA

A three dimensional reconstruction technique has been applied to quantitatively characterize the three dimensional morphologies of ferrite and cementite precipitates in isothermally transformed alloy steels. This method involves the use of serial sectioning with very fine material removal increments, high resolution optical microscopy and advanced computer visualization techniques. Used in conjunction with electron microscopy, the three dimensional reconstruction technique can provide a complete and quantitative description of all of the precipitate morphologies in an alloy. The present results are compared to earlier morphological classification systems for proeutectoid precipitates in alloy steels.

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STEREOLOGICAL CHARACTERIZATION OF SERRATED GRAIN BOUNDARIES IN NICKEL-BASE SUPERALLOYS: *Mr. Ahmed M Qurashi*[1]; Dr. Robert T DeHoff[2]; [1]Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332 USA; [2]University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611 USA

Serrated grain boundaries form during processing of some IN-100 derived nickel based superalloys. In such a structure the grain boundary network can be viewed as consisting of serrated and unserrated areas. Triple lines (grain edges) in this system fall into four classes, depending upon the nature of the three grain boundaries that meet there: sss; ssu; suu and uuu, where "s" refers to a serrated segment and "u" an unserrated segment of adjacent grain boundary area. Computer-assisted stereology performed on digitized optical images provided unbiased estimates of the surface area of serrated and unserrated boundary regions, and the total length of each of the four classes of triple points. An analysis of fractions of each of these measurements provided insight into the connectedness of the serrated.

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MICROSTRUCTOLOGY OF MUSHY ZONES: *Dr. Martin E. Glicksman*[1]; [1]Rensselaer Polytechnic Institute, Materials Science and Engineering Department, Troy, NY 12180-3590 USA

Mushy zones represent cast microstructures in a formative state, when the processes of net solidification, phase coarsening, micro-convection, and alloy segregation are all ongoing. Microstructology provides an effective way to follow the microstructural pathways obeyed by such systems. Indeed, microstructological data now provide inputs into macro-codes that, originally, were limited to tracking the large-scale behavior of castings and ingots, such as their macroscopic heat transfer and fluid flow. Such approaches attempt to incorporate some microstructural detail, occurring on small scales, into the process macro-codes. Examples will be discussed in which alloy solidification and liquid-phase sintering were studied using microstructology. Deeper insight is still being sought to merge microstructology methods with fundamental solidification scaling laws to learn the "rules behind the rules," as to how and why cast structures evolve the way they do.

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A STEREOLOGICAL APPROACH TO MICRO-MACRO MODELING OF SOLIDIFICATION PHENOMENA: *Dr. Steve Marsh*[1]; [1]Naval Research Laboratory, Washington, DC 20375-5343 USA

A critical challenge in applied solidification modeling involves the quantitative representation of phenomena that occur on widely disparate length scales. A formalism has been developed to integrate microscopic models with macroscopic field equations in a computationally efficient manner. This approach uses stereological parameters to quantify the geometric characteristics of the developing microstructure, which serve as average conditions over a timestep within the local unit volume of material associated with each computational node. Kinetic models and balance equations are applied to geometrical constructs that represent the characteristic local morphology and size scales of the microstructure. Application of this stereological technique to phenomena such as equiaxed solidification and directional cellular/dendritic growth will be presented.

MICROSTRUCTURE AND ITS EFFECTS ON AMORPHOUS NANOPHASE & NANOCRYSTALLINE MATERIALS: Session II - Synthesis and Consolidation

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow and Fracture Committee, Jt. Electronic, Magnetic and Photonic Materials Division/Structural Materials Division, Alloy Phases Committee, Chemistry and Physics of Materials Committee, Structural Materials Division, Physical Metallurgical Committee

Program Organizers: Ram B. Bhagat, Pennsylvania State University, 227 Hammond, University Park, PA 16802; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899; Stephen Spooner, Oak Ridge National Lab, Solid State Division, Oak Ridge, TN 37831

Monday PM Room: 205
February 16, 1998 Location: Convention Center

Session Chairs: Robert Dowding, U. S. Army Research Lab, Materials Directorate, Aberdeen Proving Ground, MD 21005; S. Berger, Technion, Department of Materials Engineering, Haifa Israel

2:00 PM Opening Remarks

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EFFECT OF CRYSTALLINE PRECIPITATIONS ON THE MECHANICAL BEHAVIOR OF BULK GLASS FORMING ZR-BASED ALLOYS: *A. Leonhard*[1]; M. Heilmaier[1]; A. Gebert[1]; J. Eckert[1]; L. Schultz[1]; [1]Institut für Festkörper- und Werkstofforschung Dresden, Institut für Metallische Werkstoffe, Dresden D-01171 Germany

Production of bulk amorphous Zr-Al-Cu-Ni alloys with a significant supercooled liquid region was carried out by die casting into a copper mould. Amorphous and partially (nano-) crystalline samples were prepared. The microstructure was analyzed by X-Ray diffraction (XRD), scanning (SEM) and transmission electron microscopy (TEM), and chemical analysis with special emphasis on the size and composition of the crystallites. The volume fraction of (nano-)crystalline regions depends on the oxygen content of the alloy. The mechanical behavior was investigated by constant compression rate tests at room temperature with cylindrical specimens of typically 3 mm diameter and 6 mm height. Independent of the chosen composition the samples exhibit relatively low Young's moduli of about 70 GPa, flow stresses around 2 GPa, and elastic strains of up to 3%. Specimens with high volume fractions of crystalline phases are extremely brittle. In contrast, amorphous samples show microplasticity up to 2% strain without significant work hardening. A first explanation is given for the influence of crystalline phases upon crack initiation and propagation.

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OPTIMIZING MICROSTRUCTURE IN NANOCRYSTALLINE ZrO2-Al203: *Bridget M. Smyser*[1]; Richard D. Sisson, Jr.[1]; [1]Worcester Polytechnic Institute, Worcester, MA USA

ZrO2-A1203 nanocomposite powders that allow a grain size of <25nm and the tetragonal phase of ZrO2 to be maintained at high temperatures have the potential to exhibit improved mechanical and thermal properties compared to traditional PSA. Colloidal suspension, sol-gel, and precipitation methods were used to produce 20 vol% Al203/ 80 vol% ZrO2 powders that were subsequently heat treated and characterized using XRD and TEM. The most successful powders were not heavily agglomerated, but rather had a matrix of tetragonal ZrO2 grains surrounded by Al203 in the grain boundaries. It was also found that the alpha-Al203 phase can be subject to extensive grain growth, suggesting that transition phases such as gamma-, theta-, or delta- A1203 might be more desirable for microstructural control.

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MICROSTRUCTURE AND MICROHARDNESS OF NANOCRYSTALLINE Al-Fe ALLOYS AFTER SEVERE PLASTIC DEFORMATION AND AGING: *O. N. Senkov*[1]; F. H. Froes[1]; V. V. Stolyarov[2]; R. Z. Valiev[2]; J. Liu[3]; [1]University of Idaho, Moscow, ID 83844-3026; [2]Ufa State Aviation Technical University, Ufa 450000 Russia; [3]ALCOA Technical Center, Alcoa Center, PA 15069

A nanocrystalline structure was produced in a number of aluminum-iron alloys with the use of a novel technique of severe plastic deformation (SPD) of ingots by torsion under high imposed pressure. This technique allows a large departure of materials from equilibrium. The microstructure of the alloys was studied with the use of TEM and EDS. The SPD led to solid solubility extension of iron in the aluminum matrix, dispersion and dissolution of second phase particles, grain size reduction into nanometer range, and partial amorphization. Microhardness of the alloys increased essentially after SPD due to the grain refinement and the solid solubility extension. Aging of the SPD samples led to further increase of the microhardness because of decomposition of the supersaturated solid solution and fine precipitate formation. The enhanced properties were retained up to 350°C.

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METAL NANOPARTICLES GENERATED BY LASER ABLATION: *Dr. Michael F. Becker*[1]; Dr. James R. Brock[2]; Dr. Hong Cai[3]; Dr. John W. Keto[3]; Mr. Jaemyoung Lee[1]; Mr. William T. Nichols[3]; [1]Univ. of Texas-Austin, Electrical & Comp. Engr., Austin, TX 78712 USA; [2]Univ. of Texas-Austin, Chemical Engineering, Austin, TX 78712 USA; [3]Univ. of Texas-Austin, Physics, Austin, TX 78712 USA

We study a new method for producing ultra-fine particles (nanoparticles) that employs pulsed-Laser Ablation of Microparticles

(LAM). Pulsed excimer laser radiation at 248 nm wavelength was used to ablate ~2 um feeedstock of silver (spherical), gold (spherical), and permalloy (irregular, Ni81%:Fe19%) particles under both normal atmospheric conditions and other gas ambients. The ejected nanoparticles were collected on silicon substrates for further analysis. Scanning electron micrographs and transmission electron micrographs of the samples were analyzed by computer-aided image processing to determine the effect of irradiation conditions on the nanoparticle size distribution. The results showed that mean particle diameters were normally in the range from 50 to 100 nm and that the particle size distributions were generally log-normal, with dispersion (diameter/standard deviation) ranging from 0.2 to 0.5. The produced nanoparticles were generally spherical except that the larger gold nanoparticles were cubes.

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SYNTHESIS AND CHARACTERIZATION OF ULTRAFINE Ti3Al FORMED BY MECHANOCHEMICAL PROCESSING: *E. G. Baburaj*[1]; Xiaofu Chen[1]; F.H. (Sam) Froes[1]; [1]University of Idaho, Institute for Materials and Advanced Processes, Moscow, ID 83844-3026

The intermetallic phase Ti_3Al has a large composition range of 22 to 35 at%Al. Conventionally desired composition of the alloy are formed by melting. This paper will discuss an alternative synthesis method to ingot metallurgy, namely the mechanochemical processing to form alloy powders directly from metal halides by co-reduction induced by mechanical alloying. The simultaneous reduction of $TiCl_4$ and $AlCl_3$ occurs by the chemical reaction $6TiCl_4 + 2AlCl_3 + 15CaH_2 + 2Ti_3Al + 15CaCl_2 + 15H_2$. The hydrogen released during the reaction partially hydrides the titanium aluminide and thereby minimizing oxidation. The milled product has been leached to produce ultrafine Ti_3Al powder in the size range of 10-500 nm.

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BOUNDARY MISORIENTATIONS IN ULTRAFINE GRAIN COPPER MADE BY SEVERE PLASTIC DEFORMATION: *Mr. Sean R. Agnew*[1]; Dr. Julia R. Weertman[1]; [1]Northwestern University, Materials Science & Eng. Dept., Evanston, IL 60208-3108 USA

Ultrafine grain metals have been produced using novel deformation techniques capable of introducing very high levels of strain, such as equal-channel angular extrusion (ECAE). A simple technique for measuring the orientation of grains in a TEM with a double-tilt specimen holder using Kikuchi patterns has been applied to ultrafine grain copper made by ECAE. Studies by Hughes et al. have recently shown that the boundary misorientations in rolled f.c.c. metals scale with the amount of strain the metal has been subjected to. The aim of this study is to determine whether or not ultrafine grain metals processed by ECAE follow the same trends. Boundary misorientations can have a dramatic impact on the materials properties (e.g. creep, since boundary diffusivity is a strong function of boundary misorientation.) The impact of the misorientation study results on our understanding of the mechanical properties of ultrafine grain metals will be discussed.

