TMS

Become A TMS Member

TMS derives its strength from its members, who take a hands-on approach to shaping the policy, programming, and publications of the society. Guided by these volunteers, TMS serves all segments of its professional community by:

FACILITATING NETWORKING:

By sponsoring numerous annual meetings and specialty conferences, TMS maximizes the opportunities for professionals from industries, universities, and government agencies worldwide to meet face to face and exchange technical ideas and experience, offer customer/client insights, find a mentor and/or serve as one, and just plain chat with peers and colleagues.

PRODUCING JOM AND OTHER PUBLICATIONS:

Every TMS member receives a complimentary subscription to JOM. Formerly Journal of Metals, this highly respected monthly journal, explores traditional, innovative, and revolutionary issues in the minerals, metals, and materials fields. Designed to be of maximum and immediate benefit to readers throughout the world, JOM is on-line before the print version is mailed.

TMS also publishes three other journals (*Journal of Electronic Materials* and *Metallurgical and Materials Transactions A and B*), numerous conference proceedings volumes and textbooks, and videos designed to give materials scientists and engineers the latest information on scientific and applied advances in areas as diverse as electronic materials, automotive manufacture, and extractive metallurgy.

PROMOTING LIFE-LONG LEARNING:

TMS is dedicated to the education of the materials science and engineering professional as well as to cultivating an interest in the field by young people.

For the practicing professional, TMS and its five technical divisions sponsor continuing education courses, primarily technical but also nontechnical, to promote the education and development of current and future professionals.

For student members, TMS participates on both the Accreditation Board for Engineering & Technology (ABET) and the National Council of Examiners for Engineering and Surveying (NCEES) to help, respectively, maintain the highest possible standards in the accreditation of metals and materials programs in academia and in the registration of professional engineers.

—All individuals registering for the 131st Annual Meeting & Exhibition at the non-member fee will automatically receive a one-year complimentary introductory membership for 2002. Your membership will be activated upon completion of your registration form, membership application, and payment of the non-member registration fee. You will receive a membership card and new member packet immediately after the meeting.

- *Members from 77 countries and six of the world's seven continents.*
- *All new members will begin receiving a monthly subscription to JOM.*
- *New members will also be able to continue networking with a prestigious membership at future TMS meetings that fit their area of interest at a discounted member fee.*
- *Additional benefits include access to, and inclusion in the TMS Membership Directory on TMS OnLine at www.tms.org., professional development and continuing education opportunities, and group insurance programs. See the membership page on TMS OnLine for a complete list of membership benefits.*
- *Please direct any questions regarding your complimentary membership to the TMS Member Services Department via email to castello@tms.org or via phone to Margie Castello (724) 776-9000 Ext. 241.*

TMS THE MINERALS, METALS & MATERIALS SOCIETY

PROMOTING THE GLOBAL SCIENCE AND ENGINEERING PROFESSIONS CONCERNED WITH MINERALS, METALS, AND MATERIALS

184 THORN HILL ROAD
WARRENDALE, PA 15086-7514
USA

TELEPHONE: (724) **776-9000**
(800) **966-4867**
FAX: (724) **776-3770**
WEB: **www.tms.org**

THE VISION OF TMS IS TO BE THE PROFESSIONAL SOCIETY OF CHOICE FOR THE WORLDWIDE MINERALS, METALS AND MATERIALS COMMUNITY.

GREAT MEMBER BENEFITS

- Five distinct technical divisions which are composed of 52 separate, highly specialized committees
- Periodicals: JOM, Metallurgical and Materials Transactions A and B, Journal of Electronic Materials
- Conference Proceedings, Monographs, and Textbooks
- TMS OnLine & the TMS Document Ordering Center
- TMS Conferences: TMS Annual Meeting & Exhibition, TMS Fall Meeting, TMS Fall Meeting for Extraction & Processing, Electronic Materials Conference, Specialty Conferences
- Professional Development and Continuing Education Opportunities
- Professional Registration
- TMS Young Leaders
- TMS Resume Referral Service
- TMS Gold or Platinum MasterCard
- Group Insurance
- TMS Membership Directory
- TMS Speakers Directory
- International Healthcare Plan
- Hertz Car Rental Discounts
- Auto and Homeowners Program
- PROinsure Program
  A Professional Liability/Errors and Omissions Program
- PRObop Program
  A Professional Business Owners Package Program
- Member Benefits Program
  Receive a 20% Rebate Buying or Selling Your Home
- Nelson Financial Services Program
- WAAIME Auxiliary Activities

ADMISSION REQUIREMENTS

FULL MEMBER
A candidate for election as full member shall be a person of integrity in activities associated with minerals extraction, processing, fabrication, or with materials applications. A candidate shall hold: (a) A baccalaureate degree in metallurgy, metallurgical engineering, materials science, or materials engineering, and at least 3 years' professional experience. (b) A baccalaureate degree in science or engineering in a discipline other than identified and at least 5 years' professional experience. (c) A baccalaureate degree from a recognized university in a discipline other than (a) or (b) and whose main activities lie in, but are not limited to, the development, management, administration, welfare, sales, or services to the minerals, metals and materials industries, with at least 7 years' experience. A credit in experience of one year for a masters degree or two years for a doctoral degree shall be granted.
Annual dues: $90.00

ASSOCIATE MEMBER
A candidate for associate member shall be a person of integrity who, while not possessing the academic or technical experience of a member, is active in fields that are sufficiently related to the advancement of, or service to, the minerals, metals or materials extraction, processing, or applications industry.
Annual dues: $90.00

The Minerals, Metals & Materials Society is a member society of the American Institute of Mining, Metallurgical and Petroleum Engineers, Inc.

LIFE MEMBER
A candidate for election as life member shall be a person who qualifies as a full member or associate member and desires to only pay dues once.
Dues: $1,350.00
Pay dues once, effective for lifetime regardless of dues increase(s).

REINSTATEMENT
Those members who may have let their dues payment lapse may reinstate in the same grade as when they left by submitting a new application and paying a reinstatement fee of $10.00 plus current dues. If original election year is desired, back dues must be paid to date (half the annual dues fee for each year of lapsed membership); otherwise, election year will be year of reinstatement.

The TMS membership year runs from January 1–December 31. Applications received January 1–September 30 will be processed for the current calendar year.

Applications received after September 30 will be processed for the remainder of the current calendar year and the entire following year. Membership benefits commence upon processing; subscriptions commence January–December of the following year.

Two weeks required for processing of complete applications submitted with full payment. Incomplete applications will not be processed. Allow eight to ten weeks for subscriptions to start.

MEMBERSHIP APPLICATION

PLEASE TYPE OR PRINT

❒ Mr. ❒ Mrs. ❒ Ms.
❒ Dr. ❒ Professor

SEND MAIL TO:
- ❒ Business Address
- ❒ Home Address

TECHNICAL DIVISION SELECTION:
- ❒ Electronic, Magnetic, & Photonic Materials Division
- ❒ Extraction & Processing Division
- ❒ Light Metals Division
- ❒ Materials Processing & Manufacturing Division
- ❒ Structural Materials Divison

THROUGH WHAT MEANS WERE YOU ENCOURAGED TO JOIN TMS?
- ❒ TMS Annual Conference
- ❒ TMS Fall Conference
- ❒ TMS Specialty Conference
- ❒ Exhibitor
- ❒ TMS Staffed Booth
- ❒ JOM
- ❒ MET TRANS, JEM, Etc.
- ❒ Publications Catalog
- ❒ TMS Mailed Brochure
- ❒ Non-TMS Advertisement
- ❒ Continuing Education
- ❒ TMS OnLine/Web
- ❒ Colleague
- ❒ Other _____

BIRTHDATE:

_____/_____/_____

Members automatically receive a monthly print subscription to JOM.

If you prefer to receive your copy only electronically, please check here. ❒

If you prefer to receive your subscription in both print and electronic formats, please check here. ❒
(You must include $8 extra charge.)

If you would like additional information about member subscriptions and discounts to JOM, Journal of Electronic Materials, or Metallurgical and Materials Transactions A and B, please check here. ❒

FOR OFFICE USE ONLY
ID
BIRTH
ELECTED
TYPE
CATEGORY
SECTION
APPROVED

NAME: _____ (LAST / FIRST / MIDDLE INITIAL)

TITLE: _____

COMPANY OR ORGANIZATION: _____

BUSINESS: _____ (STREET OR P. O. BOX / CITY / STATE / 9 DIGIT ZIP/POSTAL CODE / COUNTRY)

_____ (PHONE / FAX / TOLL FREE # / E-MAIL / WEB ADDRESS)

HOME: _____ (STREET OR P. O. BOX / CITY / STATE / 9 DIGIT ZIP/POSTAL CODE / COUNTRY)

_____ (PHONE / FAX / E-MAIL)

_____ (MONTH / DAY / YEAR)

WHAT IS THE PRIMARY ACTIVITY OF YOUR PLACE OF EMPLOYMENT? (check one)

❒ Commercial Laboratory
❒ Government/Nonprofit Laboratory
❒ Engineered Materials Producer
❒ Manufacturer of Parts/Components
❒ Manufacturer of Finished Products (OEMs)
❒ Primary Metals Producer
❒ Secondary Metals Producer
❒ Producer/Processor of Materials
❒ Educational
❒ Engineering or Consulting Firm
❒ Other _____

WHAT BEST DESCRIBES YOUR PRIMARY JOB FUNCTION? (check one)

❒ Applications/Product Development
❒ Basic Research
❒ Product Engineering and Design
❒ Technical/Lab Management
❒ Process Engineering
❒ Metallurgical Materials Selection
❒ Corporate Management
❒ R & D Engineer
❒ R & D Scientist
❒ R & D Management
❒ Manuf./Production Management
❒ Quality Engineering
❒ Marketing or Sales
❒ Consultant
❒ Educator
❒ Student
❒ Other _____

OTHER SOCIETY AFFILIATIONS: _____

EDUCATION TO DATE:

Name of School	Dates Attended Month/Year–Month/Year	Major Subject/ Engineering Field	Degree Received or Expected Graduation Date: Month/Year
B.S. ❒			
M.S. ❒			
Ph.D. ❒			

REGISTERED PROFESSIONAL ENGINEER? ❒ Yes ❒ No STATE: _____ YEAR OF REGISTRATION: _____

RECORD OF EXPERIENCE:
(List most recent record of employment. If you do not possess a qualifying degree, please include your last seven years of experience.)

From: _____ Title: _____
Company: _____
To: _____ Nature of Company's Business: _____
Total Time with Company: _____
Engineering Responsibilities: _____

TO APPLICANT

If you have been encouraged to submit this application by a current member of TMS, please complete the following information:

Member's Name _____ Member # _____

I agree, if elected, to accept election, and to abide by the TMS bylaws.

Signature _____ Date _____

PREPAYMENT IS REQUIRED (checks should be made payable to TMS in U.S. dollars drawn on a U.S. bank)

❒ Check enclosed
❒ Bill my credit card: (check one)
❒ American Express ❒ VISA ❒ MasterCard ❒ Diners Club

Credit Card # _____ Expiration Date _____

Cardholders Name _____

Signature _____

COMPLETE APPLICATION AND MAIL WITH PAYMENT TO: **184 Thorn Hill Road, Warrendale, PA 15086-7514**
Phone: 800-966-4867 or 724-776-9000 • Fax: 724-776-3770

WEB Take advantage of the convenience of on-line pre-registration via the TMS website: **http://www.tms.org** Web registration requires credit card payment.

FAX Fax this form to TMS Meeting Services **USA 724-776-3770** Fax registration requires credit card payment.

MAIL **Return this form with payment to** Meeting Services TMS 184 Thorn Hill Road Warrendale, PA 15086

TMS2002
131st Annual Meeting & Exhibition

Advance Registration Deadline: January 28, 2002

AM02-PDF

Payment must accompany form.
Forms received past this date will be processed at the on-site fee.
INSTRUCTIONS: Check your selections and fill in the necessary information. *Please print or type.*

Member of: ☐ TMS ☐ ISS ☐ SME ☐ SPE Member Number: _____

This address is: ☐ Business ☐ Home Employer/Affiliation: _____

☐ Dr. ☐ Prof. ☐ Mr. ☐ Mrs. ☐ Ms. Title: _____

_____
LAST NAME FIRST NAME MIDDLE INITIAL

Address: _____

City: _____ State/Province: _____ Zip/Postal Code: _____ Country _____

Telephone: _____ Fax: _____
COUNTRY AREA/CITY LOCAL NUMBER COUNTRY AREA/CITY LOCAL NUMBER

E-Mail Address: _____ Guest/Spouse Name: _____
GUESTS DO NOT RECEIVE ADMISSION TO TECHNICAL SESSIONS.

Registration Fees:

	Advance Fees (until 1/28/02)	On-Site Fees (after 1/28/02)
☐ Member	$395 M	$495 ML
☐ Non-Member Author	$395 NMA	$495 NMAL
☐ Non-Member *	$520 NM	$620 NML
☐ Student Member ##	$0 STU	$0 STUL
☐ Student Non-Member ## *	$25 STUN	$25 STUNL
☐ TMS Retired Member	$220 RM	$220 RML
☐ Exhibit Booth Personnel	$0 E	$0 EL
☐ Exhibit Only	$35 EO	$35 EOL

* Includes TMS membership for 2002
Students must attach a copy of their school's student identification card.

Publication Orders:

ALL pre-ordered books not indicated for shipment MUST be picked up at the Publications Sales area in the convention center.

Qty.	Order Number and Title	Book Price	Shipping*	Total
___	#514X CALPHAD and Alloy Thermodynamics	$72	+_____	=_____
___	#5158 Creep Deformation: Fundamentals and Applications	$105	+_____	=_____
___	#5166 EPD Congress 2002	$96	+_____	=_____
___	#5182 High-Cycle Fatigue, the David L. Davidson symposium	$110	+_____	=_____
___	#5190 Light Metals 2002 (Book and CD-ROM set)	$158	+_____	=_____
___	#5247 Magnesium Technology 2002 (Book and CD-ROM set)	$111	+_____	=_____
___	#5204 Science and Technology of Interfaces, International Symposium in Honor of Dr. Bhakta Rath (PDF)	$122	+_____	=_____
___	#5255 Sulfide Smelting 2002	$70	+_____	=_____
___	#5212 Surface Engineering: Science and Technology II (PDF)	$111	+_____	=_____
___	#5220 High-Performance Metallic Mat'ls for Cost-Sensitive Applications (PDF)	$117	+_____	=_____
___	#5239 Ultrafine Grained Materials II	$125	+_____	=_____
___	#5131 Computational Modeling of Materials Minerals, and Metals Processing	$129	+_____	=_____

* Shipping: in the United States–$10 per item • all other countries–$20 per item

Tutorial Luncheon Lecture Tickets

Optional Box Lunches	Fee	No.	Total
Monday 2/18/02 (sponsored by Young Leaders)			
Young Leaders Tutorial Box Lunch	$25	_____	$_____

Refund policy: Written requests must be mailed to TMS, post-marked no later than January 28, 2002. A $50 processing fee will be charged for all meeting registration cancellations in addition to all short course registrations cancellations.

Continuing Education Short Courses:

	Advance Fees (until 1/28/02)	On-Site Fees (after 1/28/02)
Aluminum Smelter Cell Dynamics		
☐ Member	$645	$695
☐ Non-Member	$735	$785
Texture and Anistrophy for the Materials Professional		
☐ Member	$395	$475
☐ Non-Member	$445	$525
Computational Modeling Techniques and Applications for Materials Processing		
☐ Member	$395	$475
☐ Non-Member	$445	$525

Social Function Tickets:

	Fee	No.	Total
Monday 2/18/02			
Bhakta Rath Honorary Dinner	$60	_____	$_____ RD
Charles McMahon Honorary Dinner	$60	_____	$_____ MD
David L. Davidson Honorary Dinner	$35*	_____	$_____ DD
* A portion of each ticket is being subsidized by the organizing committee			
Tuesday 2/19/02			
TMS Banquet	$60	_____	$_____ AD
Tables of 8	$480	_____	$_____ AD8
Table Sign to Read _____			
Extraction & Processing Division Luncheon	$30	_____	$_____ EP
Tables of 8	$240	_____	$_____ EP8
Table Sign to Read _____			
Wednesday 2/20/02			
Light Metals Division Luncheon	$30	_____	$_____ LM
Tables of 8	$240	_____	$_____ LM8
Table Sign to Read _____			

Plant Tour:

Thursday 2/21/02	Fee	No.	Total
Boeing 747/767/777	$39	_____	$_____BT

2002 Membership Dues—for current TMS members only:

☐ Full Member	$90 FM
☐ Junior Member	$55 JM
☐ ASM/TMS Joint Student Member	$25 ST

TOTAL FEES PAID ..... $_____

Payment enclosed:

☐ Check, Bank Draft, Money Order

Make checks payable to TMS. Payment shall be made in US dollars drawn on a US bank.

☐ Credit Card Expiration Date: _____

Card No.: _____

☐ Visa ☐ MasterCard ☐ Diners Club ☐ American Express

Cardholder Name: _____

Signature: _____

TMS2002

131st Annual Meeting & Exhibition

February 17-21, 2002 • Seattle, Washington, USA

Making your reservation is easier than ever through Travel Planners' real-time Internet reservation system! Just log on to www.tms.org, and follow the link to Travel Planners. You will be able to view actual availability, learn about your hotel's features and services, and obtain local city and sightseeing information. Most importantly, you will receive instant confirmation of your reservation!

Or, mail or fax this housing form to:
Travel Planners, Inc., 381 Park Ave. South,New York, NY 10016
FAX: 212-779-6128 • PHONE: 800-221-3531, ext. 1 (in 212, 718, 516, 914, 631 or international call 212-532-1660, ext. 1)
(CHOOSE ONLY ONE OPTION)

Reservations must be received at Travel Planners by: January 18, 2002

Arrival Date _____ Departure Date _____

Last Name _____ First Name _____ MI _____

Company _____

Street Address _____

City _____ State/Country _____ Zip/Postal Code _____

Daytime Phone _____ Fax _____

Additional Room Occupants _____

E-mail _____ (confirmation will be sent via e-mail if address is provided)

Non-Smoking Room Requested _____ Special Needs _____

 # after hotel correspondes with map below.

HEADQUARTERS
Sheraton Hotel • 1
$175/single • $175/double

W Hotel Seattle • 7
$179/single • $199/double

Renaissance Madison • 5
$149/single • $149/double

West Coast/Grand • 8
$135/single • $135/double

Hilton Seattle • 3
$140/single • $140/double

Mayflower Park Hotel • 4
$140/single • $140/double

West Coast/Roosevelt • 9
$132/single • $132/double

Westin Seattle • 11
$165/single • $165/double

Crowne Plaza Seattle • 2
$134/single • $134/double

Summerfield Suites Wyndham • 6
$119/single • $129/double

West Coast/Vance • 10
$125/single • $125/double

Indicate 1st, 2nd, & 3rd hotel choice:

1. _____
2. _____
3. _____

Type of Accomodations: (check one)
☐ Single 1 person/1bed ☐ Double 2 people/1bed ☐ Twin 2 people/2 beds
☐ Triple 3 people/2 beds ☐ Quad 4 people/2 beds

If all three (3) requested hotels are unavailable, please process this reservation according to: (check one) ☐ ROOM RATE ☐ LOCATION

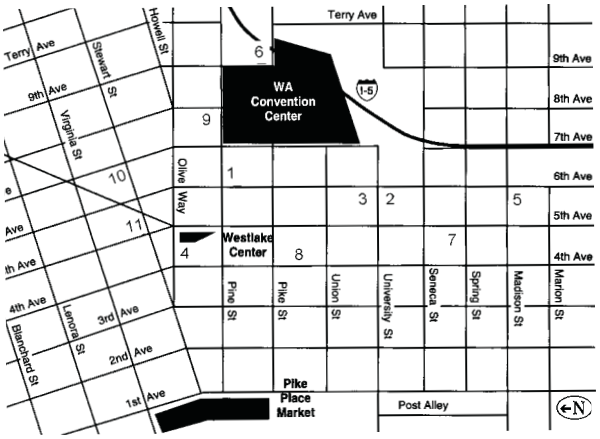
TMS has contracted a block of rooms at the headquarters hotel, Sheraton Seattle Hotel & Towers, along with each of the hotels, and therefore has assumed a financial liability for any and all rooms in that block that are not reserved. You are strongly encouraged to reserve your room(s) at the hotels listed to limit our financial liability. Please help TMS achieve overall success with the 131st TMS Annual Meeting & Exhibition by making your reservation at one of the listed hotels prior to the advance housing deadline. Thank you.

Confirmations: Confirmation will be e-mailed, faxed or mailed to you from Travel Planners, Inc. once your reservation has been secured with a deposit. You will not receive a confirmation from your hotel. If you do not receive a confirmation within 7 days, please call Travel Planners, Inc.

Changes/Cancellations: All changes and cancellations in hotel reservations must be made with Travel Planners, Inc. up until 3 business days prior to arrival and are subject to the individual hotel's cancellation policies. Cancellations and changes within 3 days of arrival MUST be made with your hotel directly. Many hotels are now imposing fees for early departure. This rate is set by each hotel and may vary accordingly. Please reconfirm your departure date at the time of check-in.

Reservations/Deposits: All reservations are being coordinated by Travel Planners, Inc. Arrangements for housing must be made through Travel Planners, Inc. and NOT with the hotel directly. Reservations via Internet, phone or fax will be accepted with a major credit card only. Housing forms and written requests will be accepted with a major credit card or deposit of one night's room and tax payable to Travel Planners, Inc. Check must be drawn in US funds on a US bank. No wire transfers will be accepted. Deposit policies are set by each hotel, and are outlined on your hotel confirmation.

Please read all hotel information prior to completing and submitting this form to Travel Planners, Inc. Keep a copy of this form. Use one form per room required. Make additional copies if needed.



Payment enclosed: ☐ Check ☐ American Express ☐ MasterCard ☐ VISA ☐ Discover ☐ Diners

Account Number _____ Expiration Date _____

Card Holder Name _____ Authorized Signature _____

ACCOMPANYING PERSONS TOUR REGISTRATION FORM

TMS2002
131st Annual Meeting & Exhibition

February 17-21, 2002 • Seattle, Washington, USA

Convention Services Northwest has arranged tours for attendees/guests.

Please make your reservation by noting choice of tour. Pre-sold tickets will be held at the tour desk located in Hall 4C of the Washington State Convention and Trade Center. Please arrive 15 minutes prior to departure time.