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VIBRATIONS OF NANOCRYSTALS: *B. Fultzl*[1]; H. Frasel[1]; C. C. Ahnl[1]; J. L. Robertson[2]; S. Spooner[2]; E. E. Alp[3]; W. Sturhahn[3]; T. S. Toellner[3]; R. McQueeney[4]; [1]Caltech, Pasadena, CA 91125; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831; [3]Los Alamos National Laboratory, MSH805, MLNSC, Los Alamos, NM 87545; [4]Argonne National Lab, Advanced Photon Source, Argonne, IL 60439

We have used inelastic neutron scattering and resonant inelastic gamma-ray scattering to measure the phonon density of states (DOS) of metallic nanocrystals of fcc Ni-Fe [1] and bcc Fe [2]. The DOS curves of nanocrystals were compared to those of equivalent metals with large crystallites. For nanocrystals there is an enhancement in the phonon DOS at "low" energies of 0 - 15 meV, and a severe broadening of all features in the phonon DOS. Some nanocrystals also show intensity at energies above the highest energies of bulk crystals (> 40 meV). These distortions of the phonon DOS affect significantly the thermodynamic stability of nanocrystals through changes in vibrational entropy. 1. Frase, et al., Philos. Mag. B 75, 335 (1997). 2. Fultz, et al., Phys. Rev. Lett., -Sept. (1997).

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DENSIFICATION AND GRAIN GROWTH KINETICS DURING THE HOT PRESSING OF NANOCRYSTALLINE Al AND CU POWDERS: *R. B. Bhagat*[1]; G. Rajesh[1]; G. Wright[1]; [1]The Pennsylvania State University, Applied Research Laboratory, State College, PA 16084

Nanocrystalline aluminum and copper powders have been consolidated to full density by hot pressing in conjunction with Ashby's HIP map predictions. The consolidation is relatively fast to minimize grain growth. We find that the HIP map predictions are not consistent with the experimental results and simple "tuning" appears inappropriate for the nanometer size particles. A new computer program has been written to study dominant mechanisms of pore removal and grain growth during the densification process for producing nanograined materials. The program has the capabilities of (a) using a statistical description of the particle size distribution and (b) implementing a statistical design of experiments.

MICROSTRUCTURE & PROPERTIES OF DIRECT FABRICATED MATERIALS: Microstructure & Properties of Direct Fabricated Materials II

Sponsored by: Materials Design and Manufacturing Division, Surface Modification & Coatings Technology Committee
Program Organizers: John E. Smugeresky, Sandia National Labs, MS 9403 Bldg. 940-0969, Livermore, CA 94551-0969; Michelle Griffith, Sandia National Labs, Albuquerque, NM 87185; David M. Keicher, Optomec Design Company, Albuquerque, NM 87123

Monday PM — Room: Centro Room C
February 16, 1998 — Location: Convention Center

Session Chairs: D. M. Keicher, Albuquerque, NM 87123; M. L. Griffith, Sandia National Labs, Albuquerque, NM 87185

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MICROSTRUCTURAL CONTROL USING LASER DEPOSITION PROCESSES: *D. J. Thoma*[1]; G. K. Lewis[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545

Solidification studies of laser deposition processes, such as Directed Light Fabrication (DLF), have demonstrated that a continuous liquid/solid interface is maintained while achieving constant cooling rates that can be varied between 10 to 105 K s-1 and solidification growth rates (that scale with the beam velocity) ranging up to 10-2 m s-1. With the demonstrated capability to process metals with melting temperatures from aluminum to tungsten, controlled solidification analyses covering at least four orders in magnitude of cooling rates and two orders in magnitude of growth rates can be accomplished on most any metal (both conventional as well as advanced materials) while maintaining at least annealed material properties of the fully dense samples. As a result, flexible microstructural control is possible through solidification pathways associated with the high solidification rates. The details of processing opportunities will be presented.

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FREE FORM FABRICATION OF METALLIC COMPONENTS USING THE 3DWIRE PROCESS: *M. L. Griffith*[1]; L. D. Harwell[1]; D. L. Greene[1]; J. A. Romero[1]; T. Buchheit[1]; V. Tikare[1]; [1]Sandia National Laboratories, Albuquerque, NM 87185

Direct metal deposition technologies produce complex, near net shape components from CAD solid models. Most of these techniques fabricate a component by melting powder in a laser weld pool, rastering

this weld bead to form a layer, and additively constructing subsequent layers. Powder feed material in these processes results in near net shape, high strength components, with the ability to blend materials for novel properties. This talk will describe a new direct metal deposition process, known as 3DWire, whereby a small diameter wire is used instead of powder as the feed material to fabricate components. This allows for faster deposition rates, smoother surface finishes, and easy material handling. Currently, parts are being fabricated from 308L stainless steel and Aermet 100. Microscopy studies show the 3DWire parts to be fully dense with fine microstructural features. Initial mechanical tests show the stainless steel parts to have good strength values (sy= 58 ksi, sult= 95 ksi, 87 HRB) with retained ductility (65%). This work supported by the U. S. 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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THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF TYPE 316 STAINLESS STEEL PRODUCED BY DIRECTED LIGHT FABRICATION (DLF) TECHNOLOGY: *P. W. Hochanadel*[1]; P. G. Dickerson[1]; G. K. Lewis[1]; M. J. Cola[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545

Directed Light Fabrication (DLF) is a fabrication technique that has many potential applications, which include rapid prototyping and rapid tooling. This technique utilizes materials that are easily processed by conventional methods, as well as advanced materials that are difficult to process or cannot be processed by conventional metal forming techniques. Type 316 stainless steel has been successfully fabricated into many complex shapes and many sizes through DLF technology. Light microscopy and scanning electron microscopy were used to analyze the microstructure of this alloy produced by DLF technology. The microstructure was primarily austenite and apparently deficient in ferrite. Uniaxial tensile and hardness tests were performed to characterize the corresponding mechanical properties. The mechanical properties of the 316 alloy deposited by DLF is comparable to those of conventional wrought products.

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ENGINEERED MICROSTRUCTURES OF LENS® PROCESSED MATERIALS FOR ENHANCING MECHANICAL PROPERTIES: *J. E. Smugeresky*[1]; D. M. Keicher[2]; M. L. Griffith[3]; J. A. Romero[3]; M. E. Schlienger[3]; [1]Sandia National Laboratories, Livermore, CA 94551-0969; [2]Optomec Design Company, Albuquerque, NM7123; [3]Sandia National Laboratories, Albuquerque, NM 87185-1411

The Laser Engineered Net Shaping LENS® process for direct fabrication with metal powders consists of localized melting and resolidification of metal particles to form net shapes in an additive mode without the need of molds or machining. Solidification rates for this newly emerging technology are sufficiently high that the benefits of rapid solidification are being realized. By including the ability to process more than one powder at a time, true engineering of materials is possible by simultaneous determination of composition, microstructure, and complex geometrical shapes with full theoretical density, using structural materials. Using statistically designed experiments, 316 SS, MicroMelt 10, H-13, and Inconel 625 have been examined to identify the range of microstructures and properties in the as-processed condition. Results with homogeneous and graded materials will be discussed in terms of controlling their microstructure to achieve enhanced mechanical properties. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. Department of Energy under contract number DE-AC04-94AL85000.

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CHARACTERIZATION OF A THIN-WALLED 316 STAINLESS STEEL COMPONENT PRODUCED BY LASER-AIDED DIRECT METAL DEPOSITION: *Dr. Richard J Grylls*[1]; Thomas J. Lienert[1]; Vishal S. Shah[1]; Dr. M E Schlienger[1]; D. M. Keicher[1]; Dr. Hamish L. Fraser[1]; [1]The Ohio Sate University, Materials Science and Engineering, Columbus, OH 43210 USA

The objective of this work was to characterize the microstructure of a complex thin-walled component of 316 stainless steel produced by laser-aided direct metal deposition (LADMD). The component was generated at a traverse rate of 20 in/min using a CW Nd:YAG laser operated at 190 watts utilizing -100/+325 size powder. Characterization was accomplished using optical microscopy, SEM and TEM. The microstructure was comprised of vermicular ferrite in an austenite matrix, suggesting a primary ferrite solidification mode. The volume fraction of ferrite was determined by optical image analysis to be in the range of 6-8%. The apparent solidification cell spacing was between 3 and 5 μm, as measured by image analysis of scanning electron micrographs, and a cooling rate of 2x104°C/s was estimated using published data. Small (~200nm) pores, occupying less than 0.3 vol%, were distributed throughout the microstructure. The average Vickers hardness number for the LADMD component was 216 as compared to 140 for a wrought 316 alloy. The increased hardness of the LADMD part relative to the wrought alloy was attributed to the small solidification cell spacing and a greater dislocation density. This presentation will examine microstructural variations in the component, especially those associated with multiple thermal cycles and with sharp changes in the deposition direction, and will address the effects of LADMD processing on the stresses present in the sample.

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TITANIUM PROCESSING WITH LENS®: *Dr. M. E. Schlienger*[1]; Dr. M. Griffith[2]; Mr. M. Oliver[2]; Mr. J. A. Romero, Jr.[2]; Dr. J. E. Smugeresky[2]; [1]Sandia National Laboratories, Albuquerque NM; [2]Sandia National Laboratories, Livermore, CA 94551 USA

LENS® (Laser Engineered Net Shaping) is a direct fabrication process in which an Nd YAG laser is used to convert metal powders into fully dense three dimensional parts. Since the process is a direct fabrication process, it is particularly well suited to the production of components out of materials which might otherwise pose a challenge to conventional machining or fabrication processes. For this reason the process has received considerable attention for its capability to process tool steels. In addition to tool steels, another material that poses some processing challenges is titanium and its alloys. This work reviews the recent efforts underway at Sandia National Laboratory in the LENS® processing of titanium powders into bulk components. A brief overview of the safety considerations will be covered. Results of a parametric factor space, associated microstructures and the microstructural changes which occur as a result of part heating as the build progresses will be discussed. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. Department of Energy under contract number DE-AC04-94AL85000.

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SIMULATION OF THE EFFECT OF VARIABLE TEMPERATURE HISTORY ON 3-D GRAIN GROWTH DURING LASER ENGINEERED NET SHAPING: *Veena Tikare*[1]; M. L. Griffith[1]; M. E. Schlienger[1]; J. E. Smugeresky[1]; [1]Sandia National Laboratories, Livermore, Albuquerque, CA, NM 94551-0969, 87185-1405

Laser Engineered Net Shaping, LENS® is an advanced manufacturing technology which can be used to fabricate complex shapes from a variety of metals. The resulting microstructures of LENS' components are unique and depend on the solidification of small regions and the microstructural evolution of the solidified regions during cooling to ambient temperatures. We have developed a 2-D model capable of simulating grain growth in a non-linear, dynamic temperature profile. This model will be extended to 3-D and be used to study the effect of different temperature profiles on the microstructural evolution. In this paper, the model will be described, it's application to simulate the LENS® fabrication process will be presented and the results of the simulations will be presented and compared to experimental results. The results will be discussed with emphasis on how the temperature distribution in the LENS® part during fabrication effects the microstructural evolution. We will show how this model can be used (a) to gain insight into the fabrication parameters on the microstructural evolution, (b) to predict microstructural evolution given sufficient information about materials parameters and processing conditions and (c) to tailor the fabrication parameters to obtain the desirable microstructural features. Sandia is a multiprogram laboratory operated by

Sandia Corporation, a Lockheed Martin Company, for the U.S. Department of Energy under contract number DE-AC04-94AL85000.