Registration:

To register for the tours, please complete and return the registration form along with full payment to:
Convention Services Northwest, Attn: Becky Haiduck
Tower Building/Suite 1414,1809 Seventh Avenue, Seattle, WA 98101, USA
PHONE: (206) 292-9198 • FAX: (206) 292-0559
*** PLEASE CHOOSE ONLY ONE OPTION FOR SENDING FORM ***

Description	Date/Time	Price	No.	Amount
Sample Seattle – A Deluxe City Tour	Monday, February 18, 2002 – 9:00am-12:30pm	$28	_____	$_____
Historic Port Townsend Day Trip	Tuesday, February 19, 2002 – 9:00am-4:30pm	$59	_____	$_____
Wine Tasting Tour	Wednesday, February 20, 2002 – 1:00pm-5:00pm	$39	_____	$_____

Total $_____

Last Name _____ First Name _____ MI _____

Street Address _____

City _____ State/Country _____ Zip/Postal Code _____

Daytime Phone _____ Fax _____

E-mail _____

☐ I have special needs, disabilities or dietary concerns. Please have someone from Convention Services Northwest contact me.

Payment options:

☐ Check or Money Order Enclosed (remit in US funds)

☐ American Express ☐ MasterCard ☐ VISA ☐ Discover ☐ Diners

Account Number _____ Expiration Date _____

Card Holder Name _____ Authorized Signature _____

Reservations:

Reservations must be received by January 16, 2002. Tickets may be purchased on-site on a space-available basis only and tickets will include an additional $2.00 charge. You may pick up your tickets at the Washington State Convention and Trade Center in Hall 4C staring at 11:00am on Sunday, February 17, 2002.

Refunds:

Refunds for cancellations will be made if written request is received at the Convention Services Northwest office by January 16, 2002, less a $5.00 handling fee per ticket. After January 16, 2002, no refunds or exchanges can be made. If minimum registration on a tour is not met, Convention Services Northwest will refund the cancelled tour or apply the refund towards another tour.

For Office Use Only:

Deposit No. _____ Amount $ _____

Check No. _____ Inn. _____

CONFERENCE PROCEEDINGS

THE FOLLOWING TITLES WILL BE AVAILABLE AT THE MEETING. RESERVE YOUR COPY USING THE TMS ANNUAL MEETING REGISTRATION FORM.

CALPHAD and Alloy Ther modynamics

Patrice E.A. Turchi, Antonios Gonis, and Robert D. Shull, editors

These proceedings emphasize all theoretical aspects of computational thermodynamics and kinetics and their impact on the science of alloys and materials design. The book will provide an assessment of the CALPHAD (Calculation of Phase Diagrams) approach pioneered by 2002 Hume-Rothery Award recipient Larry Kaufman, a review of the current status of the software applications based on the CALPHAD approach, the impact of CALPHAD on alloy thermodynamics and design, and future prospects. The book will provide a detailed picture of the development of CALPHAD and point to further research.

ISBN 0-87339-514-X

Approx. 390 pp., illus., index, hardcover

Order No. 514X

Member price: $72

Computational Modeling of Materials, Minerals, and Metals Processing

M. Cross, J.W. Evans, and C. Bailey, editors

This set contains the proceedings, in both print and CD-ROM formats, of the Computational Modeling of Materials, Minerals, and Metals Processing Symposium to be held at the 2002 TMS Annual Meeting in Seattle, Washington.

Computational models offer an effective way to design, analyze, and optimize materials, metals, and minerals processing. Computational modeling is increasingly becoming the means by which every factor in the operation of a process can be analyzed in a rational manner. Such models also represent a framework for much of our understanding of process operations.

This international symposium brings together participants from all aspects of computational modeling of materials, minerals, and metals processing, including the engineers involved primarily in specific applications, developers of computational modeling tools, and the developers of models. This allows all involved to understand the most recent advances in this rapidly developing enabling technology.

ISBN 0-87339-513-1

Approx. 700 pp., illus., index, hardcover & CD-ROM

Order No. 5131

Price: $129

Creep Defor mation: Fundamentals and Applications

Rajiv S. Mishra, James C. Earthman, and Sai V. Raj, editors

These proceedings focus on the application of fundamental creep research to the design and development of high temperature materials for engineering applications. The book deals with all aspects of creep deformation and high temperature materials development, specifically the influence of microstructures on various aspects of creep and the application of this information in the design of highly creep resistant materials. Emphasis will be placed on advanced ceramic and metal matrix composites and advanced intermetallics.

This volume brings together researchers working on fundamental issues relating to the development and characterization of high temperature materials and design engineers involved in high temperature applications.

ISBN 0-87339-515-8

Approx. 415 pp., illus., index, hardcover

Order No. 5158

Member price: $105

Light Metals 2002

Wolfgang Schneider, editor

Held at the TMS Annual Meeting & Exhibition each year, the Light Metals series has become the definitive annual reference source in the field of aluminum production and related light metals technologies. Each volume contains complete coverage of advancements and current work in cast shop technology, alumina and bauxite, carbon technology, aluminum reduction technology, and recycling. In addition, Light Metals 2002 includes coverage of reactive metals and advances in molten salt processing technology. Light Metals 2002 is sold as a package that includes the proceedings in both hardcover and text-searchable CD-ROM formats.

ISBN 0-87339-519-0

Approx. 1200 pp., illus., index, hardcover & CD-ROM

Order No. 5190

Member price: $158

EPD Congr ess 2002

Patrick R. Taylor, editor

The Extraction & Processing Division Congress, held at the TMS Annual Meeting & Exhibition each year, has become the definitive annual forum for new technological developments in the process metallurgy community. This volume will also include the proceedings of the Fundamentals of Advanced Materials for Energy Conversion symposium.

In addition to general abstracts, this year's edition will include papers on:

- Modeling of high temperature alloy processing
- General pyrometallurgy
- Generation, treatment, metal recovery, and disposal of flyash
- Imaging of dynamic processes
- Materials processing fundamentals
- General recycling of materials

ISBN 0-87339-516-6

Approx. 812 pp., index, hardcover

Order No. 5166

Member price: $96

High-Cycle Fatigue, the David L. Davidson Symposium

Kwai S. Chan and Peter K. Liaw, editors

These proceedings from the David L. Davidson Symposium on High-Cycle Fatigue review the current research activities and assess the state-of-the-art in high-cycle fatigue, which has become a major concern in the design of engineering components and structures. This volume will include papers on a basic understanding of failure mechanisms, experimental methods and studies, modeling and simulation, and life-prediction methodology. This volume will also contain papers from the Fatigue of High Temperature Alloys Symposium, which deals with fatigue behavior of high temperature alloys, including crack initiation and propagation modes.

ISBN 0-87339-518-2

Approx. 350 pp., index, hardcover

Order No. 5182

Member price: $110

High Per formance Metallic Materials for Cost-Sensitive Applications

F.H. (Sam) Froes and Lu Li, editors

These proceedings will examine the most recent advances and best practices in structural materials selection, design, and manufacturing for producing affordable components, with a focus on titanium, aluminum, and other advanced metallic materials. This volume will discuss melting, casting, powder metallurgy, forging, forming, extrusion, and machining, as well as processing advances, innovative processing techniques, process modeling and materials by design, new alloys, and related processing-microstructure-properties-performance-cost studies.

ISBN 0-87339-522-0

Approx. 295 pp., index, PDF publication

Order No. 5220

Member price: $117

Sulfide Smelting 2002

Robert L. Stephens, and H.Y. Sohn, editors

These proceedings focus on all aspects of the pyrometallurgical production of primary metals from sulfide concentrates. Papers discuss industrial operations producing copper and nickel and the direct production of lead and zinc. Key topic areas include smelting and converting processes, recently completed capital projects, current operating practices, the predicted future of sulfide smelting operations, furnace integrity and refractory design, gas handling processes and equipment, issues related to the treatment of high-strength sulfur dioxide-containing off-gases, the production of alternative sulfur products, the treatment of acid plant blowdown streams and sulfated smelter dusts, and the capture and treatment of fugitive emissions.

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Advances in Metallic Glasses: Glass Formation

Sponsored by: Electronic, Magnetic & Photonic Materials Division, *Program Organizers:* K. F. Kelton, Washington University, Department of Physics, St. Louis, MO 63130 USA; A. L. Greer, University of Cambridge, Department of Materials Science & Metallurgy, Cambridge CB2 3QZ UK; Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA

Monday AM Room: 212
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: A. L. Greer, Cambridge University, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK; Matthew J. Kramer, Iowa State University, 37 Wilhelm Hall, Ames, IA 50011-3020 USA

8:30 AM Invited
Formation of (Fe,Ni)-Based and Al-Based Structural Amorphous Metals: *S. Joseph Poon*[1]; Gary J. Shiflet[2]; Faqiang Guo[1]; V. Ponnambalam[1]; [1]University of Virginia, Phys., Charlottesville, VA 22904 USA; [2]University of Virginia, Matls. Sci. & Eng., Charlottesville, VA 22903 USA

This talk will focus on the formability, stability, and some of the physical properties of metallic glasses in several ferrous-based and light-metal-based multinary alloy systems. Ferrous-based alloys will include those that contain Fe, Ni, Mo, and Cr; light-metal alloys will contain Al and Mg as the major components. The ferrous-based metallic glasses, which are non-ferromagnetic at ambient temperatures, can be compared with several types of austenitic steels. Magnetization and susceptibility results will be presented. The formability factors for forming bulk metallic glasses will be discussed in light of experiment and atomistic model.

9:00 AM
Multicomponent (Ti Zr Hf Nb)-(Ni Cu Ag)-Al Glasses: Ki-Buem Kim[1]; Paul Warren[1]; *Brian Cantor*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

A wide range of new metallic glasses has been developed in the (Ti Zr Hf Nb)-(Ni Cu Ag)-Al system by a novel equiatomic substitution technique for the early and late transition metal components, starting from the well known Zr-Cu-Al system. The thermal stability and crystallisation of these glasses has been studied by x-ray diffractometry, differential scanning calorimetry, transmission electron microscopy and atom probe analysis. This paper describes the resulting insight into the behaviour of these novel multicomponent metallic glasses.

9:20 AM
A Topological Model for Metallic Glass Formation: *Oleg N. Senkov*[1]; Daniel B. Miracle[2]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA

An approach based on analysis of atomic size distributions has been developed and applied to multicomponent amorphous alloys with different glass forming ability. The atomic size distributions were obtained by plotting atomic concentrations versus atomic radii of constitutive elements. Amorphous alloys with high critical cooling rates were found to have single-peak distributions with a concave downward shape. These amorphous systems have at least one alloying element with a smaller radius and at least one alloying element with a larger radius relative to the base element. The concentration of an alloying element decreases rapidly as the difference in the atomic sizes of the base element and the alloying element increases. Atomic size distributions of Zr, Pd, or RE -based bulk amorphous alloys, which have low critical cooling rates, have a completely different, concave upward shape with a minimum concentration at an intermediate atomic size. A topological model that explains the concave upward shape of atomic size distributions for the bulk amorphous alloys has been suggested.

9:40 AM
Atomistic Simulations of the Phase Stability and Elastic Properties of the Nickel-Zirconium Alloy System: *Frank J. Cherne*[1]; Michael I. Baskes[1]; Ricardo B. Schwarz[1]; [1]Los Alamos National Laboratory, MST-8, PO Box 1663, MS G755, Los Alamos, NM 87545 USA

Numerous simulations of binary amorphous Zr-Ni alloys have been performed utilizing the potential formalism of Massobrio, Pontikis, and Martin (MPM). Here we present a thorough study of the phase stability and the elastic properties of both the amorphous and the crystalline nickel-zirconium alloys utilizing the modified embedded-atom method (MEAM) approach. We will compare and contrast MEAM with MPM. The MEAM potentials developed predict the correct crystal structures for NiZr and $NiZr_2$. The nickel-zirconium system was chosen primarily due to the complexity of the phase diagram and its glass forming ability. Furthermore, we are interested in examining the compositional dependence of the shear modulus. We will also present the free energies as a function of composition and temperature for both amorphous and crystalline phases.

10:00 AM
Stability of Undercooled $Pd_{40}Ni_{40-x}Cu_xP_{20}$ Liquids: *T. D. Shen*[1]; Ulrich Harms[1]; Ricardo B. Schwarz[1]; [1]Los Alamos National Laboratory, Struct./Prop. Relations Grp., MS G755, Los Alamos, NM 87545 USA

The question of what determines the stability of undercooled liquids remains unanswered. In the $Pd_{40}Ni_{40-x}Cu_xP_{20}$ system, the best bulk glasses are found for x=0 and x=30. Properties of the glass, such as shear modulus, hardness, and width of the first maximum in the X-ray diffraction pattern, show extreme values at approximately x=10, which we attribute to strong short-range ordering at that composition. We have found that the stability of the undercooled liquids correlates best with the difference $\Delta V=Vg-Vc$ between the molar volumes of the glassy and crystalline states. For $0<x<30$, ΔV is positive, whereas for $30<x<40$, ΔV is negative. Both positive and negative values of ΔV decrease Tx-Tg. The minimal in $|\Delta V|$ at x=0 and x=30 correspond to the largest values of Tx-Tg. A positive ΔV implies a negative pressure around growing crystalline embryos and this could explain the observed decrease in Tx-Tg for $x<30$.

10:20 AM Break

10:40 AM Invited
Atom Probe Studies of Metallic Glasses: *Michael K. Miller*[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Bldg. 4500S, MS 6136, Oak Ridge, TN 37831-6136 USA

The distribution of the atoms in bulk metallic glasses in both the as-prepared condition and after heat treatments in the region of the glass transition temperature provides important information on the stability of the alloy. The spatial coordinates and the elemental identities of the atoms in the amorphous alloy may be determined with near atomic resolution by atom probe tomography. The three-dimensional data collected by this technique can be analyzed to determine the local atomic configuration of each atom type and thereby estimate the tendency for solute clustering and chemical short range order. These data can be also divided into small volume elements that may be statistically analyzed to detect and quantify concentration fluctuations due to phase separation, precipitation and crystallization. A review of the decomposition of a number of different types of bulk metallic glasses will be presented. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

11:10 AM
EFTEM Study of Compositional Variations in Mg-Ni-Nd Bulk Metallic Glasses: *S. V. Madge*[1]; D. T.L. Alexander[1]; A. L. Greer[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

A range of Mg-Ni-Nd bulk metallic glasses has been cast. Around the composition $Mg_{60.5}Ni_{24}Nd_{15.5}$ (at.%), but not at neighbouring compositions, energy-filtered TEM provides evidence for strong compositional inhomogeneities in the as-cast glass. In this fully glassy material, an apparent two-phase cellular microstructure is seen; one phase is rich in Ni, the other in Mg and Nd. Small-angle X-ray scattering

offers additional evidence for phase separation in the glassy state. Annealing can eliminate the inhomogeneities, and possible explanations for the phenomena are considered, including the role of oxygen.

11:30 AM
Nanoscale Phase Separation and Local Icosahedral Order in Amorphous Alloys of Immiscible Elements: *Evan Ma*[1]; [1]Johns Hopkins University, Matls. Sci. & Eng., Baltimore, MD 21218 USA

We explore the nature of amorphous alloys formed by nonequilibrium processing in immiscible systems, to uncover their differences from those well-studied metallic glasses in systems with negative heat of mixing. We demonstrate spinodal compositional modulations with nanometer wavelengths, along with local icosahedral ordering, in amorphous structures formed in the immiscible Ag-Ni system. This unique atomic-level structure allows the amorphous alloy to lower its enthalpy such that the heat of crystallization is of a magnitude comparable to those typically seen for metallic glasses in easy glass forming systems. The development of local structure has been monitored using EXAFS[1], TEM, XRD, and magnetic measurements, as well as MD simulations[2,3], and correlated with the enthalpy states measured using DSC. [1]Phys. Rev. Lett. 83, 2826 (2001). [2]Appl. Phys. Lett. 78, 1343 (2001). [3]Phys. Rev. B Oct. 1 issue (2001).

Advances in Molten Salt Processing Technology: New Electrochemical Methods

Sponsored by: Light Metals Division, Reactive Metals Committee
Program Organizer: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division, Argonne, IL 60439-4837 USA

Monday AM — Room: 614
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Sean M. McDeavitt, Argonne National Laboratory, Cheml. Tech. Div., Argonne, IL 60439 USA

8:30 AM Opening Remarks

8:35 AM
New Advances in Molten Salt Research: Data for Pyrochemical Reprocessing of Nuclear Fuels: *Marcelle Gaune-Escard*[1]; [1]Universite de Provence, CNRS UMR 6595, IUSTI, Technopole Chateau-Gombert, 5 rue Enrico Fermi, Marseille 13453 France

Lanthanide and actinides halides and their mixtures with alkali halides play a major role in the processing of nuclear waste and recycling of spent nuclear fuel. However, reference data are missing both for the pure salts and the mixtures, whatever the halides (chlorides or fluorides) under consideration. However the related data on molten lanthanide and actinide salts (and compounds), are not easily available. Because of characteristic physicochemical properties, which do not promote experimental investigations, data are scarce. When they exist, most of the data is generally buried in grey literature. Also, when available, they reveal to arise from estimations. Coordination and standardization of existing data is therefore essential, as research efforts should be intensified together with the development of numerical prediction tools. This paper will, firstly, discuss new experimental data on lanthanide- and actinide-based halide systems. These are obtained through an intensive international research cooperation and multiinstrumental methods. It will be shown that data can be obtained not only from these physicochemical measurements (thermodynamic, electrical, electrochemical, structural), but also from numerical simulations based on structural features and proper modeling. Secondly, ongoing efforts for the development of a molten salt database will be presented.

9:05 AM
Direct Electrochemical Reduction of Niobium Pentoxide to Niobium Metal in a Eutectic of $CaCl_2$-NaCl Melt: Xiao Yong Yan[1]; *Derek J. Fray*[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

A novel electrochemical approach is being developed using the method of the electrodeoxidation to reduce solid Nb_2O_5 directly to niobium metal in a eutectic melt of $CaCl_2$-NaCl at temperatures around 900°C. Electrolysis was carried out at the applied constant voltage of 3.1 V, below the decomposition potentials of the chloride melt. After electrolysis, the oxide cathode, pellets of Nb_2O_5, transformed into porous pellets of metallic niobium that could be ground manually into niobium powders with a wide range of niobium particle sizes. In addition to the niobium powders, the niobium sponge has a similar structure to that of Kroll titanium sponge and was readily prepared by controlling the conditions for pellet-making and electrolysis. The niobium metal prepared under the present experimental conditions contained as low as 3000 mass ppm oxygen. The results obtained clearly demonstrated that this direct solid-state electrochemical reduction of Nb_2O_5 is an efficient, low cost, and environmentally friendly process compared with the existing conventional reduction processes, and it can be potentially an electrolytic reduction process for the production of niobium metal.

9:35 AM
Electrochemical Reduction of Metal Oxides in Molten Salts: *Karthick V. Gourishankar*[1]; Laszlo Redey[1]; Mark A. Williamson[1]; [1]Argonne National Laboratory, Cheml. Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA

Argonne National Laboratory (ANL) has demonstrated a direct electrochemical reduction process for converting uranium oxide to uranium metal. The reduction process is versatile and can be extended to the extraction of other actinide and rare-earth metals from their oxides. Although UO2 reduction has been demonstrated in molten LiCl-Li2O electrolyte at 650ºC, the choice of electrolyte and process temperature depends on the oxide charge. For example, the reduction of rare-earth oxides requires CaCl2-CaO melts at 800-900ºC. However, the fundamental mechanism underlying the technology is the same. The oxygen in the oxide charge is ionized into a soluble species at the cathode, leaving behind the reduced metal, and the oxide ion is transported to the anode, where it is converted to oxygen gas. The electrochemical cell consists of a non-consumable anode and a cathode that is configured to contain the oxide charge. Independent reference electrodes are used to monitor the anode and cathode potentials. The cell is operated under constant current or constant potential conditions. This paper will discuss the cell design and operation, and will present results from the UO2 reduction experiments.

10:05 AM Break

10:20 AM
Synthesis of Pb-Ca Alloys by Electrolysis of CaO Solution in Molten Salt: *Derek J. Fray*[1]; Jane Freidina[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

In the search for an environmentally friendly, efficient technology for calcium addition to lead alloys a study of calcium electrolysis from molten salts was performed. The research showed that calcium can be electrowon to a liquid lead cathode at temperatures below 923 K. Alloys containing up to 2 wt.% Ca were synthesised from CaCl2-NaCl-CaO mixtures with a current efficiency of 40-60%. Sodium impurity content was as low as one tenth of the calcium content. The effects if factors, such as temperature, current density, time of electrolysis on the performance of the cell and the quality of the alloys were investigated.

10:50 AM
Development of High Throughput Uranium Electrorefining: *James L. Willit*[1]; Eddie C. Gay[1]; [1]Argonne National Laboratory, Cheml. Tech., 9700 S. Cass Ave., Argonne, IL 60439-4837 USA

At the heart of Argonne's electrometallurgical treatment technology for spent metallic reactor fuel is an electrorefining step in which chopped spent fuel is loaded into a basket, and submerged in a pool of molten salt. A current passed between the basket (anode) and a cathode oxidizes the uranium and all active metal fission products forming soluble metal chlorides. At the cathode, uranium, the most noble of the dissolved metal chlorides, is electrodeposited. This basic concept has been scaled up from the laboratory scale to a device that holds ~100 kg of chopped fuel. The presentation will consist of a brief review of the basic electrochemistry of uranium electrorefining as well as the engineering challenges encountered during the scale-up of the technology.

11:20 AM
High-Temperature Electroforming in Molten Salts: Joseph Biondo[1]; *Anatoliy Shchetkovskiy*[1]; Alexander Smirnov[1]; Vitaly Sikin[1]; [1]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

Electroforming is a viable process for manufacturing near-net shapes of product. A number of metals can not be electrodeposited from water solution, but can successfully be obtained with electrolysis of the molten salts. Electroforming such metals as iridium, rhenium, niobium and platinum-iridium alloy is used by Engelhard-CLAL for commercial application under the name EL-Form™ process. High-temperature electroforming is based on electrodeposition of metals onto mandrel of required shape. After electrodeposition, the mandrel is removed.

The electroforming in molten salts provides seamless design of items, efficient metal use and metal purification during electrolysis. High uniformity and tight tolerances can be achieved especially for axisymmetrycal items such as tubes, crucibles and nozzles. Advanced production equipment allows the electroforming of items with a maximum diameter of 8.5" and maximum length of 15". The necessary process control provides stable and reproducible properties of the electroformed metals. Various examples of electroformed product will be discussed.

Alumina and Bauxite: Bauxite Digestion

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Jacques M. Mordini, Aluminium Pechiney, Gardanne, Cedex 13541 France; Steve Rosenberg, Worsley Alumina Pty, Ltd., Process Chemistry Group, Collie, West Australia 6225 Australia; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Monday AM Room: 609
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Jacques Mordini, Aluminium Pechiney, Direction Industrielle, Boite Postale 54, Gardanne Cedex 13541 France; Steven P. Rosenberg, Worsley Alumina Pty Ltd., Proc. Chem. Grp., PO Box 344, Collie, WA 6225 Australia

8:30 AM Introductory Remarks

8:40 AM
Improved Preparation of the Greek Diasporic Bauxite for Bayer-Process: *Dimitris Papanastassiou*[1]; Barnabás Csõke[2]; Károly Solymár[3]; [1]Silver&Baryte Ores Mining Co. S.A., Bauxite Div., 21 Amerikis str., Athens GR-10 672 Greece; [2]University of Miskolc, Dept. of Proc. Eng., Miskolc-Egyetemváros, Miskolc H-3515 Hungary; [3]ALUTERV, Ltd., Chief Techl. Adviser, Fehérvári út 144., Budapest H-1116 Hungary

The Greek diasporic bauxite plays an important role in the medium- and long-term bauxite supply of Europe. The chemical and mineralogical composition and the textural and physical properties of the high-grade, but hard and by calcite contaminated bauxite of S&B will be presented. The grindability lab tests and the industrial operation of AdG confirmed the important role of the lime dose as a grinding aid in hot caustic liquor, which can reduce the energy requirement of the mill by about 50% in comparison with the dry grinding. The calcite removal has been solved at S&B by "Dynamic Heavy-Media Separation". Plant-scale abrasion tests carried out in tube-in-shell preheaters of the tube digester at MOTIM Works Hungary did not indicate any measurable abrasion. So, the hard Greek diasporic bauxite can effectively and economically be prepared.

9:05 AM
Digestion Alternatives of the Greek Diasporic Bauxite: *Károly Solymár*[1]; Tibor Ferenczi[2]; Dimitris Papanastassiou[3]; [1]ALUTERV, Ltd., Chief Techl. Adviser, Fehérvári út 144., Budapest H-1116 Hungary; [2]ALU-LAB, Ltd., Managing Dir., Fehérvári út 144., Budapest H-1116 Hungary; [3]Silver&Baryte Ores Mining Co. S.A., Bauxite Div., 21 A, Amerikis str., Athens GR-10 672 Greece

Optimum digestion parameters (lime dosage, retention time and final A/C) of the S&B diasporic bauxite have been determined by lab simulation tests between 240 and 260°C. Co-grinding of bauxite and lime and lime addition to the slurry during predesilication resulted in similar advantageous effect on alumina yield and kinetics. Lime feeding at the final digestion temperature was found as the most effective. The required lime dose depends on the quantity and stability of the diaspore in the process bauxite and the digestion temperature. The results are in good agreement with the AdG practice and operational experiences of other European alumina refineries, achieved by processing S&B bauxite. The tests confirmed that high alumina yield and adequate cycle efficiency(high A/C ratio in blow-off liquor) can be achieved by well selected digestion parameters at 240°C already.

9:30 AM
Intensifying Method of Bayer Digestion Process of Diasporic Bauxite: *Yin Zhonglin*[1]; Gu Songqing[1]; [1]Zhengzhou Light Metal Research Institute, Shangjie, Zhengzhou, Henan 450041 China

The indirect preheating process of bauxite slurry is necessary for intensifying the Bayer digestion of diasporic bauxite. However, scale formation on indirect heating surfaces is unavoidable in single-stream digestion technology. In this paper a dual-stream and lime Bayer process is proposed, that combines the new dual-stream digestion process with the lime Bayer digestion process. The proposed process solves a series of key problems associated with indirect preheating technology and intensifies the digestion of diasporic bauxite. As a result, soda consumption can be reduced and the digestion process accelerated, allowing advanced technological and economical indexes to be obtained.

9:55 AM Break

10:15 AM
New Process Technologies of Bauxite Concentrate-Tube Digestion for Alumina Production: *Wangxing Li*[1]; *Shaolun Wang*[1]; Peikai Song[1]; Yongxing Hou[1]; [1]China Great Wall Aluminium, Tech. Dept., No 28, Changqian Rd., Shangjie Dist., Zhengzhou, Henan 450041 China

This paper provides a brief description of the bauxite reserves and status of alumina production in China. It then gives the results of studies carried out to solve the key technical problems involved in the processing of diasporic bauxite. A new process of diasporic bauxite concentrate preparation associated with tube digestion is presented that strides over the existing technology in terms of better bauxite usage, and lower energy, raw material and equipment requirements.

10:40 AM
Digestion of Diasporic Bauxite with Mass Ratio of Al_2O_3/SiO_2 No Greater than 7 by Bayer Process with an Excessive Addition of Lime: *Zhao Hengqin*[1]; Hu Hongjie[1]; Jin Mei[1]; Li Jie[2]; Li Qingyu[2]; Liu Yexiang[2]; [1]National Engineering Research Center for Multipurpose Utilization of Non-Metallic Mineral Resource, Metall. & Cheml. Eng., No. 328 Longhai W. Rd., Zhengzhou, Henan 450006 China; [2]Central South University, Dept. of Metlgcl. Sci. & Eng., Changsha, Hunan 410083 China

It is usually believed that digestion of the diasporic bauxite with mass ratio of Al_2O_3/SiO_2 no greater than 7, which constitutes over 70 percent of China proved bauxite reserves, by adopting ordinary Bayer Process, is uneconomically viable because of consumption of higher caustic soda. At higher digestive temperature simultaneously with a more addition of inexpensive lime than ordinary Bayer Process, the bauxite can be economically digested by Bayer Process. Leaching rate of alumina from the bauxites can reach over 85% after 60 minutes of digestion at 280°, with Na_2O_k 220g/l of a sodium aluminate liquor and with lime addition of over 16%wt. of bauxite. The content of Na_2O in the produced red mud is under 2.5%wt. The XRD analysis results of the red mud indicate that an excessive addition of lime causes big changes of constituents of red mud. The improved Bayer Process is of significance for exploitation of China bauxite.

11:05 AM
Thermal Behaviors of Kaolinite-Diasporic Bauxite and Desilication from it by Roasting-Alkali Leaching Processing: *Tao Jiang*[1]; Guanghui Li[2]; Zhucheng Huang[2]; Xiaohui Fan[2]; Guanzhou Qiu[2]; [1]University of Utah, Dept. of Metlgcl. Eng., Rm. 412, WBB, 135 S. 1460 East, Salt Lake City, UT 84112 USA; [2]Central South University, Dept. of Minl. Eng., Changsha 410083 China

By means of XRD, TG and DTA techniques, the thermal behaviors of kaolinite-diasporic bauxite are investigated in this paper. Results show that the kaolinite in the bauxite loses its constitutional water and turns into meta-kaolinite when temperature is elevated above 450°C. The meta-kaolinite is decomposed into amorphous silica and non-crystal alumina in the range of 990-1100°C, but latter two compose into mullite if the temperature exceeds 1100°C. It is found that diaspore is changed into a-alumina with the removal of its constitutional water at the temperature over 650°C. A desilication process of roasting-alkali leaching for the bauxite is developed on the basis of the results. The optimum roasting parameters are attained to be a grain size of 0~20mm, a roasting temperature of 1050~1100°C, and a roasting time of 15~20 minutes. The proper leaching conditions are found as 100 g/l Na2Ok and 140°C. By alkali leaching of the roasted bauxite, a concentrate of 9.92 A/S ratio is achieved for a raw bauxite ore with a 4.4 A/S ratio. It is found the silica removed by the roasting-alkali leaching processing is mainly the amorphous silica and the reaction of mullitization causes reduction in desilication. The a-alumina yielded by diaspore decomposition during roasting is not dissolved under the alkali leaching conditions. The Bayer leaching tests of the concentrate show that the desilication by roasting-alkali leaching processing improves the digestion properties of Al2O3 in the bauxite.

Aluminum Reduction Technology: Fundamentals

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Martin Segatz, VAW Aluminum AG, D-53117, Bonn Germany; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Monday AM Room: 6B
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chair: Geir Martin Haarberg, Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., Sem Saelands vei 6, Trondheim NO-7491 Norway

8:30 AM
The Role of Dissolved Metal during Electrodeposition of Aluminium from Cryolite-Alumina Melts: *Geir Martin Haarberg*[1]; Jomar Thonstad[1]; Stanislaw Pietrzyk[2]; James J. Egan[3]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., Sem Saelands vei 6, Trondheim NO-7491 Norway; [2]University of Mining and Metallurgy, Dept. of Metall. of Non-Ferrous Metals, Krakow 30-059 Poland; [3]Brookhaven National Laboratory, Upton, NY 11973 USA

In molten cryolite in contact with aluminium dissolved aluminium as well as dissolved sodium are formed. Dissolution of sodium gives rise to the formation of mobile electrons. Ac resistance measurements were performed to determine the electronic conductivity in cryolite-alumina melts as a function of temperature, electrolyte composition (AlF_3) and additives (CaF_2, LiF, MgF_2). Model calculations were performed to estimate the effect of electronic conductivity on the current efficiency for aluminium deposition. The behaviour of so-called metal fog generated at the cathode during deposition of aluminium was also investigated.

8:55 AM
Effect of the Bubble Growth Mechanism on the Spectrum of Voltage Fluctuations in the Reduction Cell: *Laszlo Istvan Kiss*[1]; Sandor Poncsak[1]; [1]UQAC Université du Québec à Chicoutimi, Dépt. des Sciences Appliquées, 555 Blvd. de l'Université', Chicoutimi, Quebec G7H 2B1 Canada

The spectrum of the voltage oscillations in the aluminum reduction cell depends on the size of the anode. Small laboratory cells and industrial size cells exhibit different fluctuation patterns. The growth of bubbles can be divided into two periods controlled by different physical mechanisms. The detachment frequency of the bubbles from the nucleation sites depends on the current density and detachment size, the latter being influenced by the material, microstructure and geometry of the anode. After detachment the main factor causing growth of the traveling bubbles is coalescence. A mathematical model that keeps track of each and every individual gas bubbles generated under the anode was developed and used to analyze the character of the fluctuations of the cell voltage. It is shown that in the case of industrial size anodes the voltage oscillations are dominated by the dynamics of the bubble interactions (coalescence) in the two-phase layer, while in small size laboratory systems the frequency of nucleation of the individual bubbles can be observed.

9:20 AM
Crystallization of Cryolite and Alumina at the Metal-Bath Interface in Aluminium Reduction Cells: *Asbjorn Solheim*[1]; [1]SINTEF, Matls. Tech., NO-7465 Trondheim, Norway

It is a well-known fact that the electrolyte (bath) facing the cathode in aluminium electrolysis cells contains less aluminium fluoride than the bulk, due to the diffusion processes taking place in the boundary layer close to the cathode. A lower AlF3 concentration gives a higher liquidus temperature. Under certain conditions, the local liquidus temperature at the cathode may become equal to the bath temperature, which provokes crystallization of solid cryolite and/or alumina. The present paper presents the relevant theory for such crystallization, as well as some calculations based on a numerical solution of the Stefan-Maxwell equation. The results show that formation of solid cryolite at the cathode may take place during normal cell operation. The rate increases with increasing AlF3 content in the bath, increasing cathodic current density, decreasing superheat, and decreasing mass transfer coefficient for AlF3. Some possible consequences of the precipitation are discussed.

9:45 AM
Electric Conductivity of Cryolite Electrolytes during the Liquid-Solid Phase Transformation: *Stanislaw Pietrzyk*[1]; Ryszard Oblakowski[1]; [1]University of Mining and Metallurgy, Dept. of Metall. of Non-Ferrous Metals, Fac. of Non-Ferrous Metals, Al. Mickiewicza 30, 30-059 Krakow, Poland

Is generally accepted that flow of current during aluminium electrolysis may take place through the layer of side ledge (frozen electrolyte) to the carbon side lining of cathode. It may lead to current losses and this way contribute to decrease of current efficiency. Interesting from this of point of view is recognition of changes of electrical conductivity of the sodium cryolite melts during the liquid-solid phase transition and in solid state. In this work electrical conductivity measurements were performed on natural cryolite and its binary solutions with AlF3, CaF2, MgF2, LiF and also on industrial electrolytes. In solidified mixtures of cryolite with additions of AlF3, CaF2, MgF2, the conductivity is higher than in pure cryolite in the same temperature range. This observation suggests that in solid state additives of AlF3, CaF2, MgF2 cause changes in structure of solid cryolite by introduces cation vacancies which facilitate the ion transport.

10:10 AM Break

10:20 AM
Electrochemical Studies of Aluminum Deposition in Ionic Liquids at Ambient Temperatures: *Venkat Kamavaram*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. of Metlgcl. & Matls. Eng. & Ctr. for Green Mfg., Tuscaloosa, AL 35487 USA

Ionic liquids are low melting salts used as potential non-aqueous electrolytes for the electrodeposition of aluminum. In the present study, an ionic liquid based on AlCl3-1-methyl-3-butylimidazolium chloride was used as an electrolyte for the electrodeposition of aluminum at ambient temperatures. Electrochemical studies were carried out using cyclic voltammetric (CV) and chronoampereometric (CA)techniques. Electrodeposition of aluminum was carried out in the above mentioned ionic liquid on copper cathode. The process of electrodeposition was found to be quasi-reversible and the kinetic mechanism was identified as instantaneous nucleation followed by diffusion controlled three-dimensional growth. Kinetic parameters such as charge transfer coefficient (α) and diffusion coefficient(D0) of the species Al2-Cl7- were obtained from the CV and CA data, which compared well with those available in the literature for aluminum deposition.

10:45 AM
The Anode Effect Revisited: *Jim B. Metson*[1]; R. G. Haverkamp[2]; M. M. Hyland[3]; Jingxi Chen[1]; [1]University of Auckland, Dept. of Chem., PB 92019, Auckland, New Zealand; [2]Massey University, Inst. of Tech. & Eng., PB 11 222, Palmerston N., New Zealand; [3]University of Auckland, Dept. of Cheml. & Matls. Eng., New Zealand

Despite an extensive literature and long history of plant experience, there are still puzzling aspects of the anode effect as observed in aluminium reduction cells. The nature of the onset of the anode effect and its variable persistence, are still the subject of debate. In this study, contact angles at carbon/electrolyte interface have been measured, as a function of electrolyte composition. In line with previous studies, CaF2 is observed to effect anode wettability, and contact angle increases with alumina depletion. Voltage/time behaviour of the cell is thus interpreted in terms of chemical changes affecting the interfacial surface tension, particularly those arising from the influence of additives in the bath and anode. Modification of the anode surface, either by doping or by reaction with the electrolyte, and the non-wettability of the modified surface by certain bath compositions, explains why these effects can be difficult to quench, even with elevated alumina concentrations.

11:10 AM
A Predictive Thermodynamic Model for the Al-NaF-AlF3-CaF2-Al2O3 System: *Patrice Chartrand*[1]; Arthur D. Pelton[1]; [1]Ecole Polytechnique, CRCT, CP 6079, Sta. "downtown", Montreal, Quebec H3C 3A7 Canada

A large body of experimental thermodynamic and phase-equilibrium data exists for the Al-Na-Ca-F-O system. All available data are critically evaluated to obtain optimized parameters of thermodynamic models of all phases. The bath model is the Quasichemical model in the quadruplet approximation that evaluates 1st- and 2nd-nearest-neighbor short-range-order. Other solution models are used for non-stoichiometric solid cryolite, the gas phase and molten Al. The models are then used to predict the thermodynamic properties and metal-bath-gas equilibria in the multicomponent heterogeneous system. All experimental data were reproduced within experimental error limits, and in most cases ternary and multicomponent data were predicted by

the model, not fitted. The model parameters form a database that is part of the FactSage Thermochemical Software. This software can be used to predict/simulate metal-bath-gas equilibrium (phase amounts, compositions, activities/partial pressures, enthalpies, Tliquidus, etc.) for 25°C < T < 1100°C, for P < 4 atm, and for BR(wt.%) from 3.0 to AlF3 saturation.

Cast Shop Technology: Modeling I

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: David H. DeYoung, Alcoa Technical Center, Alcoa Center, PA 15069 USA; John F. Grandfield, CSIRO Australia, Preston, Victoria 3072 Australia; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Monday AM — Room: 6A
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Alvaro Giron, Alcoa Inc., Alcoa Techl. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA; Asbjorn Mo, SINTEF, Box 124 Blindern, N-0312 Oslo, Norway

8:30 AM
Applications of Optimization in Metal Casting and Heat Treatment: *Ravi Vijayaraghavan*[1]; Mei Li[1]; Xuming Su[1]; John E. Allison[1]; [1]Ford Motor Company, Ford Rsrch. Labs., MD 3182, Rm. 2122, 2101 Village Rd., Dearborn, MI 48124 USA

A major thrust of cast aluminum research at Ford is the development of computer-aided engineering methods to reduce cycle time and cost for producing optimized cast aluminum automotive components. The goal is to provide tools that simulate casting solidification and predict microstructure, mechanical properties and durability in a cast component. The vision is to create and test virtual aluminum castings, reducing the need for physical prototypes. Optimization has been identified as a critical tool to accomplish this. Many major foundries are using software such as ProCAST and MagmaSOFT, to simulate casting and heat treatment and to optimize process design based on the simulations. A number of input parameters to these casting simulation programs (such as material properties and boundary conditions) are not well known. This has motivated the need for an optimization tool, which uses an inverse modeling approach to determine these parameters given the experiment results. The talk presents applications of optimization methods in casting process development and heat treatment. Use of efficient and robust optimization based methodologies can play a major role in reducing costs and improving productivity and quality of cast products.

8:55 AM
Mathematical Modeling of Melt Flow in Die-Casting Shot Sleeve: Carroll E. Mobley[1]; Junmin Park[1]; *Yogeshwar Sahai*[1]; [1]The Ohio State University, MSE Dept., 2041 College Rd., Columbus, OH 43210 USA

In a typical cold chamber die-casting operation, molten aluminum alloy is poured into a shot sleeve and a plunger rapidly pushes the metal into the die cavity. During this process, air can be trapped in the liquid alloy, and some liquid alloy can solidify in the shot sleeve. Entrapped air and Externally Solidified Product (ESP) degrade the quality of the die-cast products. To reduce the amount of trapped air and ESP, metal flow was mathematically simulated using a commercial code, FLOW3D. The effects of plunger velocity and acceleration, density, dynamic viscosity, kinematic viscosity, and surface tension of molten alloy were studied. The simulated flow patterns were compared with the experimentally obtained flow patterns in a water model. The plunger velocity and acceleration were found to have strong influence on the wave pattern and hence on the amount of trapped air. The effect of other parameters that are stated above on the wave pattern was relatively small. As plunger velocity increases, the amount of trapped air first decreases, followed by an increase. Thus, there is an optimum plunger velocity at which the amount of entrapped air is minimum. The tendency of rolling-over of wave created by metal flow in shot sleeve increases as acceleration increases. The simulations in this study agree well with the available experimental data.

9:20 AM
Determination of Boundary Conditions for Solidification Simulation in an Aluminum Permanent Mold Test Casting: *Joy Adair Hines*[1]; Ravi Vijayaraghavan[1]; Emmanuel K. Glakpe[2]; [1]Ford Motor Company, Ford Rsrch. Lab., MD 3182/ SRL, PO Box 2053, Dearborn, MI 48121-2053 USA; [2]Howard University, Dept. of Mechl. Eng., 2300 Sixth St., N.W., Washington, DC 20059 USA

Computer simulation has played an increasingly important role in the manufacture of shape castings in recent years. One of the major challenges to accurate casting simulation has been the determination of accurate boundary conditions for a particular casting. In this work, a specially designed test casting was cast using a low pressure process under a variety of conditions. Cooling curves were obtained from regions in both the casting and the mold. Those results were then compared with cooling curves from the same locations in a finite element mesh of the entire cast system. A Ford Motor Company proprietary software program was used to optimize the interfacial heat transfer coefficients between the metal and the mold as well as to verify the thermal properties within the mold itself. These results were then used to predict the solidification behavior of a cylinder head casting.

9:45 AM Break

10:00 AM
Modeling of Twin-Roll Thin Strip Casting of Aluminum Alloys: *Yogeshwar Sahai*[1]; Amit Saxena[1]; [1]The Ohio State University, MSE Dept., 2041 College Rd., Columbus, OH 43065 USA

In twin-roll thin strip casting processes, thermo-mechanical stresses in solidifying alloy may result in formation of defects. The first part of this modeling study consisted of predicting melt flow, temperature profiles, and resulting solidification during the thin strip casting process. The predicted temperature gradients in the solidifying alloy and externally applied mechanical force values, such as roll force and strip exit tension, were used to predict the thermo-mechanical stresses caused in the cast strip. A visco-plastic constitutive relation was used to describe the behavior of solidifying aluminum alloy. The effects of inlet melt feed rate and contact strip/roll heat transfer coefficient on the resulting stress profile in the cast strip and rolls were investigated. The implications of these results in industrial practice are discussed.

10:25 AM
Mathematical Modelling of Wire Rod Casting: *Dag Lindholm*[1]; Einar Arne Sørheim[1]; John Rødseth[2]; Dag Mortensen[1]; Harald Heggland[2]; [1]Institute for Energy Technology, PO Box 40, 2027 Kjeller, Norway; [2]Hydro Aluminium, RD Matls. Tech., 4265 Håvik, Norway

Wire rod casting is a complex process where molten aluminium solidifies within a rotating wheel mould. Bar shrinkage and air gap formation influence on the cooling conditions and thus the quality of the bar leaving the wheel. This work focuses on a mathematical model that is tailor-made for continuous rod casting. The two-dimensional transient model is based on a finite volume technique, and it computes the development of temperature field in the bar, mould and steel belt during the casting, as well as in the bar alone when it leaves the rotating wheel. The work is supplemented with presentation of full-scale measurements of temperatures and a technique for inverse modelling to find both internal and external heat transfer coefficients.

10:50 AM
Multiphase Modeling of Gas Purging during Continuous Metal Casting: *Anirudh A. Mukhopadhyay*[1]; Andrey A. Troshko[1]; [1]Fluent, Inc., 10 Cavendish Ct., Centerra Resource Park, Lebanon, NH 03766-1442 USA

Flow field in the continuous casting molds is critically important for quality and consistency. Two-phase flow of liquid metal and gas bubbles has been simulated with two-fluid model. Ability of the two-fluid model to accurately simulate such flows strongly depends on the correct closure of interfacial forces governing momentum transfer across the interface. The interfacial forces include: drag force: drag law is available for viscous, distorted and spherical cap regimes; lift force: modified lift coefficient takes into account inviscid lift and aerodynamic lift due to the vortexes shed by the bubble wake; turbulence dispersion force: this force accounts for the bubble dispersion caused by turbulent eddies in the mold; Another important aspect is non-uniformity of bubble size. Bubble size is controlled by liquid turbulence and several sizes are investigated. This study focuses on bubble induced flow modification in the mold.