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CONTROL OF SOLIDIFICATION AND COMPOSITION IN LIQUID METAL JETTING OF SOLDER MATERIALS: *Charles Smith*[1]; [1]University of Texas, Arlington, TX

No abstract available.

NON-AEROSPACE APPLICATIONS OF TITANIUM & ITS ALLOYS: Session II - Marine and Offshore Applications; Biomedical Applications

Sponsored by: Structural Materials Division, Titanium Committee
Program Organizers: F.H. (Sam) Froes, University of Idaho, IMAP-Mines Bldg. #321, Moscow, ID 83844-3026; P.G. Allen, Timet, P.O. Box 2128, Henderson, NV 89009; M. Niiomi, Toyohashi Univ of Technology, Dept. of Production Systems Eng., Toyohashi 441 Japan

Monday PM Room: 101
February 16, 1998 Location: Convention Center

Session Chair: P. G. Allen, TIMET, Henderson Technical Laboratory, Henderson, NV 89015

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TITANIUM AS A HIGH STRENGTH MARINE FASTENER MATERIAL: *Mr. Robert L. Tregoning*[1]; [1]Marine Surface Warfare Center, West Bethesda, MD 20817-5700

Structural alpha-beta and beta titanium alloys can serve as useful high strength (σ > 100 ksi) marine fasteners if certain design principals are followed. Titanium's lower stiffness, reduced weight, and imperviousness to general and crevice corrosion all offer improved fastener performance when compared with more traditional iron-based and nickel-based materials. Titanium is also more noble than most other marine, structural materials and offers the possibility of joining nickel-chromium-molybdenum (UNS N00625) components without impressed current to compensate for galvanic incompatibility. However, there are concerns about titanium's room temperature creep/stress relaxation behavior, stress corrosion cracking (SCC) susceptibility, fatigue resistance, and dynamic fracture toughness which could restrict its use as a fastener material. These properties have been examined for several alpha-beta (Ti 6Al-4V and Ti 5Al-1Sn-1Zn-1V-0.8Mo) and beta (Ti 3Al-8V-6Cr-4Mo-4Zr and Ti-16Mo-2.5Nb-3Al) titanium alloys to highlight the relative strengths & weaknesses of each class. Guidance will also be provided for utilizing titanium when these potential deficiencies are a concern.

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THE OFFSHORE OIL AND GAS INDUSTRY IN NORWAY: *Jarl Skauge*[1]; [1]IXP - Shultz Export Gmbh, Bergen Norway

Titanium has found wide acceptance and dramatically increased use in the Norwegian Offshore Oil and Gas Industry over the past ten years. Particularly since 1990 has consumption increased from only a few hundred kilos per project; mainly hypochlorite dosing equipment and heat exchangers, to as much as up to 500 tons per platform in sea water pipework for ballast, cooling and fire systems. This growth has been achieved against a background of prejudice and discrimination based on outdated - and false- notions of high price, limited availability, difficulties of fabrication and welding as well as a near total lack of local fabricators. It took a great deal of campaigning technically and commercially over a number of years before the benefits and cost effectiveness of titanium were registered with designers, workshops and operators, and many demonstrator programmes and tests were carried out before applications on larger scales. More than fifteen companies in Norway now offer their services to satisfy demand for local stocking, machine shop, field fabrication and welding.

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NORTH SEA APPLICATIONS OF TITANIUM: *Ms. Liv Lunde*[1]; Ms. Hilde B. Nordvik[1]; [1]Institute for Energy Technology (IFE), N-2007 Kjeller Norway

There has been a dramatic increase in the use of titanium in North Sea installations for production of oil and gas over the last ten years. More than thousand tons of titanium have been used on platforms and in sub-sea installations in the Norwegian sector. With the fast growing offshore market new companies have started titanium manufacturing in Norway. IFE has been the Norwegian centre for titanium technology and has, via the Titanium Technology Forum, served the industry with information and guidance regarding properties, manufacturing and use of titanium. Norwegian industry is now developing improved technology for titanium, one example being cold forming processes, that results in significant reduction in costs compared to more traditional methods. The paper refers some important experiences gained from titanium manufacturing in Norway and summarizes off-shore applications where titanium has been taken into recent use. Future applications where titanium are being considered are also mentioned.

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FATIGUE PROPERTIES OF TI-6Al-4V-0.1RU RISER PIPES JOINED BY RADIAL FRICTION WELDING: *Dr. Frank Torster*[1]; Dr. Jorge Fernandez dos Santos[1]; Dr. Wolfgang Dietzel[1]; Dr. Mustafa Koçak[1]; Mr. Graham Hutt[1]; [1]Stolt Comex Seaway Ltd., Aberdeen AB21 9RQ Scotland; [1]GKSS-Forschungszentrum, Institute of Materials Research, Geesthacht 21502 Germany

Current flexible pipe technology for offshore deepwater exploitation of oil and gas reserves has reached its design limit in terms of maximum water depth, pipe diameter, operating pressure and temperature. Titanium alloy pipe seems to be an ideal alternative for dynamic risers especially for the handling of untreated well products. For the economic manufacturing of titanium alloy risers in combination with high weld quality standards, the Radial Friction Welding process (RFW) has been selected. This innovative processes presents a series of advantages over the conventional Gas Tungsten Arc Welding (GTAW) process, e.g. the extreme short welding time of typically less than 40 seconds. Additional many metallurgical problems associated with the fusion welding of titanium can be avoided since the RFW is a solid state process. This study evaluates the fatigue behavior of welded joints in seawater environment (150°C, 5 bar), simulating service conditions. Tests were conducted on standard flat SN specimens mounted in a specially built autoclave. The obtained results are discussed on the basis of the observed microstructure and the mechanical properties of the welded joint. The results indicated that in general the fatigue behavior of the welded joints is comparable to that previously obtained for the base material. Results of hardness tests have shown that strength levels of heat affected zone (HAZ) and weld metal were slightly higher than those of the base material. This is a consequence of the refined microstructure resulting from the high cooling rates imposed by the welding process.

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FATIGUE OF TITANIUM BUTT WELDS - EFFECT OF DEFECTS: *Stig Berge*[1]; [1]Norwegian University of Science and Technology, Dept. of Marine Structures, Trondheim 7034 Norway

Titanium alloys offer excellent and well proven properties for applications in load carrying structures exposed to sea water and similar environments, notably good corrosion resistance and large strength/weight ratio. Fatigue design criteria for titanium, in particular for welded joints, are less documented. Fatigue testing was carried out on TIG butt welds of titanium alloy GOST 19807-91 (4.4Al 1.7V, Yield strength 660 MPa) with plate thickness 20 mm. Prior to testing, the welds were inspected by radiography and by ultrasonic methods. Small defects classified from X-ray images as pores, slag and third particles, were present but within acceptable limits. Fatigue loading was axial,

constant amplitude loading with stress ratio R=0.1. In several cases fatigue failure was initiated by internal defects with size in thickness direction less than 1 mm. This indicates that the defect sensitivity of titanium subjected to fatigue loading is different and more severe from that of steel. In the paper, the following will be presented (work is ongoing): Fatigue strength data and SN curve for welded titanium; Fracture mechanics analysis of crack growth from initial defects, surface and internal; Tentative conclusions regarding defect sensitivity of titanium subjected to fatigue loading.

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ELECTROCHEMICAL AND MICROSTRUCTURAL ANALYSIS OF TITANIUM-TANTALUM ALLOYS FOR METALLIC IMPLANT CONSIDERATION: *E. A. Trillo*[1]; H. Obispo[1]; S. W. Stafford[1]; L. E. Murr[1]; [1]The University of Texas at El Paso, Department of Metallurgy and Materials Engineering, El Paso, TX 79968

Titanium alloys have been utilized as surgical implant materials for several years, normally in the form of Ti6Al4V. This research considers relatively new types of Ti-alloys (Ti40Ta and Ti50Ta) for such applications. To evaluate the effectiveness of the surface oxide layer, in vitro electrochemical corrosion studies were conducted. The potentiodynamic tests were performed on Ti40Ta, Ti50Ta, and Ti6Al4V materials and their passive behaviors were compared in a simulated biological solution. Transmission electron microscopy (TEM) was utilized to compare the precipitate and second phase structures. X-ray diffraction and EDS analysis were employed to characterize the precipitate phase. These titanium-tantalum alloys have not been fully explored in the literature and this work is an attempt to bring the structure and property relationships to light.

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DENTAL CASTING OF TITANIUM AND TITANIUM ALLOYS: *Mr. Toru Okabe*[1]; [1]Texas A&M University System-Baylor College of Dentistry, Department of Biomaterials Science, Dallas, TX

When the commercial production of titanium sponge became successful in the 1940's, the usefulness of titanium was quickly recognized, and it became the material of choice for many critical applications due to its excellent mechanical properties and corrosion resistance. In particular, the combination of high strength-to-weight ratio made this metal the backbone material for aerospace applications. Recent safety concerns and the possibility of adverse biological effects of various biomaterials rapidly turned attention to the use of titanium and titanium alloys, first in medicine and then in dentistry, because of titanium's excellent biocompatibility. For the possible application of titanium to dentistry, several net shapes or near net-shape fabricating technologies have been tried. These include casting, powder metallurgy, superplastic forming, and electrical discharge machining and milling. Among these efforts, casting appears to be the most studied area. Although casting technologies were first established on a large, industrial scale, various modifications were needed for small-scale biomaterials applications. With a tremendous effort, particularly in the area of casting equipment and investment materials, by dental materials investigators and clinicians, and at research labs in dental manufacturers and metal industries, the technique of casting this difficult metal has greatly advanced during the last 20 years. Using specially developed dental titanium casting systems, a great deal of data on cast commercially pure (CP) titanium and some alloys, especially of Ti-6Al-4V, have been gathered in the areas of strength, surface quality, castability with internal soundness, and casting accuracy. A considerable number of clinical trials testing titanium crowns, bridges, and dentures, have also been performed at various institutions. It appears that dental titanium casting has now almost reached the stage where its practical applications should seriously be assessed, even though some problems have not been completely solved. In this presentation, a brief review is given of the present understanding of some aspects of titanium dental casting.