11:15 AM
A Cooperative Process for Conducting R&D in the Aluminium Casthouse Products Sector: *John A. Taylor*[1]; David H. StJohn[1]; [1]CRC for Cast Metals Manufacturing (CAST), Dept. Mining, Minls. & Matls. Eng., The University of Queensland, Brisbane, QLD 4072 Australia

CAST conducts research, commercialisation and education activities across four sectors within the light metals industry. The Aluminium

Casthouse Products sector spans activities ranging from improving production processes, to expanding product range and quality, to providing technical support for downstream users. Strategic and industry-focussed projects with medium to long term goals are conducted with Centre partners and are supplemented with shorter-term projects with fee-for-service customers. The strength of the Cooperative Research Centre approach for providing innovative solutions lies in the networking of appropriately-skilled teams and the creation of value chains across diverse organisations. Flexibility and creativity in funding, IP and commercialisation arrangements exist. Cross-fertilisation of ideas from adjacent sectors enhance the solution-generation process. This paper explores the CRC process as a model for conducting industrial R&D and uses examples to elucidate the basic principles. Comparisons are made with more traditional R&D approaches.

Computational Modeling of Materials, Minerals & Metals Processing: Monday AM Opening Plenary Session 9:00 AM – 10:30 AM

Sponsored by: Materials Processing & Manufacturing Division
Program Organizer: Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Monday AM — Room: 619-620
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Mark Cross, The University of Greenwich, Sch. of Compg. & Mathl. Scis., Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK; James W. Evans, University of California, Dept. of Matls. Sci. & Minl. Eng., 585 Evans Hall, Berkeley, CA 94720 USA

Keynote
Multi-Physics Simulation of Metal Processing: *Mark Samonds*[1]; [1]UES Software, Inc., 175 Admiral Cochran Dr., Annapolis, MD 21401 USA

Manufacturing processes which involve liquid to solid and solid state transformations in metals encompass a wide variety of physical phenomena. The past twenty years have seen a steady evolution in the complexity and scope of numerical models of these processes, beginning with thermal analyses without phase change. Recently, a few software packages have become available which have true multi-physics capabilities. This permits the treatment of thermal, fluids, stress, electromagnetic, chemical reaction and microstructure development aspects altogether in a fully coupled simulation. This paper will consider various aspects of these types of models.

Keynote
Computing the Dynamic Interaction of Magnetic Fields and Turbulent Conducting Fluids in Metals Processing: *Koulis Pericleous*[1]; [1]University of Greenwich, Greenwich Maritime Campus, Queen Mary Ct., Rm. 361, Greenwich, London SE10 9LS UK

Magnetic fields have many actual applications in the metals processing industry. Externally applied magnetic fields give rise to electromagnetic (Lorentz) forces formed by the cross product JxB, between the induced current density J and the magnetic field density B. When the metal is in liquid form, the Lorentz force generates motion in the fluid which in applications of practical interest becomes turbulent. In modelling terms, the Lorentz force appears as a source in the momentum equations. In addition, the induced current generates heat (Joule heating) in the metal that is in proportion to J2, with a corresponding source of heat in the energy equation. Whether as heat or as a force, these effects represent action at a distance–a most useful attribute when dealing with hot metal. The Lorentz force is used to stir solidifying alloys, pump liquid metal in conduits, dampen the flow in the meniscus of a continuous caster, levitate metal drops, induce artificial gravity conditions in suspensions or contain liquid metal. Elsewhere, the Lorentz force may be a by-product of some other operation, so leading to wave excitation in aluminium electrolysis cells, or altering the shape of the weld pool in arc welding. Joule heating is most commonly used with applied AC fields, to melt metal in induction furnaces. The author and his colleagues have been involved in the modelling of most of these processes in the past decade. Modelling is not however straightforward, since most of the examples mentioned represent genuine multi-physics challenges. There is a strong coupling between the flow field and electromagnetic field. The addition of a dynamically varying metal free surface and the moving solidus front means the flow, heat and electromagnetic fields need to be computed simultaneously. In situations involving metal containment, the metal free surface position is governed by the interplay of gravity, Lorentz force, surface tension and fluid inertia. Since all the interesting effects often happen in thin boundary layers at the surface due to the skin effect, mesh generation and mesh control during the computation become non trivial problems that need to be addressed. This paper presents a review of numerical methods used to model droplet levitation, semi-levitation melting and cold crucible induction melting of metals. The first method is based on spectral collocation techniques and the second is the traditional FV approach. Steps taken to validate the computations and typical transient results are also given.

10:30 AM Break

Computational Modeling of Materials, Minerals & Metals Processing: Track A - CFD Modeling - I

Sponsored by: Materials Processing & Manufacturing Division
Program Organizer: Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Monday AM — Room: 619
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Mark Samonds, UES Software Inc., 175 Admiral Cochran Dr., Annapolis, MD 21401 USA; Mayur Patel, University of Greenwich, Ctr. of Numcl. Modlg. & Proc. Analy., London SE109LS UK

10:45 AM
Simulation of Turbulent Flow and Particle Transport in the Continuous Casting of Steel: Q. Yuan[1]; T. Shi[1]; S. P. Vanka[1]; *B. G. Thomas*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mechl. & Indl. Eng., 1206 W. Green St., Urbana, IL 61801 USA

The quality of continuous cast steel is greatly affected by fluid flow in the mold region, especially involving transient phenomena. Mathematical models are being applied to investigate many different aspects of these phenomena, but their accuracy must be validated before they can be applied with confidence. As part of a long-term effort to develop and apply comprehensive models of the continuous casting process, this work evaluates the relative accuracy of models of three different fluid flow phenomena in continuous casting through comparison with measurements. Firstly, transient flow simulations of velocities in the mold region are compared with digital particle image velocimetry (PIV) measurements in a single phase water model. Large-eddy simulations (LES) are found to reasonably match the flow measurements, including transient flow variations, except at long time scales, which could not be modeled owing to the excessive computation costs. The standard K-ε model produced very good agreement with time-averaged velocities for relatively little computation time, although it is inaccurate at predicting the transient variations. Secondly, particle trajectory calculations are compared with water model measurements to study the distribution and flotation removal of inclusion particles. The LES model was able to match the measurements both qualitatively and quantitatively. Thirdly, steady, multiphase flow computations are compared with flow patterns observed in both a water model and an operating steel caster with argon gas injection. For the same conditions, the water model and steel caster produced very different flow behavior. The computational model was able to match the measured flow patterns in both cases. This work suggests that computational flow modeling has the potential to match real processes as well or better than water models, especially when complex related phenomena such as particle motion and multiphase flow are involved. Much work is still needed to further improve the models and to apply them in parametric studies.

11:10 AM
CFD Modeling of the Hydrodynamics of Fluidization in the Sand Surrounding a Lost Foam Casting Pattern: *Nathanael Harrison Hudson*[1]; *Sushil Bhavnani*[1]; Ruel A. Overfelt[1]; [1]Auburn University, Dept. of Mechl. Eng., 213 Ross Hall, Auburn University, AL 36849 USA

In the aerospace and automotive industries, shapes arise which require casting. The problem with these cast parts is the empiricism and expense in developing an efficient casting process. There has been an

interest in using the fluidized bed to allow sand to better encapsulate the complicated surface geometry of a lost foam pre-casting mold. Fluidization helps to eliminate the voids in the sand and improves the integrity of the casting. At issue is the hydrodynamics of the sand and air around the pre-casting mold. The software PHOENICS, employing a two-fluid approach, is used to simulate the flow of sand and air as interpenetrating continua. The kinetic theory of granular flow for the sand phase is incorporated into the re-compilable PHOENICS code. The results of this study consist of voidage patterns and the velocity components of the respective phases around the pre-casting submerged in a two-dimensional fluidized bed. The model is benchmarked against experimental voidage patterns without a foam pre-casting and to some trials with a pre-casting. The final test consists of a series of computer runs with an obstacle submerged in the bed at various aspect ratios of length to width.

11:35 AM

Continuum Modelling of Granular Flows using PHYSICA, a 3-D Unstructured, Finite-Volume Modelling Framework: *Nicholas Christakis*[1]; Mayur K. Patel[1]; Mark Cross[1]; John Baxter[2]; Hadi Abou-Chakra[2]; Ugua Tünün[2]; [1]University of Greenwich, Ctr. of Numcl. Modlg. & Proc. Analy., Sch. of Compg. & Mathl. Scis., 30 Park Row, London SE10 9LS UK; [2]University of Surrey, Dept. of Cheml. & Proc. Eng., Guildford, GU2 5XH Surrey, UK

In recent years significant effort has been put in using Continuum Mechanics for the description of granular flows. Although these models are partially successful in capturing some flow characteristics, they lack essential information on material properties, which are needed to account for the interactions between different particles. Thus, they are incomplete and can not be used to describe processes such as hopper filling/emptying and pneumatic conveying, where particle-particle interactions can lead to phenomena such as segregation, degradation and agglomeration. In this paper, a 3-D unstructured Finite-Volume framework is presented, which employs interface tracking techniques (VOF/SEA algorithms) to determine the material-air interface. Separate routines are employed to perform the tracking of the individual material components of the granular mixture in the bulk. Various transport processes, arising from the micro-mechanical properties of the different particle species in the granular mixture, can be obtained through kinetic theory. The transport coefficient(s) of each of the individual species can be determined and analysed in a micro-mechanical framework and the transport process parametrised in the form of constitutive model. These models provide the continuum theory with information on the micro-mechanics. This work describes in detail the numerical schemes employed in the Continuum Mechanics framework and the micro-mechanical parametrisations that are implemented in the transport equations. Model predictions for different flow conditions and comparisons with experimental data are presented and conclusions are drawn on the model capability to realistically predict and quantify the main characteristics of granular flows.

12:00 PM

Mathematical Model of Inclusion Removal during Steel Degassing: Michel Cournil[1]; Frédéric Gruy[1]; *Pascal Gardin*[2]; Hubert Saint-Raymond[3]; [1]Ecole des Mines de Saint-Etienne, SPIN Div., 158 Cours Fauriel, Saint-Etienne, Cedex 2 42023 France; [2]IRSID, Themef, Voie Romaine, BP 30320, Maizières-lès-Metz, Cedex 57283 France; [3]IRSID, PCMO, Voie Romaine, BP 30320, Maizières-lès-Metz, Cedex 57283 France

The control of inclusion elimination is getting more and more important to obtain clean steel. But having a predictive tool is still a challenge, because the number of phases is important in steelmaking industry: liquid steel, slag layer, bubbles and inclusions (with a large range of composition and rheology). The paper presents the methodology which is developed at IRSID to predict oxygen content evolution during RH degassing.The main mechanisms which have to be considered are:-inclusion growth by turbulent aggregation of elementary inclusions (keeping in mind that liquid steel is a non-wetting medium for inclusions); the difficulty is to express collision efficiency for alumina particles, -inclusion removal by flotation; the difficulty for alumina clusters stems from the complex morphology of particle; fortunately, the use of fractal concept makes it possible to cope with this problem.The proposed paper describes the general modelling of inclusion removal taking into account the previous mechanisms. Hydrodynamic parameters are obtained by means of Fluent CFD package and a specific coding is developed for cluster growth. Influence of different parameters (fractal dimension, argon flow rate) on time-dependant inclusion size distribution is given.

12:25 PM

Application of Coupled Continuum-Mesoscopic Computational Methods for the Simulation of Complex Fluids in Industrial Processes: Greg Glinski[1]; *Chris Bailey*[1]; Koulis Pericleous[1]; [1]University of Greenwich, 30 Park Row, Greenwich SE10 9LS UK

Recent advances in new computational modelling techniques such as Lattice-Boltzmann and Dissipative Particle methods offer the prospect of simulating complex fluids such as colloidal and dense suspensions in industrial processes. These methods provide a means to overcome challenges that arise in modelling such fluids due to the disparate temporal scales present. These methods are termed mesoscopic methods as they lie between computational intense molecular dynamics and traditional macroscopic CFD methods. This paper will discuss the use of the methods to simulate the movement and subsequent processing of solder paste material in electronic component manufacture. Results will be presented that show how these methods are coupled within a macroscopic CFD code to provide detailed predictions of solder paste deposition onto a printed circuit board. Comparisons between the model results and experimental data will also be presented.

Computational Modeling of Materials, Minerals & Metals Processing: Track B - Heat & Mass Transfer - I

Sponsored by: Materials Processing & Manufacturing Division
Program Organizer: Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Monday AM — Room: 620
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Koulis Pericleous, University of Greenwich, Greenwich Maritime Campus, Greenwich, London SE10 9 LS UK; Stavros A. Argyropoulos, University of Toronto, Dept. of Metall. & Matls. Sci., Toronto, Ontario M5S 3E4 Canada

10:45 AM

Heat Load Control of Blast Furnace Wall using Statistical Optimization Techniques: *Tae-hwa Choi*[1]; Yong-hwan Chu[2]; Chonghun Han[2]; [1]Pohang Iron and Steel Company(POSCO), Techl. Rsrch. Labs./Iron & Steel Making Rsrch. Grp., 1 Goedong-dong, Namgu, Pohang-shi, Gyungbuk 790-785 Korea; [2]Pohang University of Science and Technology, Dept. of Chem. Eng., 31 san Houja-dong, Namgu, Pohang-shi, Gyungbuk 790-300 Korea

In the blast furnace, various complex phenomena take place including mass transfers, heat transfers, a lot of reactions and phase equilibriums, but systems of these phenomena are not found out clearly even until now. Consequently, it is very difficult to make fundamental models on these systems, which results in the operation based on the heuristics of industrial operators by changing operating condition little by little due to ignorance of the optimum point. However, since this method is not systematic, we propose statistical optimization technique based on analysis of historical data and empirical model building. In this approach, we first find the variables which must be optimized and the manipulated variables to adjust those response variables. Next, we collect the necessary data for these variables after preprocessing such as removal of noise and outlier. Finally, we construct empirical models describing the patterns of response variables in terms of manipulated variables by PLS regression method, and then these models are used as objective functions for entire optimization problem. By using appropriate optimization algorithm, this multi-objective optimization problem is solved, which gives us the compromising optimum operating condition considering all response variables from the past operating conditions.

11:10 AM

Mathmatical Modeling and Experimental Verification of Assimilation of Exothermic Additions in Liquid Metals: *Stavros A. Argyropoulos*[1]; Henry H. Hu[2]; [1]University of Toronto, Dept. of Metall. & Matls. Sci., 184 College St., Toronto, Ontario M5S 3E4 Canada; [2]University of Windsor, Dept. of Mech., Automot. & Matls. Eng., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada

The assimilation of exothermic additions in liquid metals exhibit an array of unique coupled heat, mass and momentum transport phenomena. The phenomena are further complicated with the presence of a moving boundary. In this paper a mathematical model will be described which solves these coupled complex phenomena. The SIMPLER algorithm was employed to solve numerically the pertinent partial differ-

ential equations. The computational results indicated that the exothermic heat of mixing leads to a rapid increase of temperature around the moving boundary, which produced an enhanced convective flow in the liquid phase. The intensification of fluid flow around the moving boundary resulted in an acceleration of the melting process. An extensive verification of the mathematical model was carried out and will be described in the paper. First, in a low temperature physical model consisting of ice immersion in different sulfuric acid solutions. In this physical model, both temperature and velocity measurements were carried out. The model results were compared with experimental measurements and they were found to be in good agreement. Second, in a high temperature work involving assimilation of silicon in high carbon liquid iron. The model was also applied to predict fluid flow, heat and mass transfer for this high temperature experiments and a reasonable agreement was obtained. In addition new dimensionless heat transfer correlations that quantify these complex phenomena will be presented.

11:35 AM
A Model of the Cathode Dynamics in Electric Field-Enhanced Smelting and Refining of Steel: *David Michael Dussault*[1]; Adam Powell[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139 USA

A mathematical model of coupled diffusion, electrochemical reactions and fluid dynamics at the cathode in electric field-enhanced smelting and refining of steel is developed using the Navier-Stokes equations and the phase field method. Experimental evidence indicates that the reaction rate is limited by ferrous ion transport to the cathode, resulting in a Mullins-Sekerka instability at the slag-metal interface and the growth of liquid iron fingers into the liquid slag. The differential equations are discretized using finite differencing with a uniform mesh, and the resulting system of nonlinear equations is solved using a multidimensional Newton-Krylov method. Presented are the formulation and two dimensional results, and issues expected to arise on extension to three dimensions are discussed.

12:00 PM
A Mathematical Model for the Control of Metallurgical Properties of the Product Sinter: *Ndabezinhle Manengi Dube*[1]; E. F. Vegman[2]; [1]University of Zimbabwe, Fac. of Eng., Dept. of Metall. Eng., PO Box MP167, Mount Pleasant, Harare Zimbabwe; [2]Institute of Steel and Alloys, Moscow Russia

In the conventional practice of sintering, the quality of the product sinter down the cross-section of the bed is quite variable,with the top layer consisting of weak sinter and the bottom layer, strong over-fused sinter of poor reducibility. This points to a variable distribution of the heat balance in the sintering process down the sinter bed. This causes losses in the productivity of the sinter machine and the blast furnace. The experienced phenomenon caused by varying input of regenerated heat requires a zonal approach to the study of the sintering process with the aim of producing a uniform quality of the sinter cake. The current work includes a mathematical model that would make possible optimization of multiple-layer sintering and segregation technologies. It is based on a zonal heat balance. This calculation method presents the following prospects: 1. The minimum theoretically possible coke fines consumption during the sintering of any type of iron ore fines can be established. The optimum difference between the coke fines rates in the top and bottom layers during multiple-layer sintering should be easily determined with the aid of this model. 2. On the basis of this model an automatic control system for the coke rates in the top and bottom layers during two-layer sintering is possible. 3. The model can be used to control the quality (chemical, physical and metallurgical) of the sinter in any point down the sinter bed since the data input includes basicity, combustion zone temperature, and FeO content in the product sinter.

12:25 PM
Modelling the Magnetostriction of Textured Ferromagnetic Materials with a Cubic Structure: *Ruben Decocker*[1]; Leo Kestens[1]; Yvan Houbaert[1]; [1]University Ghent, Metall. & Matl. Sci., Technologiepark 9, Ghent, East-Flanders 9052 Belgium

A magneto-elastic model is presented to calculate the orientation dependence of the magnetostrictive strain, observed at saturated magnetization in ferromagnetic materials with a cubic crystal structure and an arbitrary crystallographic texture. The formula of Becker and Döring is used to express the anisotropy of magnetostriction for a single crystal. In order to simulate the macroscopic average magnetostriction of a polycrystalline aggregate (with an arbitrary texture) the Reuss assumption of the elasticity theory was applied. According to this assumption the various orientations of the polycrystal can deform without constraints, producing local strain incompatibilities in the microstructure but observing a total stress equilibrium. The macroscopic strain is calculated as the weighted average of the individual strains of all orientations composing the polycrystal. The weight factors are determined by the volume fractions of the corresponding orientations of the given texture, which can be measured by standard X-ray diffraction techniques. The model is applied to simulate the variation of magnetostriction (at saturation) with respect to the rolling direction for a standard grade of non-oriented electrical steel. A brief comparison with experimental data allows validating the basic model assumptions.

Computational Phase Transformations: Stress-Dominated Processes

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Jt. Computational Materials Science & Engineering, Thermodynamics & Phase Equilibria Committee
Program Organizers: Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; Mark Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; James Aaron Warren, NIST, CTCMS and Metallurgy Division, Gaithersburg, MD 20899-8554 USA

Monday AM — Room: 201
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Long-Qing Chen, Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16802 USA

8:30 AM Opening Remarks

8:35 AM
3D Phase Field Microelasticity in 3D Mesoscopic Modeling of Complex Structures: Yongmei M. Jin[1]; Yu U. Wang[1]; *Armen G. Khachaturyan*[1]; [1]Rutgers University, Dept. of Cer. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854-8065 USA

Recent developments in the Phase Field Microelasticity theory and its applications to phase transformations and plastic deformation (dislocation dynamics) of structurally inhomogeneous single and polycrystals as well as the development of the computer simulation techniques based on this theory open the way to a realistic 3D simulation of technologically important materials. The strength of the Phase Field Microelasticity is its ability to describe the long-ranged strain-induced interaction between structural inhomogeneities of arbitrary topological complexity in the spontaneous self-organization processes without any a priory assumption. The incorporation of dislocations into the phase transformation theory and taking into account the grain structure allow one to attack the entire spectrum of important problems of the computer-aided materials design. Examples of 3D simulations of a real martensitic transformation in Au-Cd polycrystalline shape memory alloy and a simulation of the evolution of multi-dislocation systems in single and polycrystals during plastic deformation are presented.

9:20 AM
Phase Field Modeling of Fracture and Deformation of Non-Crystalline Materials: *Mo Li*[1]; [1]Johns Hopkins University, Dept. of Matls. Sci. & Eng., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA

Deformation and fracture are non-equilibrium dynamic problem. Continuum and, most recently, atomistic dynamic modeling are used for fracture in crystalline and other homogeneous materials. These methods, however, are not quite suitable for fracture and deformation in non-crystalline materials. The primary reason is that there is no clear understanding of the constitutive behaviors as well as detailed microscopic mechanisms in these materials. In addition, the minimum dimension of the fracture or deformation regions in non-crystalline materials is of the order of submicrons, which is too large for atomistic modeling and too small for continuum modeling. To overcome this difficulty, we developed a new approach using the phase field model. In this talk, I will describe the formulation of this new method for deformation and fracture in non-crystalline materials; I will also give examples from the modeling using this method in crack initiation, propagation, and branching or shear localization in amorphous metals.

9:50 AM
Computer Simulation of Interactions of Migrating Grain Boundaries and Dislocations with Solutes and Precipitates: *Yunzhi Wang*[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

The interactions between migrating defects such as grain boundaries and dislocations with impurities and precipitates determine many important properties of materials. In this presentation, we discuss recent advances in phase field modeling of solute segregation and phase transition at migrating grain boundaries and dislocations and migration of dislocations through g channels in single crystal cuboidal gamma/gamma microstructures and gamma/gamma multilayers of superalloys. In particular, the transition from low solute segregation to high solute segregation will be characterized as function of temperature, solute concentration and defect migration velocity. The motion of confined dislocation propagation within g channels and layers will be characterized as function of channel width and layer thickness and misfit dislocation content. The simulation results will be discussed against the ones obtained from other methods that update defect and microstructural states according to a path of steepest decent in energy or according to a Monte Carlo process.

10:20 AM Break

10:40 AM
Study of Strain-Mediated Transitions via Discrete Atom Method: *Jong K. Lee*[1]; [1]Michigan Technological University, Matls. Sci. & Eng., 1400 Townsend Dr., Houghton, MI 49931 USA

Materials processing presents various strain-mediated phenomena. For example, heteroepitaxially-grown thinfilms split into islands, or certain gamma prime precipitates during the processing of nickel-based superalloys split into smaller particulates. Such elastic instabilities occurring in complex boundary conditions were studied through the discrete atom method (DAM), which is predicated upon both statistical mechanics and linear elasticity. Precipitate splitting was found to depend on the commensurateness in elastic anisotropy between a precipitate and its matrix phase. A driving force for ordering was long known to be strain relaxation due to difference in atomic size. Such an ordering in a substitutional alloy was also examined, and the transition was found to be, depending on the degree of mismatch, of either a second-order or a first-order kinetics. The DAM work was recently extended to simulate the Jahn-Teller distortion which is partially responsible for the colossal magnetoresistance, i.e., a large decrease in resistance upon application of a magnetic field for certain perovskite-type manganese oxides such as La0.7Ca0.3MnO3. The distortion due to the spin-orbit coupling of Mn ion was approximated to be harmonic and its related ordering behavior such as ferromagnetic phase transformations was studied.