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NEW ß TYPE ALLOYS WITH HIGH BIOCOMPATIBILITY: *M. Niinomi* [1]; D. Kuroda[2]; M. Morinaga[3]; Mr. Kato[4]; T. Yashiro[4]; [1]Toyohashi University of Technology, Toyohashi Japan; [2]University of Technology, Department of Production Systems Engineering, Toyohashi, Toyohashi Japan; [3]Nagoya University, Department of Materials Science and Engineering, Nagoya Japan; [4]Daido Steel Co.,Ltd, Market Development Department, Nagoya Japan

New ß type alloys composed of non - toxic elements for biomedical materials with lower moduli of elasticity were designed based on the d - electron alloy designing method. The basic mechanical properties of the designed alloys were investigated. Ti-Nb-Ta-Zr, Ti-Nb-Ta-Mo and Ti-Nb-Ta-Sn system alloys are expected to have greater performance for metallic biomedical materials. The designed Ti-29Nb-13Ta-4.6Zr in aged conditions exhibits comparable strength, 0.2% proof stress and elongation to those of titanium alloys developed for biomedical materials to date. The Young's modulus of the alloy is lower comparing with those of titanium alloys for biomedical materials developed to date. Ti-Nb-Ta-Mo and Ti-Nb-Ta-Sn system ß type alloys designed also exhibit relatively lower Youngs moduli and greater balance of mechanical properties.

PROCESSING-STRUCTURE-PROPERTY RELATIONSHIPS OF COMPOSITE INTERFACES: SESSION I: ROLE OF INTERFACES IN DISCONTINUOUSLY REINFORCED COMPOSITES

Sponsored by: Jt. ASM International: Materials Science Critical Technology Sector, Composite Materials Committee, Materials Design and Manufacturing Division, Powder Metallurgy Committee

Program Organizers: Sunil G. Warrier, UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894; Mary Lee Gambone, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433; Ray Y. Lin, University of Cincinnati, Dept. of Materials Sci. & Eng., Cincinnati, OH 45221-0012; Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433

Monday PM — Room: Centro A
February 16, 1998 — Location: Convention Center

Session Chairs: Mary Lee Gambone,WL/ MLLM, Wright Lab Materials Directorate Wright-Patterson AFB, OH 45433; Prof. Ray Y. Lin, Department of Materials Science and Engineering, University of Cincinnati, Cincinnati, OH 45221

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ARMY FOCUSED RESEARCH TEAM ON METAL MATRIX COMPOSITES: *Ernest S. Chin*[1]; [1]Army Research Laboratory, Weapons and Materials Research Directorate, Aberdeen Proving Ground, MD 21005 USA

With the emphasis on smaller but more mobile combat forces to meet a broad range of mission requirements, future weapon platforms and upgrades will require materials to do better, do more, weigh less and cost less. Metal matrix composites (MMCs) are material candidates for niche Army applications such as armor, armament and vehicle structures. Under the shadow of shrinking resources, a Focused Research Team (FRT) composed of the Army Research Laboratory (ARL), academia, and small business was assembled to address MMC issues for Army applications. The object of this team is to provide both the fundamental and technological break-throughs necessary to design and fabricate functionally graded metal matrix composites (FGMMCs). The immediate focus is on the development of FGMMCs for ballistic protection. Technical issues being addressed by this team encompass: the lack of microstructural understanding on the dynamic and ballistic behavior of MMCs; the need for non-destructive evaluation (NDE) techniques; the challenge in joining FGMMCs to dissimilar materials with minimal interfacial reactions; the availability of low-cost near-net-shape processing of FGMMCs. In this paper, highlights from these

activities will be presented. They include near-field optical microscopy, micro- and nano-scale self-propagating high-temperature synthesis (SHS) reactions, high-strain rate testing and modeling, ballistic evaluation, pressure and pressureless casting. These research efforts represent key technologies necessary for the rapid transition of FGMMC armor to the field for the benefit of our warfighting capabilities.

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EFFECT OF ALUMINUM PARTICLES ON THE FRACTURE TOUGHNESS OF 15 VOL.% SiCp/7093 Al COMPOSITE: *A. B. Pandey*[1]; B. S. Majumdar[2]; D. B. Miracle[3]; [1]Systran Corporation, Dayton, OH 45432 USA; [2]UES, Inc., Dayton, OH 45432 USA; [3]Materials Directorate, Wright Laboratory, Wright-Patterson AFB, OH 45433 USA

Discontinuously reinforced aluminum (DRA) composites have an enormous potential for applications which require high specific stiffness and strength. The use of DRA as a structural material will, however, depend to a large extent on the degree of their fracture resistance. This study is part of an overall effort to improve the fracture toughness of DRA composite. The approach taken in this study was to incorporate large unreinforced aluminum particles into a control DRA, 15 vol.% SiC/7093 Al, by a powder metallurgy technique. The unreinforced aluminum particles were introduced to provide extrinsic toughening in DRA by a crack blunting mechanism. Aluminum particles with two different sizes (300 μm and 10 mm), volume fractions (10% and 25%), and compositions (commercially pure Al and Al-Zn-Cu-Mg alloy) were used for this purpose. Tensile and fracture toughness, JIc, tests using ASTM E-813 standard were performed to obtain an idea about the complete fracture process. Two different heat treatment conditions, under aged and slightly overaged, were used for all the materials. Damage in the tensile as well as CT specimens a head of crack tip were analyzed to provide insight into the damage micromechanisms. Silicon carbide particle fracture and matrix/SiC interface debonding were the dominant damage modes in all the conditions. The crack initiation and growth toughness of DRA were modeled using a critical strain formulation and the predicted toughness was in reasonably good agreement with the experimental values. This work was performed at Materials Directorate Wright Laboratory, Wright-Patterson AFB, OH

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EFFECT OF HEAT TREATMENT CONDITION OF THE MATRIX ON THE FRACTURE TOUGHNESS BEHAVIOR OF PARTICULATE-Al2O3-REINFORCED Al(6061) MMC: *Hamid R. Shakeri*[1]; Zhirui Wang[1]; [1]University of Toronto, Department of Metallurgy and Materials Science, Toronto, Ontario M5S 3E4 CANADA

The main task of this study is to examine the idea of improving fracture toughness of metal matrix composites via alteration/improvement of ductility of the matrix through heat treatment. Tensile and fracture toughness testing based on relevant ASTM standards have been performed on samples with different heat treatment conditions. Both scanning and transmission electron microscopy have been used to study and correlate fracture mechanism(s) and microstructural aspects. Based on our up-to-date results the overall effect of heat treatment on fracture toughness values is similar to that of the monolithic alloy, however, the rate of recovery in fracture-related properties is lower in over-aged condition. To describe this lower rate of recovery at over-aged MMCs the following observations have to be taken into account (i) clear increase in size of the shear lips by increasing aging time and, (ii) a shift from particle fracture to interface (or near interface) debonding when moving from under-aged condition to over-aged. Formation of spinel at the interface is recognized as the main cause of this behavior. Although, spinel products may form during material processing, their formation may continue in solid state as well. Alternatively, the spinel/matrix bonding may go through a drastic change in the long aging process.

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PROCESSING AND CHARACTERIZATION OF SINTERED METAL REINFORCED ALUMINUM MATRIX COMPOSITES: *Mr. Richard P. Baron*[1]; Coleman Jones[2]; Dr. Frank E. Wawner[1]; Dr. John A. Wert[1]; [1]University of Virginia, Materials Science and Engineering, Charlottesville, VA 22903 USA; [2]GM, Powertrain,Ypsilanti, MI 48198 USA

The current investigation involves the fabrication and characterization of squeeze cast aluminum metal matrix composites reinforced with sintered metal preforms. Four types of metallic powder preforms were used (steel, stainless steel, A6 tool steel, and a wear-resistant stainless steel) and a variety of casting conditions (e.g. cast metal temperature, maximum infiltration velocity, etc.) were investigated using systematic design-of-experiments techniques to determine the effect of casting conditions on the composite microstructure and resulting mechanical properties. It was observed that a detrimental reaction phase containing iron, aluminum, and silicon formed at the interface between the metallic preform particles and aluminum matrix, with a lower volume fraction of reaction phase forming at the lower melt casting temperature. This reaction phase appears to promote premature fracture by facilitating crack initiation and propagation. The two types of stainless steel-reinforced composites had smaller volume fractions of reaction phase and exhibit superior tensile properties compared to the steel-and tool-steel reinforced composites. This work has been supported by General Motors.

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EFFECTS OF PRESSURE-INDUCED REINFORCEMENT DEGRADATION IN THE PROCESSING OF SHORT-FIBER REINFORCED Al-Si COMPOSITES: *J. U. Ejiofor*[1]; R. G. Reddy[1]; [1]The University of Alabama, Department of Metallurgical and Materials Engineering, Tuscaloosa, AL 35487-0202 USA

The use of applied pressure in the processing of fiber-reinforced metal matrix composites ultimately plays a critical role on the effectiveness of load transfer in the resulting composites. This study has investigated the fiber degradation effects due to applied pressure in the fabrication of Al-13.5Si-2.5Mg alloy reinforced with Al_2O_3-4%SiO_2 (saffil) short fibers. The conventional, double-compaction reaction-sintering method was used with a compaction pressure of 350 MPa. Fiber deformations and breakage as well as fiber clustering were observed by SEM and X-radiography. An SEM/EPMA analysis of the fractured surfaces of the interface-failed parts showed absence of reaction zones. Localized fiber-matrix reactions were identified, and this is contributory to the determined low tensile and thermal properties. At the applied pressure, no significant change in tensile properties were observed as additions of the reinforcing fibers were made. A 15% decrease in linear thermal expansion coefficient of the matrix alloy was determined at 1.2 vol.% fiber.

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INFRARED ALUMINUM MATRIX COMPOSITE WITH PARTICLE REINFORCEMENTS: *J. Spring*[1]; R. Y. Lin[1]; [1]University of Cincinnati, Dept. Materials Sci. Eng., Cincinnati, OH 45221-0012 USA

Aluminum matrix composites with particle reinforcement have been fabricated with an infrared processing technique. SiC particles of up to 30 vol% have been incorporated in A356 aluminum alloy matrix through the infiltration process. Several types of preforms have been used for the infiltration. To facilitate infiltration, carbon coating has been deposited on the particle surface through the chemical vapor deposition technique at temperatures up to 1100°C. The degree of infiltration has been significantly improved with carbon coatings on the particle. The SEM cross section examination of the as fabricated composite indicated that the infiltration density is better than 95%. SiC preforms from Canmet, Selee Corp. and Aisin Company have been used in this study. The pore size in these preforms appeared to have played a very important role in the success of infiltration. Wear properties of the as-fabricated composites were determined with a pin-on-disk wear test machine using cast iron disks. Microstructural analysis indicated that Al_4C_3 existed at the interface between particles and the matrix.

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ROLE OF INTERFACIAL BEHAVIOR ON THE CREEP RUPTURE CHARACTERISTICS OF A DISCONTINUOUSLY REINFORCED COMPOSITE: *S. B. Biner*[1]; [1]Iowa State University, Ames Laboratory, Ames, IA 50011 USA

In this study, the role of interfacial behavior in the creep cavitation and rupture characteristics of discontinuously reinforced composites is investigated numerically. Free sliding of the grain boundaries, a continuous nucleation of the grain boundary cavities, their diffusional growth and coalescence to form grain boundary facet cracks are fully accounted for in the analyses. The results indicate that, although the composites having failure at the interface exhibit significantly lower minimum creep rates, their rupture time could be as short as or even shorter than that seen for the unreinforced polycrystalline matrix material. This work was supported by USDOE, Office of Basic Energy Science, Division of Materials Science under Contract No. W-7405-ENG-82.

PROFESSIONAL DEVELOPMENT

Sponsored by: Education Committee, Young Leaders
Program Organizers: Mary Wells, University of British Columbia, Dept. of Metals & Matls Eng., Vancouver, BC V6T 124 Canada; Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433; Anthony Mulligan, Advanced Ceramics Research, 851 East 47th St., Tuscon, AZ; Eric Rodeghiero, Cornell University, 416 Bard Hall, Ithaca, NY 14853-1501

Monday PM Room: Centro D
February 16, 1998 Location: Convention Center

Session Chair: Mary Wells, University of British Columbia, Vancouver, British Columbia V6T 1Z4

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THE FOUNDING AND START-UP OF A SMALL HIGH TECH MATERIALS COMPANY: *Mr Anthony C Mulligan*[1]; [1]Advanced Ceramics Research Inc., Tuscon, Arizona 85713 USA

Eight years ago, three recent Engineering Graduate Students and a Professor from the University of Arizona founded a small high tech materials company by the name of Advanced Ceramics Research, Inc. (ARC). In the first year of business, ACR had a small lab and office with no employees and revenues less than $50 000 dollars. With virtually no financial investment, ACR has since grown to over $5 million dollars in gross sales and seventy (70) full-time employees. The company was initially founded with 100% federal funding through the SBIR Program, and over the years has transitioned to greater than 80% of its revenues coming from the commercial sector. ACR's three (3) main areas of business focus are: high temperature structural ceramic composite materials, advanced materials used for rapid prototyping technologies and high performance composite polymer components used in the manufacturing of computer hard drive disks. This paper will discuss the techniques and strategies implemented by the original founders and other insight for operating and growing a small high tech business. Of particular relevance, the paper will include a discussion as to how the founders over-came lack of business experience, including financial and marketing capabilities and management expertise. This paper will also include a discussion of ideas that worked well - as well as those that did not.

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THE CORTLAND CABLE COMPANY INC. 1980-1997: *Dr. E. Pete Scala*[1]; [1]Cortland Cable Company, Cortland, New York 13045

High Strength Fibers were of great research interest in the mid 1960's, extending beyond the synthetic fibers of Nylon and Polyester, and of steel or tungsten wires. Solid State Theory predicted tensile strength and modulus potentials of ten to a hundred times greater, for metals and polymers. All of the world's materials research centers were in the hunt, and the prestigious Gordon Conference - Fiber Science Symposium had early reports of several corporate research successes. Dupont had produced Fiber B (later Kevlar), and several laboratories reported ultra-high strength fibers. By the early 1970's, pilot operations had begun. CLC, a world class finishing line company in Cortland, NY proceeded to braid the Kevlar fiber, to form endless lines that could out-perform steel piano wire, in both strength and stiffness (E). Small braiding machines could outproduce the complex sequences of forming steel wire and cables. Since the high strength fibers could be braided over insulated copper wire, one could form conductor cables to compete with sophisticated electrical cable companies. What followed was the investment and development of a new Cortland Cable Co. that grew to sales of $10 Million/year. Many miles of Ropes and Cables have been produced, ranging from 50 lb Medical lines, to 100 Km long tethers for outer space, or ropes of over 200 000 lb Break Strength for moorings and guys. But it all started with Dupont investing many millions to produce the Kevlar fiber, planning to replace steel wire in tires. This talk will explore the development of the Cortland Cable Co. over the past 17 years and look at the High Strength Fiber market to determine which products failed and which succeeded.

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NORTHWEST METTECH CORP - A CASE STUDY IN ENTREPRENEURSHIP: *Mr Alan Burgess*[1]; [1]Northwest Mettech Corp, Richmond, British Columbia V6V 1Y4 Canada

This talk will outline the development of Northwest Mettech, a company which was started in 1990 by a young Metals and Materials Engineering graduate from the University of British Columbia. Initially, the focus of Northwest Mettech was to sell commercial software to the steel industry. Since that time it has moved into the thermal spray coatings industry providing both coatings and equipment for thermal spray applications and has grown to sales of $3 Million in annually.

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THIRTY YEARS OF FOUNDING, BUYING, MERGING AND SELLING - ENTREPRENEURSHIP IN A BROWN PAPER BAG: *Dr. E. Daniel Albrecht*[1]; [1]Buehler International Inc., Lake Bluff, Illinois

An Entrepreneur must have an idea - a product - a market - and lots of confidence. Then he needs some money and lots of ambition. He needs help from other professionals and he must recruit the best support team he can find. We will tell about corporate lawyers, accountants, banks, insurance companies, patent attorneys, investors and family. We will talk about image and advertising as well as production, quality control and shipping. We will talk about how to make money and then, how to make more money. Purchasing and discounts, borrowing and repayment, negotiation and deferment, facts and truth are all fundamentals of a successful entrepreneurial start-up. We will talk about success and failure and how to turn a failure into more success. Lets quit procrastinating and get into business - do you have a product and a plan?

4:00 PM Coffee Break

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MAKING THE CAN LOOK MORE LIKE THE CLASSIC COKE BOTTLE — A PROJECT MANAGER'S PERSPECTIVE: *Nick Grasso*[1]; [1]American National Can, Elk Grove Village, 2550 Lively Blvd, Elk Grove Village, IL 60007

Recent trends in rigid packaging have seen more and more product differentiation using shaped cans, for both the beer and soda markets. Producing a shaped can industrially presents many technical, organizational, and financial challenges. Performance criteria for axial load strength and shipping abuse resistance, among others, must be maintained while increasing metal thickness as little as possible. The shaping process must be integrated into the production line, without any loss in line speed. Coordination of the efforts of technical support teams, vendors, and plant personnel are critical to the success of the project. Some principles of project management will be highlighted using the Coca-Cola contour can project as a case study. About the speaker: Nick Grasso has been

involved in project management at American National Can Co. for 18 years, while holding various managerial positions. He holds a B.S. in Mechanical Engineering from New Jersey Institute of Technology.

4:45 PM Panel Discussion - Professional Development

RAPID SOLIDIFICATION: MODELING & EXPERIMENTS: Rapid Solidification Fundamentals - Session II

Sponsored by: Materials Design and Manufacturing Division, Solidification Committee

Program Organizers: W. Hofmeister, Vanderbilt University, P.O. Box 1543 Station B, Nashville, TN 37235; R. Trivedi, Iowa State University, 100 Wilhelm Hall, Ames, IA 50011

Monday PM Room: Plaza Room D
February 16, 1998 Location: Convention Center

Session Chair: Douglas Matson, MIT, Matls Science & Engr., Cambridge, MA

2:00 PM INVITED

MODEL OF GRAIN REFINEMENT IN SOLIDIFICATION OF UNDERCOOLED MELTS: *Prof. Alain Karma*[1]; [1]Northeastern University, Department of Physics and Center for Interdisciplinary Research on Complex Systems, Boston, MA 02115 USA

The microstructure of a wide range of metallic systems solidified from the undercooled melt typically consists of either coarse dendritic grains or considerably finer equiaxed grains. Furthermore, the abrupt changes of grain size occur in alloy melts at multiple transitional undercoolings, such that grain refined microstructures are generally observed both at high undercooling and over an intermediate range of lower undercooling. We discuss a model of grain refinement that assumes that these microstructural changes result from the fragmentation of primary trunks of dendrites by remelting during the period after recalescence where the interdendritic melt solidifies. This model predicts naturally the existence of multiple transitional undercoolings and the characteristic size of refined equiaxed grains in relatively good quantitative agreement with experiment. *Research supported by DOE.

2:45 PM

GRAIN REFINEMENT AND THE STABILITY OF DENDRITES GROWING INTO UNDERCOOLED PURE METALS AND ALLOYS: *Dr. Andrew M. Mullis*[1]; Dr. Robert F. Cochrane[1]; [1]University of Leeds, Department of Materials, Leeds LS2 9JT U.K.

We present an analysis of the stability of a dendrite against a small perturbation to the tip velocity. We find that dendritic growth in pure metals and alloys will become unstable above some upper critical undercooling. In alloys above a critical concentration, dendritic growth may also become unstable below a lower critical undercooling. In the example systems studied, Ni-Cu and Ag-O, the location of these unstable regions shows remarkably close agreement with the onset of spontaneous grain refinement. We obtain grain refinement undercoolings for Ni and Ag of 195 K and 160 K respectively, in good agreement with the observed values of 170 K and 133-153 K. In strongly partitioning systems, above some critical concentration, instability is predicted at all undercoolings. In the system Ag-O we calculate this concentration as 190 ppm dissolved oxygen, again in close agreement with the experimentally determined value of 200 ppm.

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Fe EPITAXY WITH Nd2Fe14B IN THE Nd-Fe-B SYSTEM DURING SOLIDIFICATION: A LIMITING CONSTRAINT FOR SOLIDIFICATION MODELS IN THIS SYSTEM: *Dr. Matthew J. Kramer*[1]; Dr. R. William McCallum[1]; Mr. Chang-Ping Li[1]; [1]Ames Lab/ISU, Ames, IA 50011 USA

Few efforts have been devoted to understanding the microstructure development in melt-spun $Nd_2Fe_{14}B$ (2-14-1)alloys. Based on the competition between free energy of nucleation versus growth, traditional solidification models have a number of short comings when applied to the 2-14-1 system. Two primary deficiencies are the lack of consideration of peritectic reactions and the failure to consider the effects of the heat of crystallization, i.e. recalescence. The transition from amorphous to nanophased and subsequent increasing grain size with decreasing wheel speed can be explained using a solidification model which takes into account both the degree of undercooling and recalesence. In addition, this model can be used to explain the development of the textured 2-14-1 in the underquenched condition in regions of high thermal gradients. It also explains the phase selection as a function of undercooling. Results from Energy Disperse Spectroscopy (EDS) and Convergent Beam Electron Diffraction (CBED) demonstrate the existence of an epitaxy between fine Fe inclusions and the matrix of Nd2Fe14B (2-14-1) equiaxed grains. The epitaxy is identified as (110)α-Fe//(330)2-14-1 and [1-10]α-Fe//[001]2-14-1, which suggests that the c-axis texturing is caused by the alignment of primarily solidified Fe dendrites. The Fe inclusions are a result of formation of Fe dendrites during the primary solidification at a temperature above the peritectic (Tp) in those alloys where the undercooling is insufficient to form amorphous or nanophase microstructure. At low wheel speeds, an insufficient amount of heat is extracted and not all of the alloy cools below Tp. Fe dendrites then grow in the direction of the thermal gradient where $Tp < T < Tl$ (temperature of the liquidus). As the temperature cools back below Tp, the remaining liquid reacts with the aligned Fe dentrites to induce the (001) texture in the 2-14-1.

3:55 PM Break

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PARTICLE MIGRATION IN RAPIDLY SOLIDIFIED ALUMINIUM COMPOSITES: *Prof. Laurens Katgerman*[1]; [1]Delft University, Laboratory of Materials, Delft 2628 AL Netherlands

The effect of rapid solidification process parameters on the microstructure and particle distribution in meltspun ribbons have been investigated. SiC particles tend to segregate to the air-side of the ribbons and this segregation effect is influenced by particle size and volume fraction. Similar experiments with in-situ composites, containing TiAl3 particles, were carried out. In this case also particle migration towards the top side of the ribbons was observed. Particle movement during meltspinning is a complex coupled phenomenon including hydrodynamic and solidification effects. In determining what causes particles to migrate many variables such as growth velocity of the solidifying phase, interfacial energies, shape and volume of the particles, velocity distribution, viscosity of the liquid and the solid/liquid interphase shape must be considered. Static particle pushing models have been extended with a boundary layer model for fluid flow and heat transfer in the puddle, to give valuable insight in the process physics of the segregation. In particular the complex interaction between particle movement and fluid flow have been taken into account.