11:10 AM
Ostwald Ripening in Elastically Stressed Solids: K. Thornton[1]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Phase separation in crystalline solids is usually accompanied by long-ranged elastic stress. We have investigated the dynamics of Ostwald ripening in elastically stressed crystalline solids through large-scale numerical simulations. Using the insight provided by the calculations, a theory for the kinetics of ripening in elastically anisotropic homogeneous solids is developed. Both the theory and simulations show that elastic stress does not alter the exponent of the temporal power law for the average particle size, but does affect the amplitude of the power law in a manner that is only a function of the symmetry of the particle morphology. At sufficiently large misfit or volume fraction of precipitate interparticle coalescence may play a significant role in the coarsening process. The results of calculations that follow this coalescence process using a hybrid boundary-integral level-set method will be presented. Work supported by the National Science Foundation.

11:40 AM
A Mesoscopic Electromechanical Theory of Polycrystalline Ferroelectrics: *Jiangyu Li*[1]; [1]University of Nebraska, Dept. of Eng. Mech., Lincoln, NE 68588 USA

We present an effective mesoscopic theory on the electromechanical behavior of polycrystalline ferroelectrics which takes into account both the effect of domains and grains, and gives remarkably good predictions on the polarizability, saturation polarization and strain, the effective texture, and the effective piezoelectric constants of ferroelectric polycrystals. The superior electromechanical property of PZT at morphotropic phase boundary is explained, and optimal texture of polycrystals for high-strain actuation is identified.

Creep Deformation: Fundamentals and Applications: Fundamentals Behavior - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing & Manufacturing Division, Jt. Mechanical Behavior of Materials, Powder Materials Committee
Program Organizers: Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA; Sai V. Raj, NASA Glenn Research Center at Lewis Fields, Cleveland, OH 44135 USA

Monday AM — Room: 214
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: K. L. Murty, North Carolina State University, Box 7909, Raleigh, NC 27695-7909 USA

8:30 AM Opening Remarks

8:35 AM Keynote
Elevated Temperature Crystalline Plasticity at Diminished Length Scales: *Amiya K. Mukherjee*[1]; [1]University of California, Cheml. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

Creep and superplasticity have been studied in metallic and ceramic nanocrystalline materials. The nanocrystalline structures have been produced using one of the following techniques: high pressure torsion, pyrolysis of polymer precursor, crystallization from bulk metallic glass and electrodeposition. The processed nanocrystalline microstructure consisted of neat matrix, dispersion strengthened alloy, nano-nano and micro-nano composite and nanocrystalline thin film multilayers. The ceramic nano-nano composites exhibited extremely low creep rates. The metallic nanomaterials demonstrated both high strain-rate as well as low-temperature superplasticity. They also exhibited very high flow stresses and strain-hardening rates that cannot be explained by either grain growth or dislocation storage. The thin film multilayers revealed interesting scale-dependent behavior at elevated temperatures. The results will be discussed in terms of creep and superplastic deformation mechanisms in the domain of significantly dimin ished length scales. This investigation is supported by grants from NSF (#DMR-9903321) and ONR (#N00014-00-1-0186).

9:05 AM Invited
From Power Laws to Constitutive Relations–Creep as Special Case of Plastic Deformation: *Wolfgang Blum*[1]; Philip Eisenlohr[1]; [1]University of Erlangen-Nuernberg, Inst. f. Werkstoffwissenschaften LS1, Martensstr. 5, 91058 Erlangen, Germany

Research in creep is usually focusing on the power law relation between minimum creep rate and stress at constant temperature. Regions with a particular constant value of the stress exponent n are generally interpreted in terms of a particular deformation mechanism. The aim of the present contribution is to interpret creep as a special case of plastic deformation, starting with overelastic loading of the material up to a certain stress σ and then continuing at constant σ with work hardening or softening towards the steady state. The state of development of constitutive relations for the evolution of the dislocation structure with particular emphasis on dynamic recovery of dislocations and the kinetics of dislocation motion is presented. It is shown that contemporary constitutive relations developed for describing work hardening are also suited to describe creep. The constitutive modeling is helpful in checking the validity of the traditional power law interpretations.

9:30 AM Invited
Transitional Creep Mechanisms in Zr-Alloys: Application to Dry Storage of Spent Nuclear Fuel: *K. L. Murty*[1]; Y. Zhou[1]; B. Devarajan[1]; [1]DMR, National Science Foundation, Metals Rsrch., Prog. Dir., 4201 Wilson Blvd., Arlington, VA 22230 USA

Transitions in creep mechanisms are investigated in thin walled tubing of Zircaloy (Zr alloyed with Sn and Fe) and Nb-added Zircaloy under biaxial loading using internal pressurization superimposed with axial load. The diametral strains are monitored in-situ using a Laser telemetric extensometer. Both the alloys followed an exponential stress variation of the creep-rate at high (>10-3E) stresses. At very low stresses, viscous creep (n=1) was noted identifiable with Coble creep corresponding to small grain sized materials. At intermediate stress levels, Zircaloy behaved like class-M alloy with the climb of edge dislocations as the rate-controlling mechanism whereas addition

of Nb seems to indicate class-A type with stress exponents of 3 and 5 at lower and higher stress levels respectively. These findings are consistent with the earlier creep and TEM studies. In addition, short-term burst rupture properties of Zircaloy and Nb-added Zircaloy are investigated at temperatures ranging from 638K to 843K by internal pressurization of closed-end tubing samples. The data enabled an evaluation of Larson-Miller parameter and the present experimental results on Zircaloy-4 were in excellent agreement with those reported in the literature. Negligible differences were noted in the rupture characteristics between the standard Zircaloy and Nb-added Zircaloy. These results have significance on the feasibility of surface storage of spent fuel where the creep deformation of the cladding could be a failure mode because of the residual heat and fission products following exposure to neutron irradiation in commercial nuclear plants. Although blind extrapolation of short-term creep results to low stresses encountered during dry storage could lead to nonconservative estimates of the creep-rates and creep-strains, our estimations indicate extremely small contributions of viscous creep to the total strain.

9:55 AM Invited
Creep Processes at Low Stress Levels: *Terence G. Langdon*[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matl. Sci., Los Angeles, CA 90089-1453 USA

Creep experiments on pure metals and metallic alloys often show evidence for the occurrence of different creep mechanisms at very low stresses. For example, the stress exponent typically shows a transition to ~1-2 at low stresses. Possible mechanisms of flow under these conditions are grain boundary sliding, diffusion creep and Harper-Dorn creep. This paper examines the characteristics and viability of these various creep mechanisms.

10:20 AM Break

10:30 AM Invited
Thermal Activation in Plasticity: *U. Fred Kocks*[1]; [1]PO Box 89, Placerville, CO 81430 USA

In a recent paper, Cahn and Nabarro[1] and, earlier, Nabarro[2] have raised some old issues in this area–without, however, providing the answers that have been generally accepted (or at least widely used) since the publication, in 1975, of "Thermodynamics and Kinetics of Slip" by Kocks, Argon and Ashby[3], which has been recently summarized by Kocks[4]. The present short contribution aims at emphasizing some salient points, using the assertions in[1] as an organizing principle. Particular points to be discussed are: the meaning of activation parameters (including the 'pre-exponential factor') and their tensor character; the role of the 'mechanical threshold'; the influence of multiple slip and 'multi-axial tests'; a distinction of the directional from the scalar relation between stress and strain-rate; the relevance of 'reverse jumps' and of the hyperbolic-sine relation. [1]Cahn JW, Nabarro FRN, Thermal Activation under Shear: Phil. Mag. A81 [5],4409-4426; [2]Nabarro FRN, Thermal Activation and Andr ade Creep: Phil. Mag. Letters 75, 227 (1996) [3]Kocks UF, Argon AS, and Ashby MF, Thermodynamics and Kinetics of Slip: Prog. Mater. Sci. 19, 1-288 (Pergamon 1975) [4]Kocks UF, Plasticity: Thermal Activation Approach: Encyclopedia of Materials Science and Technology (Elsevier 2001).

10:55 AM
Deformation Processes during Creep of Pure Aluminium: *B. Wilshire*[1]; C. J. Palmer[1]; [1]University of Wales Swansea, Dept. of Matls. Eng., Singleton Park, Swansea SA2 8PP UK

When power-law relationships are used to quantify the dependences of the secondary or steady-state creep rates on stress (σ), the gradients (n) of the resulting log σlog (strain rate) plots vary in different stress/temperature regimes. Thus, pure metals are commonly considered to show regimes with n ≅ 1 at low stresses and n ≅ 4 at higher stresses, with n increasing rapidly as the stress increases further into the 'power law breakdown' range. Traditionally, the stress dependences of the stress exponent (n) are rationalised on the assumption that different creep mechanisms become dominant over different stress ranges. Yet, while creep is known to occur by diffusion-controlled generation and movement of dislocations when n ≅ 4, no general agreement has been reached on the precise mechanisms involved. Moreover, an unresolved debate continues over whether creep in the n ≅ 1 regime takes place by dislocation processes or by diffusional creep process which do not require dislocation movement. Given the controversies still surrounding the mechanisms controlling the creep properties of pure metals after more than 50 years of study, it is necessary to consider whether the continued adoption of power-law approaches has any prospect of proving successful in the future. For this reason, the creep behaviour patterns observed for pure aluminium have been re-evaluated. This analysis suggests that the concept of 'steady-state' creep should be abandoned on the grounds that a steady-state creep rate is rarely achieved. Moreover, standard stress/creep rate plots are shown to be well represented by continuous curves, with the dislocation processes governing the rates of creep strain accumulation being essentially the same at all stress levels, irrespective of the n value exhibited.

11:20 AM
Activation Volume for Shear under Low Stresses: *F. R.N. Nabarro*[1]; [1]Materials Research Institute, Univ. of the Witwatersrand, PB 3, Johannesburg WITS 2050 S. Africa

A system shows a strain rate ε under a shear stress σ. The customary definition of the experimental activation volume $V'^{e}(\sigma) = kT(\delta 1n\varepsilon/\delta\sigma)$ at constant temperature is valid only when σ is of the order of the flow stress at low temperatures. For Newtonian flow at low stresses $V'^{e}(\sigma) = kT/\sigma$, independently of the nature of the system. A similar anomaly occurs for power-law creep. We show that, for a potential that is symmetrical with respect to forward and backward jumps, if the true activation volume V'(σ) tends to a constant value V'(O) as σ tends to zero, then V'(O) cannot be determined by strain-rate changes in the Newtonian regime.

11:40 AM
A Microstructurally Based Model of Steady-State and Constant Structure Creep: *Jeffery C. Gibeling*[1]; Martin Heilmaier[2]; [1]University of California, Dept. Cheml. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Plansee AG, Tech. Ctr., A-6600, Reutte, Tirol, Austria

Current models have proven unsuccessful in fully rationalizing the unusual creep characteristics of dispersion strengthened metals. The most notable characteristics are the high value of the activation energy as compared to particle-free materials and the specific quantity and temperature dependence of the particle-induced threshold stress. While existing models generally provide satisfactory descriptions of creep under steady-state conditions, second order effects such as the response to stress changes have not been investigated to discriminate between models. To overcome these shortcomings, we propose a microstructurally based model that rests on the idea that internal back stresses develop during creep due to dislocation/dislocation and dislocation/particle interactions. Thus, the effective stress rather than the applied creep stress is the key factor for dislocation motion. This approach enables a unified description of the steady-state creep behavior of copper with and without dispersoids with a single s et of microstructurally founded parameters. In addition, the temperature dependencies of the creep resistance (incorporating the activation energies for self-diffusion and core-diffusion) and of the threshold stress (via the shear modulus) are correctly reflected. Finally, the difference in constant structure creep after stress reductions is predicted: while in pure copper the constant structure creep rate is found to be lower than the eventual steady-state rate, the opposite behavior is observed in dispersion strengthened copper.

David L. Davidson Symposium on Fatigue: Fatigue Mechanisms

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Jt. Mechanical Behavior of Materials, High Temperature Alloys Committee
Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; Thomas Zogas, Carpenter Technology Corporation, Reading, PA 19612-4662 USA

Monday AM — Room: 208
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Kwai S. Chan, Southwest Research Institute, 6220 Calebra Rd., San Antonio, TX 78238 USA; Wole Soboyejo, Princeton University, Mechl. & Aeros. Eng., Princeton, NJ 08544 USA

8:30 AM Opening Remarks

8:35 AM Introduction

8:45 AM Keynote
Fatigue Crack Initiation Mechanisms and Fatigue Life in High-Cycle and in Ultrahigh-Cycle Fatigue: *Hael Mughrabi*[1]; [1]University Erlangen-Nuernberg, Institut fuer Werkstoffwissenschaften, Martensstr. 5, Erlangen D-91058 Germany

First, the different fatigue crack initiation mechanisms will be reviewed. Emphasis will be placed on cyclic strain localization phenomena, trans- and intergranular crack initiation mechanisms and crack initiation at second-phase particles and at near-surface pores and cavities. Then, attention will be turned to fatigue life diagrams both in the form of S-N curves and Manson-Coffin plots. Points of particular interest will be the consideration of fatigue life-controlling mechanisms in the ultrahigh-cycle fatigue (UHCF) or gigacycle range (fatigue lives in excess of ca. 10^8 cycles). Microstructural interpretations of so-called "two-stage" or "multi-stage" fatigue life diagrams exhibiting a second lower fatigue limit in the UHCF range will be proposed. In particular, it will be argued that such fatigue life diagrams are not confined to materials containing inclusions, such as steels, and in which internal fatigue cracking, originating from the inclusions, has been observed in the UHCF range. Rather, similar fatigue life diagrams are also to be expected for pure single-phase metals and alloys in which cracks always initiate at the surface.

9:15 AM Invited
Fatigue: SEM Based Studies of Crack Initiation and Early Growth: *Angus J. Wilkinson*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxon OX1 3PH UK

This talk will illustrate our use of SEM based diffraction methods in the study of some fatigue problems. The electron channelling contrast imaging (ECCI) method has allowed us to image dislocation substructures in bulk samples in the SEM. Some advantages afforded by ECCI are (i) large areas can be observed, (ii) repeated intermittent observations of the same area can be made, and (iii) sample containing cracks are readily studied. Examples of direct imaging of dislocation configurations at the tips of stage I and stage II cracks will be given. The use of electron back scatter diffraction in studying the effects of grain boundary geometry on the strength of short fatigue crack retardation will also be discussed. Studies in Al-Li 8090 alloy have shown that cracks propagate onto slip planes in the next grain so as to minimise the twist deflection, and for those boundaries at which the minimal twist deflection is large the crack retardation tends to be strong.

9:45 AM Cancelled
Mechanisms of High-Cycle Fatigue Crack Growth and Investigations on the Influence of Loading Frequency: *Stefanie Stanzl-Tschegg*

9:45 AM Invited
A New Concept of Sn Curve and Fatigue Strength in the Gigacycle Fatigue: Claude Bathias[1]; [1]CNAM/ITMA, 2 rue Conte, Paris 75003

Safe-life design based on the infinite-life criterion was initially developed through the 1800s and early 1900s, one of which is the stress-life or S-N approach related to the asymptotic behavior of steels. A lot of materials display a fatigue limit or "endurance" limit at a high number of cycles (typical $>10^6$). Most other materials do not exhibit this response, instead displaying a continuously decreasing stress-life response, even at a great number of cycles (10^6 to 10^9), which is more correctly described by a fatigue strength at a given number of cycles. Time and cost constraints usually rule out the use of conventional fatigue tests of more than 10^7 cycles to check the structural materials. In contrast to conventional fatigue tests, which require a long duration of time due to the high numbers of load cycles at low frequencies (typical < 50 Hz), the proposed piezoelectric fatigue technique operates at much high frequency (about 20 KHz). Thus the required time for measurements in the high cycles fatigue range is considerably reduced. In the area of application and design, high-cycle fatigue (HCF) failures are very common. Unfortunately, they are not well understood nor there is a methodology available. The current standardized design methodologies are unable to accurately predict HCF damage. The goal of the research is to develop experimental and analytical techniques to understand the onset of HCF behavior of structural materials. The mechanisms of fatigue cracks have been determined thanks to microfractographic analyses on the scanning electron microscope. The fatigue strength is also estimated by the Murakami model, which has been applied to many fatigue problem of high strength steels including nonmetallic inclusions and small defects, but the model does not fit the gigacyclic fatigue well. A modified empirical equation was proposed to predict high cycle fatigue life of high strength steels. Investigations concerning long fatigue lives ($>10^7$ cycles) are relatively rare. The reason is obvious, the testing time and costs are too much to perform the fatigue tests of more than 10^8 cycles using a conventional testing machine. A possibility of accelerated tests of the structural materials (metals, alloys, composites) is considered by using high frequency cyclic loading. The ultrasonic fatigue is one of the most economical and practical ways to tests the structural materials. The S-N curves for the high strength aloys were obtained between 10^5 and 10^9 cycles. The most noticeable was that the continuous failure could occur over 10^7 cycles and the fatigue limit could not be obtained until 10^9 cycles. It should be described by a fatigue strength at a given number of cycles, and realized the risk of obtaining the fatigue limit at 10^7 cycles.

10:15 AM Break

10:25 AM Keynote
Assessment of the Origin of a Gas Turbine Disk Crack: *David L. Davidson*[1]; [1]Southwest Research Institute, 117 Elm Spring Ln., San Antonio, TX 78231 USA

An analysis has been made to determine the cause of cracking found in the disk of a fighter aircraft gas turbine engine. The investigation integrates (1) fractography of the attachment surface and metallography of the cracked disk, (2) stress analysis of the blade/disk attachment, (3) recent theoretical analyses of fretting fatigue, and (4) fatigue crack growth and stress-life characterization for the disk material. Information about the cracking gained from each of these techniques has uncertainty associated with it, for reasons specific to each. Thus, the cause of the crack remains uncertain (at least at the time of this abstract), but the analysis helps to clarify additional research that is needed to prevent this type of disk cracking.

10:55 AM Invited
Analysis of Fretting Fatigue with Spherical Contact in 7075-T6 Aluminum Alloy: *Jaime Dominguez*[1]; [1]ESI, University of Seville, Dept. of Mechl. Eng., Camino de los Descubriminentos s/n, Sevilla 41092 Spain

This paper analyses some aspects of fretting fatigue in 7075-T6 aluminum alloy. The case of uniaxial elements under cyclic loading and the action of a spherical fretting pad subjected to constant normal loading is considered. Friction forces and stresses involved in partial slip and small amplitude global sliding between the surfaces in contact are examined. The analysis includes an approach to calculate analytically the displacement of the stick zone induced by the bulk stress applied to the specimen. A series of tests controlling normal and tangential contact loads, and cyclic displacements in addition to the fatigue loading parameters cyclic stress amplitude, frequency, and load ratio has been carried out. Metallographic and fractographic examinations have been done, and the experimental results have been critically examined in the light of a variety of known multiaxial fatigue criteria. A method for estimating the total fatigue life in fretting fatigue is proposed.

11:25 AM
Orientation Dependence of the High Cycle Fatigue Properties in an Al-Li 8090 Alloy: *Tongguang Zhai*[1]; Angus J. Wilkinson[2]; John W. Martin[2]; [1]University of Kentucky, Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506 USA; [2]University of Oxford, Matls., Parks Rd., Oxford OX1 3PH UK

High cycle fatigue properties of a hot-cross rolled Al-Li alloy plate (45 mm thick) were studied in the rolling, transverse and short transverse directions of the plate. The fatigue tests were conducted at a stress amplitude between 40%-100%sy, R=0.1, room temperature in air using a self-aligning four-point bend rig which was able to minimize all the possible misalignment commonly associated with a four-point bend test and allowed measurement of the fatigue property in the short transverse direction of the Al-Li alloy plate. It was found that the high cycle fatigue property in the short transverse direction was significantly inferior to those in the rolling and transverse directions. The crack in the short transverse samples were predominantly intergranular, whereas they were mainly crystallographic (along a 111 plane in each grain) in the rolling and transverse samples. The number of secondary cracks observed on the surface between the two loading bars varied markedly with the stress amplitude as well as the loading direction.

11:45 AM
Temperature Evolution during Fatigue: *Bing Yang*[1]; Peter K. Liaw[1]; Hsin Wang[2]; J. Y. Huang[3]; R. C. Kuo[3]; J. G. Huang[4]; [1]University of Tennessee-Knoxville, Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [3]Institute of Nuclear Energy Research (INER), PO Box 3-14, 1000 Wenhua Rd., Chiaan Village, Lungtan 325 Taiwan; [4]Taiwan Power Company, Nucl. Oper. Dept., Taipei 100 Taiwan

An infrared (IR) thermography technique, as a nondestructive evaluation technique, was applied to investigate the fatigue damage of Reactor Pressure Vessel (RPV) Steels during 0.5 Hz, 20 Hz and 1,000 Hz fatigue testing. Five stages of temperature evolutions were observed: an initial increase of the average specimen temperature, a followed temperature decrease, an equilibrium (steady-state) temperature region, an abrupt rise of the temperature, and a drop of temperature following specimen failure. The relationship among the temperature, stress-strain state, and fatigue behavior is discussed. Both thermodynamics and heat transfer theories are applied to model the observed temperature variation during fatigue. The predicted and measured temperature evolutions during fatigue were found to be in good agreement. Temperature evolution along the specimen gage length is predicted. Furthermore, the back calculation from the observed temperature to obtain inelastic deformation indicates the material damage during fatigue. Life prediction using measured temperature was performed.

Energy Issues in the Aluminum Industry – A Keynote Symposium

Sponsored by: Aluminum Association
Program Organizer: TMS, Warrendale, PA 15086 USA

Monday AM — Room: 602-603
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: John Allison, Ford Motor Company, Scientific Rsrch. Lab., MD 3182, Dearborn, MI 48124-2053 USA

8:30 AM Keynote
The Perspective and Affects of Energy and Industry: *Brett Wilcox*[1]; [1]Golden Northwest Aluminum, Inc.

For years, the aluminum industry and the Pacific Northwest have maintained a mutually beneficial relationship based on the region's supply of sufficient and affordable electricity until power shortages and subsequent increased pricing almost completely curtailed primary aluminum production.