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FINITE ELEMENT ANALYSIS OF HEAT TRANSFER AND MICROSTRUCTURE IN MELT SPINNING OF Ti-Al ALLOYS: *Dr. K. F. Zhang*[1]; Dr. J. S-J. Chen[1]; Dr. A. A. Tseng[2]; Dr. W. E. Frazier[3]; [1]Temple University, Center for Thermal Processing, Philadelphia, PA 19122 USA; [2]Arizona State University, Mechanical Engineering; [3]Naval Air Warfare Center

The solidification behavior of melt spinning of binary alloys was investigated using the Finite Element Method (FEM). In the model, growth undercooling due to the nonequilibrium of rapid solidification was taken into consideration. The time-dependent interfacial heat transfer coefficient between the ribbon and the spinning wheel was incorpo-

rated in the numerical model. Based on the KGT solidification model and measured experiment data, the dendrite arm spacing resulted from different processing parameters was predicted. The example alloy of TiA1 was calculated and compared with results from other model and researchers' experiment work. It was concluded from the simulation that the ribbon thickness, heat transfer coefficient between ribbon and substrate, and microstructure are all affected by the wheel speed. When the wheel speed is varied in range of 5-43 m/see, the interfacial heat transfer coefficient h of TiAl alloy on Mo wheel in single-roll casting is about 10^5 W/m^2 K, the cooling rates is in the range of 10^5 - 10^6 K/sec, and the growth rate is in the range of 50-230 mm/sec. Both the cooling rate and the growth rate are higher near the ribbon-wheel interface and lower near the free surface. The dendrite arm spacing, which decreases with increasing wheel speed, is predicted to be in the range of 0.5 - 5.0 microns. The predicted results are in good agreement with experimental data.

STRENGTHENING IN HIGH TEMPERATURE INTERMETALLICS: STRENGTHENING IN HIGH TEMPERATURE INTERMETALLICS II: Iron Aluminides and Silicides

Sponsored by: Structural Materials Division, Mechanical Metallurgy Committee

Program Organizers: Young-Won Kim, UES, Inc., Materials & Proc. Division, Dayton, OH 45432-1805; Michael J. Kaufman, University of Florida, Dept. of Materials Sci & Eng., Gainesville, FL 32611-2066; Chain T. Liu, Oak Ridge National Lab, PO Box 2008 Bldg 4500S, Oak Ridge, TN 37831-6115

Monday PM Room: 107
February 16, 1998 Location: Convention Center

Session Chairs: Bruce McDonald, National Science Foundation, Mathematical & Physical Sciences Directorate Materials Research Division, Arlington, VA 22230; Easo George, ORNL, Metals and Ceramics Division, Oak Ridge, TN 37831-6115

2:00 PM

THE MECHANICAL BEHAVIOR OF FeAl: *Prof. Ian Baker*[1]; [1]Thayer School of Engineering Dartmouth College, Engineering Sciences, Hanover, NH 03755-8000 USA

This paper presents an overview of the mechanical properties of the B2 compound FeAl. Only in the last few years has considerable progress been made in obtaining reproducible mechanical properties data for FeAl alloys. Two sets of observations are the foundation of this progress. The first is that the large vacancy concentrations that exist in FeAl at high temperature are easily retained at low temperature and that these strongly affect the low-temperature mechanical properties. The second is that room temperature ductility is adversely affected by the presence of water vapor. This paper will highlight our understanding of key phenomena, such as the yield strength anomaly, and show how an understanding of the factors which control the yield strength and fracture behavior has followed from the discovery of the above two effects. Research sponsored by U.S. Department of Energy, under contract DE-FG02-87ER45311 with Dartmouth College.

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MINOR ALLOYING ADDITIONS FOR IMPROVED HIGH-TEMPERATURE STRENGTH OF FeAl ALLOYS: *Dr. Philip J. Maziasz*[1]; R. W. Swindeman[1]; J. L. Wright[1]; [1]Oak Ridge National Laboratory, Metals Ceramic Division, Oak Ridge, TN 37831-6115 USA

Minor additions of Mo, Zr, and B produce significant improvements in yield strength (YS) and creep-rupture resistance of B2-phase FeAl alloys with 36-38 at.% Al. However, additions of C instead of B were necessary to improve weldability. Recently it has been found that additions of C and Zr together with microalloying additions of B (100-400 at.ppm) to such FeAl alloys produce YS values greater than 400 MPa at temperatures up to 800°C, and improved creep resistance at 700-800°C. In Cast FeAl alloys, proper heat-treatments produce high-temperature strength based on fine ZrC precipitation-strengthening without sacrificing room-temperature ductility, strength and impact-toughness. Strategies for further optimizing the effects of microstructure and minor alloying additions on elevated-temperature YS and creep-rupture strength in FeAl alloys will be discussed. Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials (AIM) Program under contract DE-AC05-96OR22464 with Lockheed-Martin Energy Research Corp. 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.

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STRENGTHENING OF IRON AND NICKEL ALUMINIDES BY CERAMIC PARTICULATES: *Joachim H. Schneibel*[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

In recent years techniques have been developed to incorporate between 30 and 80 vol.% of ceramic particulates into iron and nickel aluminides (FeAl and Ni_3Al). In some cases room temperature flexure strengths as high as 1800 MPa have been reported. Clearly, the interfacial strength between the particles and the matrix is an important factor governing the strength of these composites. However, even in very strong composites debonding between the intermetallic matrix and the ceramic particles does frequently occur. To date, attempts to improve the interfacial strength in iron aluminide composites in order to improve their overall strength have shown only small improvements. On the other hand, in iron aluminides some degree of debonding is beneficial since it results in the creation of extremely fine ligaments which fracture not by cleavage, but rather in a ductile manner. This research was sponsored by the Division of Materials Sciences, U. S. Department of Energy, under contract DE-AC05-96OR 22464 with Oak Ridge National Laboratory, managed by Lockheed Martin Energy Research Corporation.

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ANISOTROPY OF MECHANICAL PROPERTIES OF Fe_3Al BASTED ALLOY: *Dr. Dezhi Zhang*[1]; Jimei Xiao[1]; [1]University of Science and Technology Beijing, Department of Materials Physics, Beijing 100083 China

Iron aluminides based on Fe3Al offer many advantages for structural uses at elevated temperatures because of their sufficient high strength, excellent oxidation resistance and relatively low material density. However, the low room temperature embrittlement in air and the environmental sensitivity severely restrict their use. Although the exact mechanism is still unclear, the addition of chromium has been found to increase elongation. Likewise, retaining unrecrystallized microstructure with some degree of B2 order by oil quenching from about 650-750°C has produced increased room temperature ductility in Fe3Al based alloy. This treatment results in a strong texture. In this paper, relationship of mechanical properties and rolling direction of Fe3Al based alloy has been studied. There is a greatly anisotropy in mechanical properties of Fe3Al based alloy. The ductility and ultimate tensile strength are 15.7% and 1161 Mpa in rolling direction respectively, while ductility and ultimate tensile strength are 5.2% and 641 Mpa in vertical rolling direction. The scanning electron micrographs of the room temperature tensile fracture surfaces of all samples looked similar. The difference of tensile properties in various direction may be as structure texture.

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STRENGTHENING AND TOUGHENING ISSUES IN Nb-Al-Ti BASED INTERMETALLICS: R. Grylls[1]; S. Perungulam[1]; H. A.

Lipsitt[1]; *Prof. Hamish L. Fraser*[1]; R. Wheeler[2]; S. Banerjee[3]; [1]The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43221 USA; [2]UES, Inc., Dayton, OH 45432 USA; [3]Bhaba Atomic Research Centre, Bombay India

B2 alloys based on Nb aluminides show a great deal of promise for application at about 800°C. It has been found that for the alloy Nb-40Ti-15Al (in at.%) the mechanical properties are characterized by a lack of work hardening, slip localization and also at intermediate temperatures serrated yielding. The work hardening behavior and slip localization have been attributed to the presence of a very refined dispersion of the w phase, whose resistance to dislocation motion decreases as the particles become repeatedly cut. The serrated yielding is accompanied by a negative strain rate sensitivity, and therefore has the characteristics of a Portevin-Chatelier effect, and this is attributed to the dynamic reforming of the w -phase particles at intermediate temperatures. In addition, at these temperatures, the orthorhombic phase precipitates out on dislocations, and causes an increase in strength. Interestingly, Cr additions result in significant increase in work hardening. Attempts to increase the strength of the alloys by decreasing the Ti content (relative to Nb) result in a marked decrease in toughness and ductility, an effect which has been attributed to the occurrence of pseudo-twining. This work has been supported by the US ONR, Dr. George Yoder as Program Manager.

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ELEVATED TEMPERATURE DEFORMATION OF Cr_3Si ALLOYED WITH Mo: *Dr. S. V. Raj*[1]; J. Daniel Whittenberger[1]; B. Zeumer[2]; G. Sauthoff[3]; [1]NASA Lewis Research Center, Materials Division, Cleveland, OH 44135 USA; [2]McKinsey & Co, Inc., Duesseldorf D-40027 Germany; [3]Max-Planck-Institut für Eisenforschung Gmb, Düsseldorf D-40074 Germany

Recent work has shown that Mo addition to Cr3Si promotes the growth of protective oxide layers at both low (Cr2O3) and high (Si2O3) temperatures in a Cr-30(at.%) Mo-30Si alloy. Preliminary compressive creep tests indicated that the cast form of this material possess superior creep resistance over MoSi2 and SiC particulate reinforced MoSi2 composites, while the properties of a powder metallurgy (PM) Cr-30Mo-30Si are equivalent to those MoSi2. The present paper reports results obtained from additional compressive constant load creep, constant engineering strain rate and bend tests conducted on this alloy in the temperature range 1400-1700 °K. Normal primary creep followed by secondary creep was observed for both arc-melted and PM specimens. The arc-melted specimens occasionally exhibited a tertiary creep. A comparison of the deformation properties of the arc-melted and PM materials revealed that the former was much stronger than the latter with about a 200°K advantage to achieve similar levels of strength. The deformation behavior of Cr-30Mo-30Si could be well described by a power-law relation with stress exponents of 1.8 and 2.7 for the PM and arc melted material, respectively. However, the activation energy was observed to be about 470 kJ/mol for the alloy irrespective of the processing condition.

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EFFECTS ON RHENIUM ALLOYING ON THE MECHANICAL BEHAVIOR OF $MoSi_2$ SINGLE CRYSTALS: *Dr. Amit Misra*[1]; F. Chu[1]; P. Peralta[1]; K. J. McClellan[1]; J. J. Petrovic[1]; T.E. Mitchell[1]; [1]Los Alamos National Laboratory, Center for Materials Science, Los Alamos, NM 87545 USA

The mechanical properties of single crystal $ReSi_2$ and $(Mo_{0.5}Re_{0.5})Si_2$ alloys grown by the Czochralski technique were investigated. The substitution of Mo by Re in $MoSi_2$ is expected to increase the electron-to-atom ratio and hence, significant changes in the atomic bonding and mechanical properties may be expected. Both $ReSi_2$ and ternary $(Mo_xRe_{1-x})Si_2$ alloys were significantly harder than $MoSi_2$. $ReSi_2$ exhibited an orthorhombic distortion from the tetragonal $C11_b$ structure of $MoSi_2$ but the Mo-rich $(Mo_xRe_{1-x})Si_2$ alloys had the $MoSi_2$ structure. The strengths of these single crystals were evaluated as a function of temperature by hot hardness experiments, and fracture toughnesses were estimated from the lengths of the cracks emanating from the indents. The dependence of toughness on the crystal orientation is interpreted in terms of anisotropic yield and cleavage fracture strengths. The temperature dependence of strength and the solid-solution strengthening of $MoSi_2$ by Re are related to the observed dislocation substructures.

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EFFECTS OF SMALL ALUMINUM ADDITIONS ON THE HARDNESS ANISOTROPY OF MONOCRYSTALLINE C11b $MoSi_2$: *Dr. Pedro D. Peralta*[1]; Dr. Stuart A. Maloy[1]; Dr. Fuming Chu[1]; Dr. Patricia Santiago[1]; Dr. John J. Petrovic[1]; Dr. Terence E. Mitchell[1]; [1]Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, New Mexico 87545-0001 USA

The Vickers hardness of monocrystalline C11b MoSi1.94Al0.06 was measured at room temperature as a function of the crystallography of the indentation plane and the orientation of the indenter diagonals and compared to similar measurements performed in pure MoSi2. It was found that the presence of aluminum results in an uniform decrease of the hardness and significant changes in the slip patterns around the indents, as compared to pure MoSi2. The trend shown by the hardness as a function of crystallography was the same for both alloys. The variation of the hardness and slip patterns with the orientation of the indenter was analyzed in terms of the resolved shear stresses on different slip systems predicted by the elastic fields resulting from a square punch on a half-space. Conclusions are drawn from these results about the effects of small aluminum additions on the dislocation plasticity of MoSi2.

5:10

THE INDUCED ANISOTROPY OF YIELD STRESS FOR PRESTRAINED INTERMETALLICS: *Prof. Bella A. Greenberg*[1]; Prof. M. A. Ivanov[2]; [1]Russian Academy of Science, Institute of Metal Physics Ural Division, Ekaterinburg GSP-170 Russia; [2]National Academy of Sciences, Institute of Metal Physics, Kiev Ukraine

The effect of anisotropy of the yield stress for prestrained intermetallics is believed to exist. It can be detected if, after the high-temperature prestraining and cooling, an intermetallic is deformed at different orientations. The following specific features of the anisotropy are remarkable. First, a sufficiently high anisotropy. The lowest yield stress may be close to the one observed at room temperature without prestraining, while the highest value may approximate the high-temperature yield stress. Second, an induced anisotropy rather than a property inherent in the material. The induced anisotropy may arise owing to the fact that the rigid regular framework proves to be transparent at certain orientations and opaque at other orientations for the subsequent plastic flow. The conditions, at which the high-temperature preliminary deformation may lead to appearance of a unique structural state of intermetallics with a high induced anisotropy of the yield stress are determined.

SUPERPLASTICITY AND SUPERPLASTIC FORMING: Session II - Alloy Composition, Processing and Microstructure

Sponsored by: Materials Design and Manufacturing Division, Shaping and Forming Committee

Program Organizer: Amit K. Ghosh, Univ of Michigan, Dept. of Matls Sci & Eng., Ann Arbor, MI 48109-2136; Thomas R. Bieler, Michigan State University, Dept. of Materials Science and Mechanics, E. Lansing, MI 48824-1226

Monday PM — Room: 106
February 16, 1998 — Location: Convention Center

Session Chair: Thomas R. Bieler, Michigan State University, Dept. of Matls Sci & Mechanics, East Lansing, MI 48824-1226

2:00 PM INVITED

DEVELOPMENT OF SUPERPLASTIC MICROSTRUCTURES DURING THE THERMOMECHANICAL PROCESSING OF Al-Cu-Zr ALLOYS: *N. Ridley*[1]; E. Cullen[1]; F. J. Humphreys[1]; [1]University of Manchester/UMIST, Materials Science Centre, Manchester M1 7HS UK

For an alloy to exhibit superplasticity it must have a uniform equiaxed fine grain (< 10 μm) microstructure of high angle boundaries which must remain stable at temperatures 0.5 Tm. For Al alloys the required structure is obtained by static recrystallization prior to superplastic forming (SPF), e.g., 7475, 5083, or it is developed during the early stages of SPF, e.g., Supral 100, Al-Li 8090. The alloy Supral 100, Al-6Cu-0.4Zr, is basically an Al-Cu solid solution containing some CuAl2 precipitates and a uniform dispersion of very fine (< 10 nm) dispersoids. It is typically cast from a high superheat and aged to precipitate the ZrAl3. Solution treatment and break-down hot rolling then take much of the CuAl2 into solution. Subsequent warm/cold rolling produces a heavily worked structure which evolves into a fine grain structure during the early stages of SPF. By examining a range of binary and ternary alloys containing various amounts of Cu and Zr, the present work has investigated the role of composition and also the role of low temperature working prior to hot deformation on the evolution of a fine grain superplastic microstructure.

2:25 PM INVITED

RECRYSTALLIZATION AND SUPERPLASTICITY IN ALUMINUM, ALLOYS: *T. R. McNelley*[1]; M. E. McMahon[1]; [1]Naval Postgraduate School, Department of Mechanical Engineering, Monterey, CA 93943-5146

There are at least two microstructural transformation routes to enable superplastic response in aluminum alloys. One consists of recrystallization processes involving heterogeneous nucleation within the deformation microstructure, and subsequent growth of grains by the migration of newly-formed high-angle grain boundaries. The other comprises processes involving more gradual changes that occur homogeneously in the deformation microstructure and which have been described as continuous, or "in-situ", recrystallization. For alloys representing these two different routes, the evolution of grain boundary character has been studied by means of computer-aided electron backscatter diffraction (EBSD) analysis methods. These two transition mechanisms result in distinctly different grain boundary misorientation distributions. Grain boundaries developed by the former route are predominantly disordered, mobile high-angle boundaries of high interfacial energy character. The high mobility of such boundaries may facilitate grain boundary sliding but lead to susceptibility to rapid grain growth and limited superplastic flow; grain refinement to improve superplastic response requires control of the density of nucleation sites during processing of such materials.

2:50 PM INVITED

USE OF Sc, Zr AND Mn FOR GRAIN SIZE CONTROL IN Al-Mg ALLOYS: *J. S. Vetrano*[1]; C. H. Henager, Jr.[1]; S. M. Bruemmer[1]; [1]Pacific Northwest National Laboratory, Richland, WA 99352

Tailored microstructures for superplastic deformation were created by the addition of up to 0.5 wt% Sc, 0.1 wt% Zr and 1 wt% Mn to an Al-4 wt% Mg alloy. By altering the composition and heat treat conditions, particles sizes could be manipulated to create either a recrystallization-resistant material, or fine-grained microstructures that did not coarsen significantly even at high homologous temperatures. Uniaxial tension tests at elevated temperatures revealed that the fine-grained materials were highly superplastic. Recrystallized structures were generated by aging the materials at 500°C, which created large (0.5 μm) Al6Mn particles and fine, coherent Al3(Sc,Zr) precipitates, followed by cold rolling and heating. Grains as small as 5 μm were formed even when the cold rolling reduction was as low as 66%. Microstructures resistant to static recrystallization were formed in samples with 1% Mn and at least 0.3% Sc by aging at 300°C. This heat treatment formed fine Al3(Sc,Zr) precipitates and smaller Al6Mn particles than at 500°C. These small particles effectively pinned the substructure even at temperatures up to 550°C. Materials in this condition recrystallized dynamically when deformed at high temperatures. (Work supported by U.S. Department of Energy DE-AC06-76RLO 1830.)

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EFFECT OF Mg AND Mn CONTENT ON SUPERPLASTIC DEFORMATION OF 5XXX ALLOYS: *M. T. Smith*[1]; J. S. Vetrano[1]; E. A. Nyberg[1]; D. R. Herling[1]; [1]Pacific Northwest National Laboratory, Richland, WA 99352

There has been much interest in superplastically forming (SPF) Al-Mg-Mn alloys (5000-series) due to their low cost and weldability. Most research to date has focused on AA5083 (4.5%Mg, 0.8% Mn) or materials with higher Mg content. In this study several 5000-series alloys were processed with Mg levels varying from 2.5 to 4 wt% and Mn from 0.02 to 0.8 wt%. The objective of the work was to better understand the influence of these additions on the ability to form and maintain a fine grain size, and to allow deformation by grain boundary sliding. It was found that to form grains smaller than 10 μm at least 0.25% Mn was necessary. To maintain fine grains at superplastic forming temperatures and allow substantial grain boundary sliding, the critical amount increased to greater than 0.25%. The level of Mg had a weak effect on the recrystallization process with increasing Mg leading to a decreasing grain size. During SPF the principle influence of Mg was to increase intergranular strength and prevent premature failure due to interlinkage of cavities. (Work supported by U.S. Department of Energy DE-AC06-76RLO 1830.)

3:35 PM Break

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SUPERPLASTICITY IN NANOMATERIALS: *R. S. Mishra*[1]; A. K. Mukherjee[1]; [1]University of California, Davis, CA 95616

Fine, equiaxed grain size has been one of the primary prerequisites for superplasticity. Reduction of this grain size has spurred recent investigations on low temperature superplasticity as well as high strain rate superplasticity. Nanocrystalline material provides the ultimate limit to these reduced grain size considerations. Nanometal, nanointermetallic and nanoceramics were investigated. These materials were produced primarily by ball-milling and subsequent consolidation by severe plastic deformation or by high pressure sintering. Polymer precursor derived amorphous ceramic powders were also consolidated using high pressure sintering. The mechanical properties were determined either by constant strain rate compression testing in MTS equipment or by tensile testing in a specially designed mini tensile tester with very good resolution for load and displacement. This computer-interfaced equipment can be used at constant tensile strain rate. The entire tensile specimens were less than 8 mm in length. Preliminary mechanical and microstructural results from this ongoing program will be discussed in the context of the appropriateness of applying the currently available models for superplasticity. (Work supported by NSF-DMR-9630881 and NSF-CMS-96-34179.)

4:10 PM INVITED

MICROSTRUCTURAL ASPECTS OF SUPERPLASTICITY IN ULTRAFINE-GRAINED ALLOYS: *R. Z. Valiev*[1]; [1]Ufa StateAviation Technical University, Institute of Advanced Materials Physics, Ufa 450 000 Russia

It is now well-known that severe refinement of the grain size significantly enhances superplastic properties of metallic materials. The effective route for refining a microstructure up to nano- and submicrometer range can be realized through the techniques of severe plastic deformation. This new method enables processing of ultrafine-grained structures in bulk samples suitable for thorough mechanical tests. Moreover the resulting microstructures are characterized by specific defect structures, crystallographic texture and changes of phase composition. This paper focuses on results from recent studies of the relationship between microstructure and superplastic behavior in several nano- and submicrometer-grained metals and alloys prepared by severe plastic deformation. Structural features of these materials, studied by TEM, HREM, x-ray and DCS are described and used for developing a structural model of ultrafine-grained metals. The results obtained are utilized for analysis of phenomenology and origin of low temperature and high strain rate superplasticity revealed in the processed alloys. (Work supported in part by NSF #DMR-96-30881 and ARO #68171-97-C-9006.)