9:00 AM Keynote
Dealing with the Energy Crises by Reducing Demand: *Paul E. Norman*[1]; [1]Power Business Line

In April of 2001, BPA was faced with the possibility of raising wholesale rates to Northwest utilities and large industries by 250 to 300 percent. High market prices were the reason. BPA knew that it had to limit its exposure to the market if it was going to get the rate increase down to double digits. In less than two months, using an aggressive load reduction strategy, BPA–with the tremendous cooperation from the region and a major contribution from the aluminum industry–reduced the rate increase to 46 percent.

9:30 AM Keynote
Energy Policy Position of the Aluminum Association: *Robin King*[1]; [1]The Aluminum Association

Energy represents about one third of the total production cost of primary aluminum. Electricity is an essential ingredient in primary aluminum production. These factors together make energy efficiency and energy management prime objectives for the industry. The Aluminum Association supports efforts to create an effective national policy that resolves the power-shortage crisis in the Northwest while sustaining and protecting the regional aluminum industry–accounting for almost 40 perfect of domestic primary production and 5 percent of world supply.

10:00 AM Break

10:20 AM Keynote
Aluminium and Energy–An International Perspective: *Richard Evans*[1]; [1]Alan Aluminium Limited and Alcan Fabrication Group

A review of the effects of regional energy and trade issues on the global aluminium industry and its markets and possible future international trends and scenarios.

10:50 AM Keynote
New and Emerging Technologies: *Jud W. Virden*[1]; [1]Energy and Technology Division, Pacific Northwest National Laboratory

How quickly does science fiction become science fact? You may be surprised, especially with the upcoming technologies to help increase your energy efficiency and reduce your energy usage. Jud Virden will speak on new and emerging technologies that will soon change where you could get your energy, how you build your buildings and what will power your equipment. Hear about fuel cells for distributed power systems and automobiles, miniature technologies, smart appliances and more–the stuff of science fiction coming to you soon!

11:20 AM Panel Discussion

Flyash: Generation, Treatment, Metal Recovery and Disposal - I

Sponsored by: Extraction & Processing Division, Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; I. Gaballah, Laboratoire Environmental et Mineralurgie, Associated to CNRS, ENSG-LEM, Vandoeuvre les Nancy 54501 France; David G. Robertson, University of Missouri-Rolla, Department of Metallurgical Engineering, Rolla, MO 65409-1460 USA

Monday AM — Room: 604
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Junji Shibata, Kansai University, Dept. Cheml. Eng., Osaka 564-8680 Japan; Ji-Whan Ahn, Korea Institute of Geoscience and Mineral Resources, Teajon, Korea

8:30 AM Invited
R&D for Recycle and Decontamination of Fly Ashes from Municipal Waste Incineration: *Nobuyuki Masuda*[1]; Nobuyuki Okamoto[1]; Eiichi Arai[1]; Kazuyuki Kikuta[1]; [1]Metal Mining Agency of Japan, 1-24-14, Toranomon, Minato-ku, Tokyo, 105-0001 Japan

In Japan, the municipal solid waste, which amounts to 50 million tons, is generated every year and most of it is incinerated. The bottom and fly ashes are disposed to the registered disposal areas under the provisions of The Waste Disposal and Public Cleaning Law. Especially, as the fly ash from the municipal waste incineration (the primary fly ash) contains heavy metals (lead, zinc, etc) and dioxins, it cannot be disposed directly without decontamination, such as melting, cementation, chelating and dissolving processes provided in the law. However, these procedures for decontamination, except melting, are not enough for the elimination of dioxins. Even in case of melting, the fly ash from the process (the secondary fly ash) contains high concentration of heavy metals (e.g., Zn; 1-20%, Pb; 1-10%). Because of these reasons, Metal Mining Agency of Japan (MMAJ) that is a semi-governmental organization started a four-year project to develop the treatment technologies of these fly ashes in 1999. The purpose of the project is to establish the integrated technologies to recover the valuable metals from, and to decontaminate, the primary and secondary fly-ashes in the practical scale by utilizing the existing metallurgical processes and facilities, along with the energy saving and the reduction of the environmental impact.

9:00 AM Invited
Fly Ash: A Resource Material for Zeolite Production: *R. N. Singh*[1]; S. S. Rayalu[1]; S. U. Meshram[1]; Pawan Kuwar[1]; M. Z. Hasan[1]; [1]National Environment Engineering Research Institute, Nehru Marg, Nagpur 440 020 India

India is bestowed with substantial coal reserves, with generally high ash contents (40-50%). Utilization of this coal, as a source of energy, especially in thermal power plants generates extremely high quality of fly ash. Realizing adverse effects of fly ash, stringent regulations for its disposal have been formulated. According to ASTM (D-5239-92) classification, Indian fly ashes generally fall in the class F type; wherein the sum of silica and alumina and iron oxide corresponds to a minimum of 75%. These fly ashes have been employed for a promising application in vogue viz. production of fly ash based zeolites (FAZs). Studies conducted on zeolite synthesis from fly ash of class F-type indicated that this particular fly ash is the most amenable material for synthesis of FAZs. Due to lack of sufficient information on this virtuous value added product from fly ash, the fly ash producers and users did not exploit this technology; which definitely has edge over other fly ash utilization technologies in vogue due to attractive profit margins. Fly ash based zeolite (FAZs) was synthesized and investigated for surface area, crystallinity, sorption capacity, calcium binding capacity, particle size, etc. Calcium binding capacities of FAZ-A and FAZ-Y are about 540meq/100 g and 420meq/100 g respectively. The crystallinity of FAZ-A and FAZ-Y is found to be 99% and 97% respectively in comparison with commercial standards. Surface areas of FAZ-A, FAZ-Y are 580 m2/g, and 480 m2/g, respectively. The presentation high-

lights innovative usage of fly ash, and hydrothermal chemistry of novel FAZ synthesis.

9:30 AM

Treatment and Recycling of Incinerated Ash using Thermal Plasma Technology: T. W. Cheng[2]; *Jinn P. Chu*[1]; C. C. Tzeng[3]; Y. S. Chen[1]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., No. 2, Pei-Ning Rd., Keelung 20224 Taiwan; [2]National Taipei University of Technology, Dept. of Matls. & Minl. Resources Eng., No. 1, Sec. 3, Jung-Hsiao E. Rd., Taipei 106 Taiwan; [3]Institute of Nuclear Energy Research, Phys. Div., 1000, Wenhua Rd., Chia-An Village, Lung-Tan 235 Taiwan

Thermal plasma vitrification is a robust technology to treat and recycle the ash residues. Under the high temperature plasma environment, incinerated ashes are vitrified into benign slag with large volume reduction and extreme detoxification. Several one-step heat treatment processes are carried out in the present study at four temperatures (i.e. 850°C, 950°C, 1050°C and 1150°C) to obtain various "microstructure materials." The major phase to form these materials is a solid solution of gehlenite (Ca2Al2SiO7) and åkermanite (Ca2MgSi2O7) belonging to the melilite group. The physical and mechanical properties of the microstructure materials are improved by using one-step post-heat treatment process after plasma vitrification. These microstructure materials with good quality have great potential to serve as a viable alternative for construction applications.

9:50 AM Break

10:05 AM

Environmentally Friendly Usage Method of Zeolite Derived from Coal Fly Ash: *Junji Shibata*[1]; Norihiro Murayama[1]; Hideki Yamamoto[1]; [1]Kansai University, Dept. of Cheml. Eng., Fac. of Eng., 3-3-35, Yamatecho, Suitashi, Osaka, 564-8680 Japan

Zeolite synthesized from coal fly ash has many intelligent properties such as cation exchange ability, adsorption ability and so on. The usage method of the zeolite, however, is not established in actual commercial scale yet. In this study, from the viewpoints of an effective usage for coal fly ash and a source recycling, we propose the new concept for environmentally friendly usage of zeolite, in which coal fly ash safely returns to the soil with growing plants and purifying wastewater. This method can make great contribution to recycle of coal fly ash, to purification of high nutritious water and to tree planting.

10:25 AM

Industrial Fly Ash as a Soil Amendment Agent for Raising Forestry Plantations: *Dinesh Goyal*[1]; K. Kaur[2]; R. Garg[2]; V. Vijayan[3]; S. K. Nanda[4]; A. Nioding[4]; Sunil Khanna[1]; V. Ramamurthy[2]; [1]School of Biotechnology, Thapar Inst. of Eng. & Tech., Patiala, 147 004 India; [2]Thapar Center for Industrial Research & Development India; [3]Institute of Physics India; [4]Ballarpur Industries, Ltd.

Industrial fly ash from (electro static precipitator) was mixed with soil at different levels on v/v basis for raising of pulpwood tree species. In field trials conducted on degraded soils increase in the plant growth of Eucalyptus tereticornis, Acacia auriculiformis and Casuarina equisetifolia by 10%, was observed during the initial six months after plantation at 18-24% (v/v) fly ash concentration which was at par with chemical fertilizer treatment. Level of As, Se and Mo in fly ash was 6.2, 3.6 and 4.0 ppm respectively. The radioactivity analyses of soil and fly ash showed that the activity levels of gamma emitting radionuclides 40K, 226Ra, 228Ac were within the permissible limits and mixing of fly ash with soil at tested concentration would have no deleterious effects. Available Pb, Co, Cr, Ni and Cd in rhizospheric soils were below detection limit. Fly ash at 10-20% level (v/v) was also found to be a good potting mix material in forestry nurseries in terms of improving biomass production by 20%, when tested in nursery seedlings of Eucalyptus tereticornis. Nursery addition of fly ash in soil decreased soil dehydrogenase activity and the soil bacterial population was negatively affected. These results indicate that use of fly ash in forestry sector involving nursery and plantations as a soil amendment agent up to 10-20% v/v is a gainful utilization of fly ash.

10:45 AM

Hydrothermal Synthesis and its Reaction Mechanism of K Type Zeolite from Coal Fly Ash using KOH Solution: *Norihiro Murayama*[1]; Hideki Yamamoto[1]; Junji Shibata[1]; [1]Kansai University, Dept. of Cheml. Eng., Fac. of Eng., 3-3-35, Yamatecho, Suitashi, Osaka, 564-8680 Japan

Hydrothermal synthesis for K type zeolite is carried out from coal fly ash by using KOH as an alkali source. Through the overall reaction of zeolitization, the changes in various properties of obtained zeolite, such as the kind of zeolite crystal, cation exchange capacity, surface structure of zeolite, particle size distribution, concentration of various ion species in alkali solution and so on, are investigated in this study. From these results, the reaction mechanism of K type zeolite from coal fly ash can be clarified.

11:15 AM

Making Use of Fly Ash to Protect Environment–An Experience at Hindalco Industries, Ltd.: *A. K. Karmakarsr*[1]; [1]Hindalco Industries, Ltd., Renukoot, UP 231217 India

Hindalco Industries, Ltd., Renukoot is situated at the bank of Rihand Dam. This leads to stringent control of Fly ash disposal in the open area- by the side of the Dam. The co-generation plant at Renukoot is responsible to feed steam required to produce 4,50,00 MTPY alumina from "Low alumina-Bauxite" ore. This leads to the problems of disposal of 'Red-Mud' generated out of the alumin are finery and the Fly ash disposal from the Co-generation Plant. Primarily these disposable items are being used in the land filing of the Hill-area surrounding Renukoot. Looking into the disposal problems, Hindalco decided to change over the 'Wet Ash Disposal System' to Dry ash Disposal System and to utilize 'Dry Ash' -To protect environment from washing off the red-mud and fly ash, during rainy season, -To abide by the Regulation of fly ash disposal of Govt. of India. The fly ash from Electrostatic Precipitators is collected in silo under vacuum conditions. The fly ash, thus, collected is mainly used in Cement Plants located in near vicinity and also to develop land for vegetation. This article presents the chronological changes brought by the Co-generation Plant to use Fly ash and to protect environment by growing vegetation in the ash disposal area. This has yielded good results in protecting environment. This paper shares the experience of utilization of fly ash at Hindalco Co-generation Plant.

11:35 AM

Simultaneous Removal Technology of N and P Components in Aqueous Solution by Ca Type Zeolite Synthesized from Coal Fly Ash: *Hideki Yamamoto*[1]; Norhiro Murayama[1]; Junji Shibata[1]; [1]Kansai University, Dept. of Cheml. Eng., Fac. of Eng., 3-3-35, Yamatecho, Suitashi, Osaka, 564-8680 Japan

The simultaneous removal for NH4+ and PO43- in aqueous solution is carried out by using Ca type zeolite synthesized from coal fly ash. The effect of pH on cation exchange capacity and adsorption properties of obtained zeolite, the simultaneous removal ability for NH4+ and PO43- in various solutions and the mechanism of simultaneous removal were investigated, respectively. The Ca type zeolite used in this study can simultaneously remove NH4+ and PO43 in aqueous solution without releasing co-ion, which gives great advantages over the other ordinary removal methods.

Fundamentals of Advanced Materials for Energy Conversion: Superconductors

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee

Program Organizers: Renato G. Bautista, University of Nevada-Reno, Department of Chemical and Metal Engineering, Reno, NV 89557-0136 USA; Dhanesh Chandra, University of Nevada-Reno, Metallurgical & Materials Engineering, Reno, NV 89557 USA

Monday AM — Room: 613
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Amit Goyal, Oak Ridge National Laboratory, PO Box 2008, MS 6116, Oak Ridge, TN 37831-6116 USA; Renato G. Bautista, University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA

8:30 AM Plenary

An Overview of Advanced Materials for Energy Conversion: *Dhanesh Chandra*[1]; Renato G. Bautista[1]; [1]University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA

An overview of advanced materials for energy conversion and storage will be presented. In this symposium, emphasis will be given to superconducting, magnetic, fuel cells/membrane, hydrogen and tritium storage, battery, and thermal energy storage materials. The advances in second-generation superconductors for large-scale applications, magnetic materials for heating and cooling used in home air conditioners and refrigerator/freezers, thermoelectric power developments for electronic applications, and battery materials will be presented. Ad-

vances in core materials for transformers and motors using amorphous (glassy) ferromagnetic glasses rather than crystalline Fe-Si alloys, which show very low coercivity and hysteresis losses have recently received considerable attention. Several advances have been made in the development of fuel cells, membrane materials, interconnects for high temperature planar SOFC's. Hydrogen and tritium storage in intermetallic and other complex hydrides look very promising; recent advances will be discussed. In addition, lightweight alanate hydrides have shown significant improvements. The fundamental understanding of emerging Clathrate hydrates materials for solid-state methane storage, and organic thermal energy storage materials will also be discussed.

8:50 AM Plenary
Second Generation High Temperature Superconducting (HTS) Wires for Large-Scale HTS Devices: *Amit Goyal*[1]; [1]Oak Ridge National Laboratory, PO Box 2008, MS 6116, Oak Ridge, TN 37831-6116 USA

A review of advances made towards the development of second generation superconducting wires which are necessary for most large-scale, bulk applications of high temperature superconducting (HTS) materials will be given. Particular emphasis will be given on wires made using the rolling-assisted-biaxially-textured-substrates (RABiTS), a technique to fabricate long lengths of flexible, single-crystal-like, HTS wires. The technique involves creating a single-crystal-like, biaxially textured metal substrate which can be done using simple rolling and annealing procedures. This is followed by epitaxial deposition of oxide buffer layers and superconductors. Superconducting properties of wires made using the second generation wire technology approach those of single-crystal superconductors. Such wires appear to be very promising for many large-scale applications involving superconducting devices.

9:20 AM Invited
Superconducting Electric Power Applications: HTS Conductor Requirements: *Richard Blaugher*[1]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA

Recent worldwide successes in demonstrating high-temperature superconductors (HTS) in electric power apparatus have buoyed the prospects for this technology. These achievements were only possible due to the rapid worldwide progress in developing HTS wire and tape. This talk will discuss the overall progress in developing superconducting electric power components. The early LTS work on energy storage (SMES) and electric power generators will first be reviewed followed by a discussion on current programs using HTS conductor. The major problems facing this technology will also be discussed, as well as the prospects for commercialization and integration into the utility sector. The eventual widespread utility acceptance for superconducting power equipment is dependent on demonstrated improvements in system performance and efficiency. The reliability and maintenance must also be comparable to conventional power equipment and offer reduced life-cycle costs and a "competitive" installed cost. The latter is impacted by the current high cost of HTS conductors that must be lowered to costs comparable to conventional copper wire. This cost factor, coupled with the requirement for improved HTS transport performance in magnetic fields greater than one tesla at 77K, are the critical factors that will limit or seriously compromise the consideration of HTS power components for the power sector. (Supported by the Department of Energy Superconductivity Program for Electric Power Systems.)

9:45 AM Invited
Flywheel Electricity Storage System using High Temperature Superconductors: *Michael Strasik*[1]; [1]Boeing, PO Box 3707, Seattle, WA 98124-2207 USA

Boeing Phantom Works, Seattle, Washington, and six partners are developing a 10-kWh flywheel energy storage system using superconducting bearings. The system will help utility companies and other large power users eliminate power surges that often disrupt reliable transmission of power. Flywheels based on frictionless superconducting bearings improve power quality and reliability. Boeing has also had a leading role in the push to use flywheels in satellites and to replace batteries in the International Space Station. The reason is because flywheels wont wear out like batteries. Batteries fail after a limited number of charge/discharge cycles. By simply replacing batteries with flywheels, you can extend the life of some satellite systems. A proof-of-concept system has been designed, built and tested.

10:10 AM Break

10:25 AM Invited
Operation of the Southwire 30-m High Temperature Superconducting Cable Installation: *David T. Lindsay*[1]; R. L. Hughey[1]; Mark Roden[1]; Uday Sinha[1]; Mike Gouge[2]; John Stovall[2]; [1]Southwire Company, Wire & Cable Tech., One Southwire Dr., Carrollton, GA 30119 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Southwire Company of Carrollton, Georgia in cooperation with Oak Ridge National Laboratory has designed, built, installed and is operating the world's first field installation of a High Temperature Superconducting (HTS) cable system. The cables supply power to three Southwire manufacturing facilities at the company's main campus in Carrollton, GA. The system consists of three 30-m single phase cables rated at 12.4 kV, 1250 Amps, liquid nitrogen cooling system, and the computer-based control system. The cables are built using BSCCO-2223 powder-in-tube HTS tapes and a proprietary cryogenic dielectric material called CryoflexÔ. The cables are fully shielded with a second layer of HTS tapes to eliminate any external electric fields. The Southwire HTS cables were first energized on January 6, 2000. To date, they have logged over 8,200 hours of operation while supplying 100% of the required customer load. The cables have worked without failure and operations are continuing.

10:50 AM Invited
High Temperature Superconductor-Based HTS Electric Power Devices: *Venkat Selvamanickam*[1]; [1]IGC-SuperPower, 450 Duane Ave., Schenectady, NY 12304 USA

IGC-SuperPower is developing High-Temperature Superconductor (HTS)-based products for the electric power industry, specifically cables, transformers, and fault-current controllers. These HTS devices are expected to result in higher efficiency, lower losses, increased capacity, and generate environmental benefits. The HTS conductor that will be used in all these devices is a high-performance coated conductor fabricated by thin film processes. GC-SuperPower has established pilot-scale facilities for scale up of Coated Conductor processes to manufacturing. These facilities have been designed for continuous processing of substrates, buffer layers, and superconducting layers over time periods of a week. These facilities are being used to scale up coated conductor technology to manufacturing. The overall activities on scale up of Coated Conductor processes at IGC-SuperPower and the progress with HTS devices for electric power will be summarized in this presentation. Part of the work was performed under a CRADA with Los Alamos and Argonne National Labs.

11:15 AM Invited
Integrated Momentum and Energy Storage for Mini-Satellites: *Ki Bui Ma*[1]; Yong Zhang[1]; Yevgeniy Postrekhin[1]; Wei-Kan Chu[1]; [1]University of Houston, TX Ctr. for Superconductivity, 3201 Cullen Blvd., Houston, TX 77204-5002 USA

Momentum wheels with superconducting bearings can integrate both attitude control and energy storage functions, with attendant savings in the total volume and weight of hardware, and power consumption, all of which are in short supply on mini-satellites. We have built and tested a prototype with a wheel of mass 1.9 kg, diameter 3.25 in, and height 3 in, which can store 3.5 J-sec of angular momentum and 5 kJ of energy when it rotates at an angular speed of 15000 RPM. The energy density of 2.5 kJ/kg achieved is comparable with the lower range for lead-acid batteries. To show that the wheel is energy efficient, we have performed spin down tests and measured the input power required to sustain rotational speed. Results indicate that power consumption, even accounting for the needs of cooling system, is significantly smaller than that for state of the art commercial reaction wheels.

11:40 AM Invited
Crystal Lattice Defects, Nanoscale Structure, and the Properties of Advanced Superconductors: *David O. Welch*[1]; [1]Brookhaven National Laboratory, Matls. & Cheml. Scis. Div., Bldg. 480, PO Box 5000, Upton, NY 11973-5000 USA

Optimization of the properties, especially the current carrying capacity, of advanced superconductors requires a very high level of control of crystal lattice defects and their organization into nanoscale structure in order to minimize the effects of weak links and maximize the pinning of magnetic flux. This degree of control gives rise to the need for a deep understanding of the effects of defects on charge distributions, elastic strain fields, as well as local atomic and electronic structure. A combination of modern methods of electron microscopy, microscale transport property measurements, and theoretical analysis facilitates the achievement of such understanding. I will use results from such an approach to contrast the character and effects of defects in high-Tc cuprates and the latest addition to the family of advanced superconductors, the intermetallic compound MgB2. This research was supported by the US Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under Contract No. DE-AC02-98CH10886.

Fundamentals of Structural Intermetallics: Environmental Effects and Fe Aluminides

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials

Program Organizers: Young-Won Kim, UES, Inc., Materials & Processing Division, Dayton, OH 45432 USA; Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Vijay K. Vasudevan, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Monday AM — Room: 615-616
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Ronald Gibala, University of Michigan, Dept. of Matls. Sci. & Eng., 2026 H.H. Dow Bldg., Ann Arbor, MI 48109-2136 USA; Seetharaman C. Deevi, Chrysalis Technologies Inc., Rsrch. Ctr., 7901 Whitepine Rd., Richmond, VA 23237 USA

8:30 AM Opening Remarks

8:40 AM
Moisture-Induce Hydrogen Embrittlement of Ordered Intermetallics: *Chain T. Liu*[1]; Easo P. George[1]; Lee M. Pike[2]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., #1 Bethel Valley Rd., Oak Ridge, TN 37831 USA; [2]Haynes International, Eng. & Tech., Kokoma, IN 46904-9013 USA

This paper summarizes recent advances in understanding moisture-induced environmental embrittlement in intermetallics. The embrittlement occurring at ambient temperatures is quite different in fcc- and bcc-ordered intermetallics. It causes brittle intergranular fracture without affecting the yield strength of intermetallics with the L12 structure. On the other hand, bcc-ordered alloys exhibit cleavage fracture and reduced yield strength when tested in moist air. The change in yield strength may not be detected in some cases because of excess point defects induced in bcc-ordered alloys. Hydrogen diffusion along the grain boundary and bulk is responsible for the different embrittlement behaviors observed in fcc- and bcc-lattices. This research was sponsored by US DOE Division of Materials Sciences and Engineering under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:10 AM
Surface Chemistry of Adsorbates on Intermetallics: *Masahiro Inoue*[1]; Katsuaki Suganuma[1]; [1]Osaka University, The Inst. of Scientific & Indl. Rsrch., 8-1 Mihogaoka, Ibaraki, Osaka 567-0047 Japan

The surface modification technology is expected as one of the useful techniques for extending material's lifetime because the environmental degradation of intermetallics and related materials is initiated by surface reaction of adsorbates. In order to establish the techniques for extending lifetime, the surface chemistry is necessary to understand in detail. This paper discusses the adsorption behavior of environmental species on intermetallic alloys. The adsorption usually occurs through the steps; back donation from the intermetallics, followed by relaxation and decomposition of adsorbates. The adsorption mechanism of these species on aluminides is studied using the molecular mechanics and first principle molecular orbital simulation.