4:35 PM
FORMATION AND THERMAL STABILITY OF SUBMICROCRYSTALLINE STRUCTURE IN COMMERCIAL 1420 ALLOY: *M. Kh. Rabinovich*[1]; M. V. Markushev[1]; M. Yu. Murashkin[1]; [1]Institute for Metals Superplasticity Problems RAS, UFA Russia

Imparting of submicrocrystalline (SMC) structure by thermomechanical treatment to commercial alloys produced by traditional cast technology is one of the directions of industrial application of superplasticity. The influences of grain size and precipitates of secondary phases on the formation of SMC structure in 1420 (Al-Mg-Li-Zr) alloy were investigated. To estimate the grain size effect three states of an alloy after water cooling from 450°C were studied: an homogenized ingot, a conventional hot-pressed rod, and a rod with microcrystalline structure. The quenched alloy has unimodal distribution of second phases formed by dispersoids of zirconium aluminide (Al_3Zr) 0.5-1nm in diam. The changes in size (within the range of 0.1-1 mkm) of strengthening phases (mainly S-phase (Al_2MgLi)) which form the second mode were achieved by further heterogenization at 250 and 350°C. Severe straining (e = 3.8) was carried out by torsion under the compression method at room temperature. After deformation the alloy has a specific "fragmented" structure and a fragment size (near 100 nm) does not depend on both the initial grain structure and the presence and the size of precipitates.

4:55 PM
SUPERPLASTICITY IN ALLOYS PROCESSED BY EQUAL-CHANNEL ANGULAR PRESSING: *P. B. Berbon*[1]; T. G. Langdon[1]; R. Z. Valiev[2]; M. Furukawa[3]; Z. Horita[3]; M. Nemoto[3]; [1]University of Southern California, Los Angeles, CA 90089-1453; [2]Ufa State Aviation Technical University, Ufa 450000 Russia; [3]Kyushu University, Fukuoka 812 Japan

Several commercial alloys were processed by equal-channel angular (ECA) pressing. Transmission electron microscopy showed that a significant refinement of the microstructure was introduced, and that it was possible to achieve a microstructure where all grain boundaries were high-angled. Tensile tests were performed at low temperatures and/or high strain rates and led to elongations of the order of 1000%. This result demonstrates that ECA pressing can be successfully used in order to increase the superplastic behavior of conventional commercial alloys.

ZINC-BASED STEEL COATING SYSTEMS: PRODUCTION & PERFORMANCE: Session II - Growth and Structure of Galvannealed Coatings

Sponsored by: Structural Materials Division, Ferrous Metallurgy Committee

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Monday PM — Room: Centro Room B
February 16, 1998 — Location: Convention Center

Session Chair: Josef Faderl, Voest Alpine Stahl Linz, Turmstrasse 45, PO Box 3, A-4031 Linz, Austria

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REACTIVE INTERDIFFUSION IN THE Fe-Al-Zn SYSTEM: REACTION MECHANISMS DURING HOT-DIP GALVANIZING: *Y. Leprètre*[1]; J. M. Mataigne[1]; M. Guttmann[2]; J. Philibert[3]; [1]SOLLAC, Mountataire 60761 France; [2]IRSID, Maizières les Metz 57214 France; [3]University of Paris XL, Lab. Mat. Struct., Orsay 91405 France

This article aims at giving a definite and comprehensive explanation of the temporary inhibiting effect of aluminium on the formation of Fe-Zn compounds during hot-dip galvanizing, grounded on the fundamental principles of thermodynamics and interdiffusion, as well as on experimental results. This new approach will enable us to make a logical link between galvanizing in pure zinc baths and in aluminium containing baths, which were hitherto studied in separate ways. In order to achieve this, it will be first necessary to define the notions of real and virtual diffusion paths in a precise way, and to present some new developments of these notions pertaining to their thermodynamic and kinetic properties. The inhibiting effect of aluminium will then be explained in terms of thermodynamic equilibria. The formation and development of outbursts will be detailed and we will show that this microstructure has to be considered as a mere transitionary state, evolving towards a final microstructure entirely defined by an appropriate virtual diffusion path. The different steps of this mechanism will be illustrated by experimental results coupling several analytical techniques. We will also focus on the question of the localization of the formation of the outbursts in relation to the steel's microstructure.

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ABOUT THE EXISTENCE OF A NEW PHASE T_2 IN THE Fe-Al-Zn SYSTEM: Y. Lepretre[1]; B. Fenaille[1]; [1]SOLLAC, Mountataire 60761 France

Abstract not Available

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INHIBITION LAYER BREAKDOWN AND OUTBURST Fe-Zn ALLOY FORMATION DURING GALVANIZING: *C. E. Jordan*[1]; A. R. Marder[2]; [1]Martin Marietta, Schenectady, NY; [2]Lehigh University, Bethlehem, PA

It has been proposed for hot-dip Zn coatings that the role of the Fe_2Al_3 inhibition layer is to prevent outburst formation during galvanizing and that outbursts nucleate at grain boundaries at the steel substrate/inhibition layer interface. Thus, steel substrates have been designed to contain solute additions such as Ti and P that can both

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EFFECT OF ELEMENTS ON GALVANNEALING BEHAVIOR: *S. Chang*[1]; J. B. Yoon[1]; D. C. Bae[1]; [1]POSCO, Technical Research Laboratories, Pohang, 790-785 South Korea

Galvannealing behavior is mainly controlled by heating temperature and time for the reaction of coating zinc and substrate iron. In addition, bath aluminum makes a great influence on the degree of alloying in the coating. Steel chemistry, however, also appears important to determine the galvannealing condition to obtain an optimum coating structure. Some elements in the steel contribute to the increase of strength, but they can noticeably change galvannealing behavior of the coated steel sheet. Meanwhile, minor element in the bath can affect the development of galvannealed coating by reducing the barrier effect of aluminum in the coating. Some of elements in the bath are likely to substitute the sites for aluminum in the coating, thereby promote the reaction of zinc and iron at the interface.

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IN-SITU HIGH TEMPERATURE X-RAY DIFFRACTION STUDY ON PHASE CHANGES DURING GALVANNEALING: *Shuji Gomi*[1]; C. Kato[1]; T. Fujimura[1]; K. Mochizuki[1]; [1]Kawasaki Steel Corporation, Coating Laboratory, Chiba Japan

In-situ time sharing X-ray diffraction measurements were applied to understanding the galvannealing phenomena. After determining diffracted peaks which identified -ζ δ1 and Γ +Γ1 -phases, measurements were conducted to study the effect of Al in the coating and galvannealing temperature on the phase changes. At the beginning of galvannealing at 743K, the main phase change was from -η-Zn-phase to - ζ -phase, followed by - ζ -phase to -δ 1-phase transformation. The - ζ -phase could grow as long as the η -Zn-phase existed. Upon the disappearance of the -η -Zn-phase, the - ζ -phase was transformed into the - δ 1-phase. The Γ -phase was observed after the formation of the δ 1-phase with grew steadily with time. The - ζ -phase was not detected by the galvannealing at 773K due to the temperature close to peritectic transformation temperature 903K. The high Al content in the coating and slow heating and increased amount of the - ζ -phase and decreased the -Γ -phase. By using the obtained results, comprehensive phase changes mechanism during galvannealing were suggested, which had never been revealed by the conventional studies conducted on cooled specimens.

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ALLOYING BEHAVIOR OF HOT-DIP GALVANNEALED SHEET STEELS CONTAINING SILICON: *J. S. Kim*[1]; J. H. Chung[1]; [1]Pohang Iron & Steel Co., Ltd. (POSCO), Kwangyang Rolling Research Team, Technical Research Lab., Kwangyang Korea

The effect of silicon in steel on the alloying behavior of hot-dip galvannealed sheet steels were studied. The presence of a stable silicon oxide formed on the steel surface has been shown to be very detrimental to proper wetting by liquid zinc. Growth rate of Fe-Zn alloy layer was retarded markedly as silicon content in steel increased. Addition of Ni into the molten zinc increased the Fe-Zn alloy reaction. Cross-sectional and planar views of galvanneal coatings were investigated to characterize morphology development by using SEM and TEM. A possible mechanism to explain the retardation effect of silicon is discussed in terms of phase evolution.

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TEM INVESTIGATIONS OF GALVANNEALED STEEL SHEETS: *E. Stangl*[1]; K. Spiradek[1]; M. Brabetz[1]; [1]Austrian Research Centre Siebersdorf

The microstructure of commercial galvannealed steel sheets of different suppliers was investigated with respect to the coating and the steel substrate beneath the coating by transmission electron microscopy (TEM). Cross-sectional samples were prepared by employing ion beam milling. Differences between the investigated samples were mainly found with respect to the following features of the microstructure of the steel substrate: Shape of the grains adjacent to the interface, dislocation density in these grains and Zn-rich areas at the steel grain boundaries. The FeZn coating was in both cases found to be built up of two layers. The bottom layer was formed by crystallites of Γ or Γ phase, the top layer was composed of δ phase. Since the Γ phase was found in the same "monolayer" of crystallites as the Γ_1 phase, Γ crystallites may be formed by a sudden transformation of Γ_1 to Γ. The geometry of the investigated FeZn-regions at the steel grain boundaries indicates, that there may be another effect of forming Zn-enrichments beneath the substrate surface apart from grain boundary diffusion of Zn atoms: If the steel substrate surface is warped so that "tongues" reach into the liquid zinc, FeZn phases can be formed in the hollows beneath the tongues. This work was financed by the ECSC (P 3562).

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A MODEL TO PREDICT GALVANNEAL COATING STRUCTURE AND ITS EXTENSION TO CONTROL OF AN INDUSTRIAL GALVANNEALING LINE: *K. T. Winther*[1]; R. N. Wright[1]; [1]Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA

The development of Fe-Zn intermetallic phases in the coating during the galvannealing process is primarily controlled by iron-zinc interdiffusion. The process has been modeled using a finite element model. The model is based on a one dimensional representation of the coating with moving grid points placed at the interfaces of the phases. The model was programmed in C++ and written to accommodate for variable time-temperature cycles. Experimental samples prepared using a Gleeble; 1500 thermo-mechanical testing device and production samples from Bethlehem Steel were used to calibrate the model. A set of best fit "pseudo" activation energies and pre-exponential factors was found for the diffusion within each phase by minimizing the difference between observed and predicted coating structures. The activation energies and pre-exponential factors found are in general similar to the literature values for the pure systems, however, the effect of aluminum and other complicating factors found in the actual production are indirectly built into the model this way. The model was found to have a good predicting capability of the average coating microstructure over a wide range of time-temperature paths and different coating thicknesses. The software was written so it can be used to find the optimal time-temperature path for a given desired coating microstructure. This project was funded by the New York State Energy Research and Development Authority and done in cooperation with Bethlehem Steel Corporation.