9:30 AM
Water Dissociation on Single Crystal Ni3(Al,Ti) and (Ni,Fe)Ti Surfaces: Monica de Mesquita Lacerda[1]; Jinliu Wang[1]; *Yip-Wah Chung*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

While the mechanism of moisture-induced embrittlement in polycrystalline Ni3Al is well understood, the role of boron to suppress this embrittlement is less clear. In this work, temperature-programmed desorption and x-ray photoelectron spectroscopy were carried out after D2O dosing on clean Ni3(Al,Ti) (110) surface at < 130K, with and without surface boron. Water dissociates on clean surfaces at ~190 K, resulting in D2 evolution at ~400K. This D2 evolution is completely suppressed by ~0.30 monolayer boron. We found that atomic hydrogen is strongly bound to surface boron. Surface diffusion measurements showed that the surface diffusivity of atomic hydrogen is reduced by 10 times by 0.05 monolayer boron. This should lead to lower concentration of atomic hydrogen at the crack tip, thus explaining the improvement of ductility. We have extended the same surface science approach to understand the embrittlement of NiTi alloys due to Fe addition beyond 9 a/o.

9:50 AM
High Temperature Corrosion of Fe3Al in Chlorine-Containing Environments: *Gilsoo Han*[1]; W. D. Cho[1]; [1]University of Utah, Dept. of Metlgcl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

The high-temperature corrosion of iron aluminide (Fe3Al) in environments containing chlorine and oxygen has been studied using thermogravimetric method. The corrosion experiments were carried out in various oxygen to chlorine volume ratios at the temperature range of 750-900°C. The corrosion behavior in the mixed gas was observed to be significantly different from that in pure oxygen in terms of kinetics and corrosion products. The cross-section of the specimens and the corrosion products were examined using X-ray diffraction, scanning electron microscopy, and energy-dispersive X-ray spectroscopy. Based on the analysis of kinetic data and corrosion products, the corrosion mechanism has been discussed.

10:10 AM Invited
Yield Strength Anomaly in FeAl Single Crystals: *E. P. George*[1]; D. M. Wu[2]; I. Baker[2]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., 1 Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6093 USA; [2]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755-8000 USA

FeAl alloys exhibit the so-called yield strength anomaly. We report here the effects of orientation in B-free and B-doped FeAl single crystals. The single crystals were grown by the Bridgeman technique and oriented for single slip to facilitate determination of the critical resolved shear stress (CRSS). Yield strength was measured in tension as a function of temperature from 77-1173K. Optical and scanning electron microscopy were used to confirm that only a single slip system was active during plastic flow. The CRSS was plotted as a function of temperature for different single-slip orientations to determine if there is, in fact, an orientation dependence of the yield strength or if all orientations fall on one curve. The implications of these results for our vacancy-hardening theory [E. P. George and I. Baker, Phil. Mag. A, 77, 737-750 (1998)] are discussed. Research sponsored at ORNL by the Division of Materials Science and Engineering, US Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC, and at Dartmouth College by NSF grant DMR-9973977, by DOE contract DE-FG02-87ER45311, and by the Oak Ridge Associated Universities SHaRE program contract DE-AC05-76OR00033.

10:40 AM
Strain Rate Effect on the Anomalous Temperature Dependence of Flow Stress in a Fe-24at.%Al Intermetallic Alloy: *Tae Kwon Ha*[1]; Jin-Hwa Song[2]; Young Won Chang[1]; [1]Pohang University of Science and Technology, Ctr. for Adv. Aeros. Matls., San 31, Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 S. Korea; [2]Research Institute of Industrial Science and Technology, Reliability Assessment Ctr., Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-600 S. Korea

In this study, the effect of the strain rate on the anomalous temperature dependence of flow stress was mainly investigated in a Fe-base intermetallic alloy with the composition of Fe-24 at.% Al. Phase composition and degree of order in Fe-24 at.% Al alloy were varied by aging at temperatures corresponding to the various phase fields of $D0_3$, α+D03, α+B2, and B2. The room temperature deformation behavior of the Fe_3Al alloys with the various phase compositions and degree-of-orders has also been examined. Load relaxation tests and tensile tests were conducted at temperatures ranging from room temperature to 800°C and the results were analyzed based on the internal variable theory of inelastic deformation. Room temperature strength of the Fe_3Al alloy appeared to increase with decreasing degree of order and the hardening effect caused by the precipitation of disordered α phase was found to depend on the shape of the precipitate. While the α phases in the α+$D0_3$ region precipitated in the form of a network were found to have a strengthening effect, those in the α+B2 region precipitated in the form of a coarse island had no effect of strengthening. The room temperature strength of the Fe_3Al alloy increased with decrease in the degree of order. The peak strength was observed at higher temperatures with the strain rate increased. The maximum strength was obtained at 530°C, which is right below the $D0_3$-B2 transition temperature Tc of 550°C, under the strain rate of $5X10^{-2}$ s^{-1}. By employing the internal variable theory of inelastic deformation, flow stress of the Fe_3Al alloy could be described as the sum of contributions from internal stress and frictional stress. The anomalous temperature dependence of strength in the Fe_3Al alloy was mainly attributed to the internal stress.

11:00 AM
Mechanical Behavior of $L2_1$-Structured Compounds Fe_2AlMn and Fe_2AlTi: *I. Baker*[1]; M. Wittmann[1]; S. L. Johns[1]; V. N. Durand[1]; P.

R. Munroe[2]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755-8000 USA; [2]University of New South Wales, Matls. Sci. & Eng., Sydney, New South Wales NSW 2052 Australia

Single crystals of Fe_2AlMn were grown and confirmed, by X-ray diffraction, to have the $L2_1$-structure. Mechanical tests showed that the compound exhibits a yield strength anomaly and tensile ductility at room temperature, which depend on strain rate. Transmission electron microscope analyses of strained specimens showed that deformation occurs by glide of <111> dislocations at all temperatures. Complex anti-phase domain structures, some with a/4<111> and other with a/2<100> fault vectors, were also observed, consistent with the compound ordering from a b.c.c. and then a B2 structure. Tests have also been performed on polycrystals of the related compound Fe_2AlTi as a function of temperature. The results will be compared to FeAl and both Co- and Ni-based Heusler alloys, and the origin of the yield anomaly will be discussed. This work was funded by NSF grant DMR-9812211.

11:20 AM

Effect of Alloying Additions on the Strength and Creep Resistance of FeAl Alloys: *R. S. Sundar*[1]; D. H. Sastry[2]; S. C. Deevi[1]; [1]Chrysalis Technologies, Inc., 7801 Whitepine Rd., Richmond, VA 23237 USA; [2]Indian Institute of Science, Dept. of Metall., Bangalore India

Iron aluminides based on FeAl are well known for their excellent oxidation and sulfidation resistances. They are being developed to replace conventional steels and stainless steels for intermediate temperature applications. Due to their open B2 structure, binary FeAl exhibits significant creep deformation at temperatures greater than 700C. Alloying elements are necessary to improve the high temperature deformation resistance of FeAl. The present study aims to bring out the effect of various substitution and interstitial alloying elements on the strength and creep resistance of FeAl alloys. Carefully planned experiments reveal that the importance of processing and heat treatment conditions on the properties of FeAl alloys.

11:40 AM

Superplasticity of Coarse-Grained Iron Aluminides: Thermal Cycling Effects: *Jinn P. Chu*[1]; W. Kai[1]; H. Y. Yasuda[2]; Y. Umakoshi[2]; K. Inoue[3]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., No. 2, Pei-Ning Rd., Keelung 20224 Taiwan; [2]Osaka University, Dept. of Matls. Sci. & Eng., 2-1, Yamada-oka, Suita, Osaka 565-0871 Japan; [3]University of Washington, Dept. of Matls. Sci. & Eng., Seattle, WA 98195 USA

As a general rule, superplastic metals and ceramics normally have fine-grained structures (typically <10 μm). Nevertheless, Fe-Al based alloys have been shown superplasticity even with much coarser grain structures, >500 μm. Fe-Al based alloys have been demonstrated to exhibit all the deformation characteristics that conventional fine-grained superplastic materials possess. The objective of the present study is directed toward the microstructural examination of thermal cycle effects on superplastic properties of Fe-27Al alloys. To visualize the grain-structure evolution, the electron backscattered diffraction technique has been employed because it is capable of identifying the type of grain structures. The superplastic behavior will be presented and discussed in light of the microstructure/crystal structure results.

General Abstracts: Joining, Testing and Characterization

Sponsored by: TMS

Program Organizers: TMS, Warrendale, PA 15086 USA; George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA

Monday AM — Room: 310
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: John J. Stephens, Sandia National Laboratory, Albuquerque, NM 87185-0889 USA

8:30 AM

A Study of Active Metal Braze Alloys Based on the Ag-Hf Binary System: *John J. Stephens*[1]; F. Michael Hosking[1]; Fred G. Yost[2]; [1]Sandia National Laboratories, 1833, PO Box 5800, MS0889, Albuquerque, NM 87185-0889 USA; [2]Trapezium Technology, 1901 Cleopatra Ct. N.E., Albuquerque, NM 87112 USA

This talk will present the results of an experimental project which has examined the potential of two binary Ag-Hf alloys for use as active metal braze alloys. The composition (wt.%) of these alloys are 97.5Ag-2.5Hf and 96.6Ag-3.4Hf. Due to the absence of a complete binary phase diagram for Ag-Hf in the literature, the compositions were chosen by analogy with other, related binary phase diagrams. Due to the favorable thermodynamic tendency for Hf to react with a number of ceramic substrates, both alloys show considerable promise for metal/ceramic brazing based on initial wetting and ASTM F19 tensile button results. The microstructure and products of the reactions observed at the braze/ceramic interface will be compared those observed in other active metal braze/ceramic interfaces. This work was supported by the US Dept. of Energy under Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Department of Energy.

8:55 AM

Fracture Surface and Failure Mode Analysis of Polycrystalline Silicon: *Ryan C. Brodie*[1]; Marian S. Kennedy[1]; David F. Bahr[1]; [1]Washington State University, Mechl. & Matls. Eng., 201 Sloan Hall, Pullman, WA 99164-2920 USA

Disk-shaped compact tension (DCT) specimens made from CVD grown polycrystalline silicon were fabricated using water jet methods. Compliance and fracture behavior were tested as a function of temperature, from ambient up to 1100 K. The fracture surfaces of the DCT specimens were then analyzed using SEM to identify any possible variations in fracture mode associated with increasing temperature. Such variations include a change from transgranular to intergranular failure, and changes in crack initiation mechanisms. Using the load and displacement data along with the SEM fractography, the brittle-to-ductile transition (BDT) temperature for polycrystalline silicon can be estimated. The effects of elevated temperature annealing, altering the grain size from between 0.5 and 5 microns, are also discussed. Comparisons between the DCT specimens and indentation induced fracture on annealed specimens tested at room temperature will be shown.

9:20 AM

Use of Advanced Scattering and Microtomography Techniques to Study the Complex Anisotropic Microstructures of Ceramic and Metallic Deposits: *Jan Ilavsky*[1]; Andrew J. Allen[2]; Gabrielle G. Long[2]; Anand Kulkarni[3]; Francesco De Carlo[4]; Herbert Herman[3]; [1]University of Maryland, UNICAT, Bldg. 438E, 9700 S. Cass Ave., ANL, Argonne, IL 60439 USA; [2]National Institute of Standards and Technology, Matls. Microstructure Characterization Grp., 100 Bureau Dr., MS 8523, Gaithersburg, MD 20899 USA; [3]State University of New York, Dept. Matls. Sci. & Eng., Old Engineering, Z=2275, Stony Brook, NY 11794-2275 USA; [4]Argonne National Laboratory, Experimental Facilities Div., Bldg. 431, B003, 9700 S. Cass Ave., ANL, Argonne, IL 60439 USA

Thermal-spray (TS) and electron-beam plasma-vapor deposited (EB-PVD) coatings possess unique multicomponent void microstructures which are responsible for their complex engineering properties, but which also pose major challenges for their characterization. Multiple void systems with different anisotropies, wide range of void shapes and sizes, and general deposit fragility all present major obstacles to many characterization methods. Often used intrusion porosimetry and imaging of cross-sections, provide either only basic volumetric data or information that is limited in reproducibility and reliability. In contrast, a combination of small-angle scattering of X-rays and neutrons (SAXS and SANS) and X-ray microtomography (XMT) provides a robust, nondestructive, statistically-valid, and comprehensive, microstructure characterization. SAXS and SANS can be used to characterize quantitatively the void systems through an analysis of the apparent Porod surface area distributions and yield the calibrated total specific surface area of the voids. The techniques of anisotropic multiple SANS and SAXS can yield more volumetric information about the void systems, via the introduction of model assumptions. XMT provides a complementary 3D view of the undisturbed structure of these materials. The improved resolution of this technique makes it a viable tool for providing density map data which, when combined with SANS and SAXS, can provide microstructure characterization of complex materials from the nanometer up to the millimeter-scale. Examples of recent application of this combination of techniques to TS and EB-PVD coatings will be presented, and it will be shown how the results can be related to the coating properties.

9:45 AM Break

10:20 AM
Framework for Designing Interlayers for Ceramic-to-Metal Joints: *Jin-Woo Park*[1]; Patricio F. Mendez[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-047, Cambridge, MA 02139 USA

This work introduces a framework for evaluating the strength characteristics of ceramic-to-metal joints with multiple interlayers. Strain energy in the ceramic is used as a strength metric instead of maximum tensile stress. Based on the FEM analysis and order of magnitude scaling (OMS), simple analytical formulations between the strain energy and material properties are developed, which provide a guideline in designing multiple interlayers. Our analysis reveals the important role of multiple interlayers, which reduce the strain energy in the ceramic, increasing the strength of the joint. Based on the proposed design rule, Si3N4 to Inconel 718 joints have been brazed with single, double and triple interlayers and the joint strength was evaluated using a shear test. The experimental results support the design rules and confirm that strain energy is a good strength metric.

10:45 AM
Characterization of Photovoltaic Cell Solder Joints: *F. Michael Hosking*[1]; Michael A. Quintana[2]; [1]Sandia National Laboratories, PO Box 5800, MS0889, Albuquerque, NM 87185-0889 USA; [2]Sandia National Laboratories, PO Box 5800, MS0752, Albuquerque, NM 87185-0752 USA

Photovoltaic silicon cells are electrically connected by soldering copper strips to a deposited metal grid network. System performance depends on the reliability of the resulting solder joints. Typical soldering materials and processing conditions are reviewed. Nondestructive and metallographic test results are presented for a variety of soldered modules, including field-tested units. The coring technique for obtaining test samples is described. Factors that impact module durability and efficiency are discussed. Of particular concern to photovoltaic solder joints are extended exposure to hot, humid field conditions and temperature extremes. The effects of moisture, elevated operating temperatures, and thermal cycling on corrosion, intermetallic growth, and mechanical fatigue failures are examined. The formation of shorts or opens in the photovoltaic circuit can rapidly degrade module electrical efficiency and field life expectancy. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Dept. of Energy under Contract DE-AC04-94AL85000.

11:10 AM
Non-Destructive Evaluation of Ceramic Candle Filters using Artificial Neural Networks: *Kuan-Chen Fu*[1]; Yanqin Zhou[1]; Lijun Li[1]; [1]The University of Toledo, Civil Eng., 2801 W. Bancroft St., Toledo, OH 43606 USA

Ceramic candle filters play an important role in coal-base turbine system for a modern power plant. However, after exposure to the high pressure and high temperature in the gas turbine chamber, the physical property of the ceramic deteriorates over time and the effectiveness of the filters reduces. Their failure to perform may create catastrophic consequences for the multi-million dollar equipment downstream. A non-destructive evaluation procedure using an emerging technique known as artificial neural networks is proposed to examine the filters. In lieu of experimental data, the vibration signatures of filters damaged to various degrees are created by means of analytical simulation. Then, a feed-forward artificial neural network and a radial basis function neural network are built and trained to evaluate the signatures for the purposes of determining the filters' degree of deterioration. Excellent results have been obtained. The technique may be applied to evaluate structural components made of aluminum, alloy or other materials.

11:35 AM
Temperature Discontinuity Assumption at Solid-Liquid Interface in Rapid Cellular Solidification: *Eisaku Tokuchi*[1]; Kimioku Asai[1]; [1]Musashi Institute of Technology, Mechl. Eng., 1-28-1 Tamazutumi, Setagaya-ku, Tokyo 158 Japan

On the basis of experiments, a calculation method to predict all the solidification conditions and dendrite arm spacings of rapidly solidifying cells is proposed. The assumptions are no solute pile-up at cell tip and temperature discontinuity at solid-liquid interface. The calculation results theoretically evidenced temperature none-uniformity in the cross-sectional area of a cell and the adjoining liquid, and thus validated the no-pile-up assumption by way of thermodynamic restrictions of rapid cellular thickening that is a dynamic behavior at solid-liquid interface along with the temperature discontinuity. The calculation results are also in favor of limited selection of solidification conditions in a given rapid cellular solidification regime. The method was validated in principle for use in the rapid cellular solidification range less than 7im of dendrite arm spacing.

General Abstracts: Microstructural Phase Stability

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA

Monday AM — Room: 211
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Dan J. Thoma, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

8:30 AM
Grain Refinement of Cast p-Type Bi2Te3 Alloy by Severe Plastic Deformation: *Jae-taek Im*[1]; *Karl Ted Hartwig*[1]; Jeff Sharp[2]; [1]Texas A&M University, Dept. of Mechl. Eng., College Station, TX 77843-3123 USA; [2]Marlow Industries, Inc., 10451 Vista Rd., Dallas, TX 75238-1645 USA

The objective of this work is to refine the microstructure of cast p-type Bi2Te3 alloy by severe plastic deformation using multipass equal channel angular extrusion (ECAE). Successful extrusions are accomplished at a temperature of 500C and a rate of 4.23 mm/sec. The microstructure is characterized by polarized optical microscopy. The level of grain refinement is found to be dependent on total strain. The results indicate that ECAE processing is a viable method for grain refinement of cast Bi2Te3 alloy.

8:55 AM
Microstructure and Annealing Behavior of Cold Deformed Copper: *Mohammed Haouaoui*[1]; Karl T. Hartwig[1]; [1]Texas A&M University, Mechl. Eng., 319 Engineering Physics Bldg., Spence St., College Station, TX 77843-3123 USA

CDA 101 copper was deformed at room temperature to a strain of 4.6. 25.4 by 25.4 mm square copper bars were deformed by equal channel angular extrusion through four passes in a tool containing a 90° angle at a strain rate of approximately 2 per second. The nucleation of new grains from heavily deformed material was found to occur along sites with heavy distortions. Nucleation sites for recrystallized grain start in shear bands with subsequent growth in the direction of slip lines. The annealing behavior is found to be a function of the multipass extrusion route. Processing that causes intersection of shear planes creates more sites for nucleation and leads to a shift to lower recrystallization temperatures. All annealing curves exhibit a slight increase in hardness prior to the sharp drop that accompanies recrystallization. Optical microscopy reveals uniform recrystallized microstructures for the different routes.

9:20 AM
In-Situ Annealing Observations during Epsilon-to-Tau Phase Transformation in MnAl-Based Ferromagnetic Alloys: Cagatay Yanar[1]; Eric A. Stach[1]; *William A. Soffa*[1]; Jörg M.K. Wiezorek[1]; [1]LBNL, NCEM, Berkeley, CA USA; [1]University of Pittsburgh, Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

Mn-Al alloys of nearly equiatomic composition show a metastable ferromagnetic tau-phase (L1o) after suitable heat treatment, the magnetic properties of which are extremely sensitive to microstructure and defect structure. The formation of these complex defect structures has been attributed to atomic processes occurring at the migrating interphase interfaces during the "massive transformation" of tau-phase from the high-temperature epsilon-phase (A3). Models for the defect formation have been proposed. In this study, in-situ TEM annealing experiments have been performed to elucidate the details of the epsilon-to-tau transformation and defect genesis. Dynamic observations of the formation of twins, pseudo-twin and other defects are related to the proposed models. An additional mode of epsilon-to-tau transformation, namely a displacive transformation, was observed during in-situ TEM. This alternate displacive transformation and the massive mode compete during the in-situ experiments. Local stresses are believed to trigger the displacive mode. The role of these observations for microstructural control in tau-phase alloys is discussed.

9:45 AM
Critical Phenemona at the Martensitic Transition in the Shape-Memory Alloy Gold-Zinc: *Jason C. Lashley*[1]; Timothy Darling[1]; D. J. Thoma[1]; Albert Migliori[1]; F. Chu[1]; M. Lopez[1]; B. Lang[2]; Juliana Boerio-Goates[2]; Brian F. Woodfield[2]; [1]Los Alamos National Laboratory, Matls. Sci. Div., PO Box 1663, Los Alamos, NM 87545 USA; [2]Brigham Young University, Dept. of Chem. & Biochem., Benson Bldg., Provo, UT 84602 USA

Since the discovery of the shape-memory effect, the martensitic transition has been described within the framework of classical equilibrium thermodynamics as a first-order displacive transition. However, as we investigate the physical properties (elastic moduli, specific heat, and stress/strain measurements) through the martensitic transition in AuZn at cryogenic temperatures, we find clear signatures of recoverable plastic strain and a continuous (second-order) transition at 64.7 K. It is argued that the combination of equiatomic composition (removing internal strains) and a low transition temperature (reducing both diffusion and entropy effects) constrain the chemical potential and its derivatives to exhibit behavior that lies at the borderline between that of a first-order (discontinuous) and a continuous phase transition. For these reasons, we propose a critical point in composition-temperature space located at mole fraction, x = 0.5 Zn and T ~ 65 K, connecting two coexistence lines of first-order martensitic phase transitions. Further support of the critical point is based on resistivity data of (Pops and Ridley, 1970), cold-stage optical results of (Pops and Massalski, 1965), de Haas-van Alphen measurements of (Beck et. al., 1963).

10:10 AM
Nucleation of Stress-Assisted Martensite: *R. E. Hackenberg*[1]; J. C. Cooley[2]; K. C. Chen[3]; D. J. Thoma[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G770, Los Alamos, NM 87545 USA; [3]California Polytechnic State University, Dept. of Matls. Eng., San Luis Obispo, CA 93407 USA

Many unresolved questions exist regarding the nucleation of martensite, due to the small time and length scales over which this process occurs. Although shock-loading experiments done at T>Ms have clarified some aspects of martensite growth, the gas-gun techniques traditionally used have lacked the temporal resolution to bracket the initial nucleation event apart from autocatalytic nucleation of additional units from fully-grown martensite units. This study reports the use of much shorter duration (nanosecond scale) stress pulses generated by an ultrafast laser, which allow only partial growth of martensite units to be realized, thus avoiding autocatalytic effects. Progressive reduction in the pulse duration allows the nucleation site to be more clearly identified; TEM is employed to this end. Polycrystal and single crystal specimens are examined to assess the nucleation potency of grain and twin boundaries. Preliminary results will be presented for near-equiatomic Ni-Ti and ferrous alloys.

10:35 AM
Phase Relations in the Ti-Pt System in the 30 to 60 at.% Pt Region: *T. Biggs*[1]; L. A. Cornish[2]; M. J. Witcomb[3]; M. B. Cortie[2]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 6350 Stores Rd., Vancouver V6T 1Z4 Canada; [2]Physical Metallurgy Division, Mintek, PB X3015, Randburg, 2125 S. Africa; [3]University of Witwatersrand, Electron Microscope Unit, PB 3, Wits, 2050 S. Africa

In the titanium-platinum system, around compositions of 50 at.% platinum, a phase transformation can be observed from a B2 parent phase to an orthorhombic product. This TiPt phase transformation occurs around 1000C and is displacive in nature. The transformation temperature and hardness of the alloy varied with composition, reaching a maxima and minima respectively, at 50 at.%. The morphology and hardness of the orthorhombic phase varied with cooling rate. The morphology could be lath-like or finely twinned depending on the cooling rate. At lower platinum contents a novel phase, which has not been reported before, was observed. Investigations suggested that the phase formed peritectically around 1200C. The TiPt phase transformation was observed to continue in the neighbouring two-phase regions, at high (TiPt and TiPt3) and low platinum contents (TiPt and a novel phase). At even lower platinum contents, the Ti3Pt phase and the novel phase were observed to form eutectically.

11:00 AM
Microstructure and Phase Chemistry of IN 100: Characterization and Phase Calculations: *Agnieszka Wusatowska-Sarnek*[1]; Gautam Ghosh[2]; Mark Aindow[1]; Martin Blackburn[1]; Gregory B. Olson[2]; [1]University of Connecticut, Dept. of Metall. & Matls. Eng., Inst. of Matls. Sci., 97 N. Eagleville Rd., Storrs, CT 06269-3136 USA; [2]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208-3108 USA

The fidelity of thermodynamic calculations of phase stability and constitution continues to improve rapidly and it is now possible to simulate the behavior of complex multi-component systems. In the current work the results of a comprehensive analysis of the structure of a powder metallurgical superalloy IN100 are compared with the results of such computations. A variety of analytical methods was used to establish quantitatively the volume fractions and chemical compositions of phases after complex heat treatments resulting in a trimodal distribution of gamma' particles. Thermodynamic computations were made using the Ni-database developed by Saunders and Thermo-Calc software systems. The predictions of primary phase constitution were found to be in excellent agreement with the measurements. Carbide and boride phases were also predicted accurately. Partition coefficients for most elements were close to calculated values, but Mo and Ti show more deviation. Some kinetic features of the achievement of equilibrium will be examined.

General Pyrometallurgy - I

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee
Program Organizer: Adrian C. Deneys, Praxair, Inc., Tarrytown, NY 10591 USA

Monday AM — Room: 601
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Adrian C. Deneys, Praxair, Inc., PMT R&D, 777 Old Saw Mill River Rd., Tarrytown, NY 10591 USA; Thomas P. Battle, Dupont, White Pigments & Minl. Prod., 104 Hay Rd., Edge Moor, DE 19809 USA

8:30 AM
Mechanism and Technology of Segregation Roasting of Oxide Nickel Ores with Subsequent Calcine Flotation and Concentrate Leaching: *I. D. Reznik*[1]; A. V. Tarasov[1]; T. A. Kharlakova[1]; T. A. Mayorov[1]; V. I. Goryachkin[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-Ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., Moscow 129515 Russia

1. Oxide nickel ores occur in Russia in the Urals Mountains. As to their composition they are divided into iron-bearing and magnesium-bearing ores. Both types of ore contain about 1% Ni and 0.04-0.09% Co. 2. Three nickel smelters were constructed in the Urals region in the 1930s and they have been in operation until now using the matte smelting technology. Matte is processed to produce converter matte and fire refined nickel, as well as cobalt and nickel salts as by-products. Due to the changes in the prices these smelters are currently operated with low profitability. 3. The authors believe that the most efficient way to improve the economic performance of the smelters is to apply segregation roasting of ores, flotation or magnetic separation of calcine and sulfuric acid pressure leaching of concentrate. As compared with the shaft-furnace smelting, the proposed flow-sheet will reduce the production cost by 22% and even with the lowest nickel price recorded during the recent years, i.e., US $4,180 per 1 tonne, the operation of the smelters will be highly profitable and the payback period for the construction of a new shop will be 2.3 years. 4. Thermodynamic computations and the experimental data indicate that there is no need for moisture in the chain of reactions of calcium chloride decomposition and chlorination of nickel oxide, while the reduction of nickel chlorides requires the presence of hydrogen, which can be produced by hydrolysis of water on solid carbon particles or primary metal grains. 5. The reaction mechanism has been investigated providing additional substantiation for selection of a double-stage segregation roasting flow-sheet: removal of moisture and heating of ore to 950-1000°C during the first stage and chlorination and chloride reduction in the presence of small amounts of water at 800-950°C during the second stage performed in another unit. 6. The table below presents the results of laboratory tests carried out in an intermittent mode with close-circuit flotation and magnetic separation steps. The data presented confirms the high performance values of the process. 7. In the process of oxidizing pressure leaching of concentrates with sulfuric acid with an acid requirement of 55%, at 150°C and an oxygen pressure of 4 atm, about 96-98% Ni and 88-95% Co reported to the solution virtually with complete absence of iron. 8. The overall world resources of nickel in deposits of ores with less than 1% Ni are estimated at 80 million tonnes. For this reason upgrading of low-grade nickel ores using the segregation roasting technology may be of interest for a number of countries. Comparative performance values of flotation

and magnetic separation of calcine obtained from iron- and magnesium-bearing ores (close circuits).

8:55 AM

Optimisation of Pellet Reduction in a Phosphorus Furnace: C. Dresen[1]; *J. H.L. Voncken*[1]; W. Schipper[2]; R. de Ruiter[2]; M. A. Reuter[1]; [1]Delft University of Technology, Appl. Earth Scis., Raw Matls. Tech., Mijnbouwstraat 120, Delft 2628 RX The Netherlands; [2]Thermphos International BV, PO Box 406, Vlissingen 4380 AK The Netherlands

Research is presented with respect to investigations on the reduction process of phosphate pellets. In the reduction process of phosphate ores, the following parameters were investigated: 1. Diffusion ability of the phosphorus oxide and gaseous products from the carbon. 2. Influence of reaction flux on reaction kinetics. 3. Temperature influence. 4. Influence of mineralogy and morphology (different ores). 5. Grain size influence of silica and coke. Experiments were carried out in a vertical tube furnace with 2 ores, 2 types of silica and one type of coke. Experimental conditions simulated furnace conditions. The following conclusions could be made: Reaction rate increases with the addition of SiO_2 to the pellet; Reaction rate increases above 1350°C; Mixing different ore types in the pellets leads to differences in reduction kinetics; Grain sizes of the coke particles influence reduction kinetics. The amount of slag formation is influenced by silica addition, coke sizes, temperature.

9:20 AM

Submerged Lance Injection Dynamics: *Sacha Neven*[1]; Maurits Van Camp[2]; Bart Blanpain[1]; Patrick Wollants[1]; [1]Katholieke Universiteit Leuven, Metaalkunde en Toegepaste Materiaalkunde (MTM), Kasteelpark 44, Heverlee, Brabant 3001 Belgium; [2]Union Minière Research, Kasteelstraat 7, Olen, Antwerpen 2250 Belgium

Submerged lance technologies combine high intensity processing characterized by rapid mixing and reaction rates using a simple reactor design. The Isasmelt technology uses a central submerged lance to inject oxygen-enriched air and fuel into a bath thus creating large gas bubbles and intensive turbulence and splash. The lance is equipped with swirlers to increase gas velocity and heat transfer. This enables the formation of a frozen slag layer on the lance which protects it from the aggressive environment. Water models as well as high temperature reactors were used to validate the Davidson-Schüler model on bubbling frequencies [1]. Resonance effects related to the bubbling frequency have proven to be very relevant to reactor construction and operation. Frequencies and pressure amplitudes were measured in a wide range of gas flow rates: from 10-4 m^3/sec using a water model to more than 30 m^3/sec in an industrial process. Agreement with the model was satisfactory. The experiments however also clearly showed that cross-flow and bubble wake induce slightly higher frequencies. No influence of lance depth and use of swirlers on the bubbling frequency was observed in our experiments with water models. [1] J. F. Davidson and B. O.G. Schüler, "Bubble Formation at an Orifice in an Inviscid Liquid", TRANS. INSTN. CHEM. ENGRS., vol. 38, 1960, pp. 335-342.

9:45 AM

Electric Arc Furnace Dusts Recycling Process in a Cupola to Produce Commercial Zinc Dusts and High Quality Cast Iron: *Grégory Miette*[1]; Xavier Mévisse[1]; André Van Lierde[1]; Herman Dehon[2]; [1]Université Catholique de Louvain, Unité PCIM, Place Sainte-Barbe 2, Louvain-la-Neuve 1348 Belgium; [2]Fonderie Piret S.A., Chaussée de Châtelet 273, Gilly 6060 Belgium

The paper deals with a process that allows the recycling in a cupola of EAF dusts with 18% Zn. It produces dusts with zinc grade higher than 50-55% and commercial cast iron with less than 200 ppm Zn, 50 ppm Pb, 0.15% S. The paper focuses mainly on lab tests made with the aim to develop an effective formulation of self reducing-melting pellets. With such a formulation, agglomerates develop a breaking load higher than 125-150 kg without any thermal treatment. Zinc and lead are volatilized in the cupola with yields higher than 95% at 1200°C, before any melting. Sulphur is transferred into gas and slag. All these conditions allow to lower zinc, lead and sulphur grades below the cast iron specifications. Results of first bench scale tests performed at Fonderie Piret to validate the process will be presented.

10:10 AM Break

10:20 AM

Measuring Total Gas-Liquid Interfacial Area from Submerged Gas Injection into Pyrometallurgical Reactors: *Sarah Jane Buckler*[1]; David E. Langberg[1]; Douglas R. Swinbourne[2]; [1]CSIRO Minerals, Box 312, Clayton, Victoria 3169 Australia; [2]RMIT, Cheml. & Metlgcl. Eng., PO Box 2476V, Melbourne, Victoria 3001 Australia

Many chemical and metallurgical processes use submerged gas injection to increase contact area between gas and liquid phases in order to promote faster overall reaction or refining rates. Estimating total interfacial area from submerged injection into industrial pyrometallurgical vessels from existing knowledge is a problem with two aspects, laboratory measurement and scale-up. The work presented in this paper is part of an ongoing experimental investigation, using chemical measurement methods, to quantify the total gas-liquid interfacial area available in pyrometallurgical vessels. Experiments were performed for different operating conditions in a medium-scale, high temperature (1300-1400°C) metal system, and at both medium and large-scale (1/3-scale industrial vessel) in an aqueous system. The effects of the three operating variables, injected gas flow rate, nozzle diameter and nozzle submergence, are quantified in both systems and implications for scale-up of the high temperature results are discussed in light of the scale-up characteristics in the aqueous system.

10:45 AM

Aluminum Deoxidation Equilibrium in Liquid Fe-36%Ni Alloy at 1773K: *Sang-Beom Lee*[1]; Hae-Geon Lee[1]; Chang-Hee Rhee[1]; Dong-Sik Kim[2]; [1]POSTECH, MSE, San31, Hyoja-Dong, Namgu, Pohang, Kyungbuk 790-784 S. Korea; [2]POSCO, #5 Dongchon-Dong, Nam-Gu, Pohang, Kyungbuk 790-784 S. Korea

The deoxidation equilibrium for aluminum in liquid Fe-36%Ni alloy was carried out by using Cold Crucible under Ar gas atmosphere at 1773K. The interaction parameter between aluminum and oxygen in liquid Fe-36%Ni alloy was determined as -1.42 within the composition range of [%O] < 0.015 and [%Al] < 1. The equilibrium constant for the deoxidation reaction: Al2O3(s) = 2Al + 3O for Fe-36%Ni alloy was obtained to be -13.01 in logarithmic scale.

11:10 AM

Pyrometallurgical Processing of Low-Grade Lead-Zinc-Copper Raw Materials as a Source for an Increase in Production of Non-Ferrous and Precious Metals: *A. D. Besser*[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-Ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., Moscow 129515 Russia

A significant part of natural resources of heavy non-ferrous metals is represented by refractory polymetallic ores containing precious metals. Production of standard-grade monometallic concentrates from such ores results in substantial losses of valuable constituents, especially precious metals, which report to intermediate products, when efforts are taken to improve the grade of main concentrates. A number of concentrators deliberately produce low-grade polymetallic concentrates in order to maintain an adequate recovery of precious metals from ores. The lack of suitable technology for treating sub-grade concentrates and middlings with satisfactory recoveries of zinc, lead, copper and precious metals during the metallurgical stage of processing poses a limitation to the production of these metals despite a significant shortage of supply. The technology concept proposed by the Gintsvetmet Institute is based on an improved process for electrothermal smelting of calcined charge substantially reducing thereby the total expenses (and first of all energy cost) and creates favorable conditions for solution of environmental problems associated with treatment of small volumes of metallurgical off-gas. Elimination of the need for soda in the smelting process permits production of lead bullion and good segregation of matte and slag into which valuable constituents are concentrated (copper in matte and zinc in slag). Since crude lead is the best collector for precious metals, the latter are virtually completely recovered into lead in the process of smelting, and partially into the matte, and further transferred into commercial-grade products. Lower losses of precious metals in slag in case of the new technology are attributed to the fact that slag contacts with matte with low contents of gold, silver and lead, rather than with lead bullion. The new electrothermal technology produces only small amounts of off-gas resulting in a more cost-effective operation of emission control equipment and lower emissions of deleterious pollutants into the environment.

11:35 AM

Dezincing of Iron-Containing Secondary Raw Materials in Frothed Slag Layer in an Electric DC Arc Furnace: *A. V. Tarasov*[1]; *V. A. Bryukvin*[1]; V. M. Paretsky[1]; V. G. Leontyev[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-Ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., Moscow 129515 Russia

The proposed process is based on creating a controlled frothed layer of molten slag, in which fine particles of carbonaceous reductant and raw material to be processed are mixed with emulsified iron. Frothing of the slag layer without external blowing occurs as a result of intensive evolution of gaseous reduction products. The froth reaction process is maintained due to the heat of an electric arc in the slag layer. When the metal bath is heated by the arc to 1400-1500°C, the low temperature of the slag surface of about 1200-1300°C is maintained

due to low thermal conductivity of slag froth preventing heat loss. In order to maintain the required slag froth level and control the process temperature, air tuyeres are installed above the slag bath; blowing air serves also for oxidation of zinc in fumes and for its transportation. High adsorption and thermal insulating properties of slag froth ensure virtually complete elimination of dust and mist entrainment along with low heat losses. The main macrokinetic regularities of the carbon thermal reduction of zinc-containing slag melts have been studied under frothing conditions. The mechanism of formation of gas-slag emulsion has been identified under the conditions of reaction [C]+[O]=CO occurring locally at the slag-metal interface. It has been demonstrated that under the froth reduction conditions the zinc removal rate from the melt increases on the average by 3 to 5 times. Based on the physical and chemical data obtained, basic principles of the dezincing technology have been developed for different intermediate oxide iron-containing products produced in the process of ferrous and non-ferrous metals production. Separation of zinc and iron under reducing conditions is achieved by transfer of reduced zinc into fumes and transfer of reduced iron into metal melt. Zinc fumes are carried over, without external blowing, with reduction products and undergo subsequent oxidation above the slag by air blowing. The residual zinc and iron contents of slag are 0.01-0.05% and up to 3%, respectively. The high intensity of carbothermal reduction in slag froth with good shielding with electric arc makes it possible to ensure a productivity higher by 3 to 5 times than in case of conventional electric-arc smelting with power requirement by 1.5 to 1.8 times lower. The main process parameters: zinc recovery with its initial content of over 1% is 95% with residual zinc concentration in slag of 0.01-0.05%; the degree of iron reduction depending on the technological and economic reasonability as high as 90% or higher with its residual content in slag of up to 3%; the temperature of molten metal is 1300-1650°C depending on the required level of saturation of iron with carbon within a range of 0.05 to 2.5%; the slag layer temperature of 1200-1350°C depending on viscosity for a particular slag composition; the molten slag composition varies depending on the purpose of refining and alloying of molten metal; the requirement of pulverized carbonaceous reductant (coke, coal, etc.) is about 12% of the feed material depending on the zinc content, required degree of reduction and carbon saturation of iron, carbon content of the feed material; it is close to the theoretical reaction requirement for direct reduction with formation of CO and for iron reduction up to 50%; the unit productivity of the process is within 100 to 300 kg/m2 hour; the estimated electric power requirement is 300-400 kWh per 1 t of feed material, which is lower than the common level due to lower heat losses under the slag foam and post-combustion of carbon monoxide above the slag by air blowing.

High Performance Metallic Materials for Cost Sensitive Applications: Overview and Titanium Alloys I

Sponsored by: Structural Materials Division, Structural Materials Committee, Titanium Committee

Program Organizers: Edward Y. Chen, TiTech International, Inc., Pomona, CA 91768 USA; Rod Boyer, Boeing Commercial Airplane Group, Seattle, WA 98124-2207 USA; F. H. (Sam) Froes, University of Idaho, Institute of Materials and Advanced Processes, Moscow, ID 83844-3026 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Monday AM — Room: 213
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Edward Y. Chen, TiTech International, Inc., 4000 W. Valley Blvd., Pomona, CA 91769 USA; F. H. (Sam) Froes, University of Idaho, Inst. of Matls. & Adv. Proc., Mines Bldg., Rm. 321, Moscow, ID 83844 USA

8:30 AM Invited
Metals for Cost-Sensitive Applications–An Overview: *E. Y. Chen*[1]; E. Taleff[2]; R. Boyer[3]; D. Zhang[4]; Lu Li[5]; M. Ward-Close[6]; D. Eliezer[7]; F. H. (Sam) Froes[8]; [1]TiTech International, Inc., 4000 W. Valley Blvd., Pomona, CA 91768 USA; [2]University of Texas, TX Matl. Inst., Col. of Eng., Austin, TX 78712 USA; [3]The Boeing Company, PO Box 3707, MS 6H CJ, Seattle, WA 98124 USA; [4]The University of Waikato, Dept. of Matls. & Proc. Eng., PB 3105, Hamilton New Zealand; [5]National University of Singapore, Dept. of Mechl. Eng., 10 Kent Ridge Crescent, Singapore 119260; [6]DERA, Titanium Alloys Metallics Div., Griffith Bldg. (A7), Rm. 2008, Hampshire GU14 OLX, Farnborough UK; [7]Ben-Gurion University of the Negev, Dept. of Matls. Eng., PO Box 653, Beer-Sheva 84105 Israel; [8]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

This paper will present an overview of the various factors which contribute to the cost of metals in final assembled configurations. This will include costs of extraction, melting and fabrication to mill products, machining, assembly and quality assurance. Examples will be given for various metals and for use in differing industries such as automobiles and aerospace.

9:10 AM Invited
Prospects for Cost Reduction of Titanium via Electrolysis: *Harry Rosenberg*[1]; [1]Amargosa Group, 9371 Almar Pl., Pittsburgh, PA 15237 USA

Fused salt electrolysis is one of the more versatile techniques available for producing titanium. It is quite versatile, not only in control of metal purity, particle size, and distribution, but also in cell configuration and operating mode. Three basic operating techniques are available: electrowinning, electrorefining, and electron mediated reaction. At present, only electrorefining is commercial; although electrowinning titanium was demonstrated on the industrial scale for a time by TIMET some 40 years ago. Current technologies and prospects for further progress are reviewed.

9:40 AM Invited
Titanium: Why is it Expensive and What Can be Done?: *Stephen J. Gerdemann*[1]; [1]Albany, Thermal Treatment Technologies, 1450 Queen Ave. S.W., Albany, OR 97321 USA

Titanium is considerably more expensive than the light metals aluminum and magnesium. This high cost has limited the use of titanium to specialty markets. This paper will examine the history of commercial titanium production and how that history continues to affect the cost today. A brief description modern titanium extractive metallurgy will be given. The costs associated with each step starting with the ore and ending with a titanium part will be identified. The most promising of the proposed new processes will be discussed as well as their chance of success and potential for cost savings. Finally, an estimate for the cost for titanium made by the "perfect process" will be made.

10:10 AM Break

10:30 AM Invited
Implementation of Advanced Metal Technologies to Reduce the Cost of Aerospace Systems: *Ricky L. Martin*[1]; [1]Boeing, Phantom Works, PO Box 516, MC S276-1240, St. Louis, MO 63166-0516 USA

Leveraging the inherent affordability of metallic materials and processes provides excellent opportunities for researchers to minimize costs in existing and advanced aerospace systems. Boeing is participating in a wide range of cost reduction initiatives that are demonstrating and implementing advanced metal product technologies that provide documented affordability benefits over traditional approaches to manufacturing aerospace structures. Although aerospace industry investment in structural metals research and development has been declining recently, participation in company partnerships, industry consortiums, and government-sponsored development initiatives have allowed accelerated transition of metal product technologies into production. Application of new technology to improve existing manufacturing methods, and the introduction of new revolutionary metal processes are both highlighted in this overview.

11:00 AM
Recent Developments in the Manufacturing of Low Cost Titanium Alloys: *Yoji Kosaka*[1]; Stephen P. Fox[1]; [1]TIMET, Henderson Techl. Lab., PO Box 2128, Henderson, NV 89009 USA

About a half dozen of so-called "Low Cost Titanium Alloys" were developed almost a decade ago being targeted to emerging markets such as automotive and armor applications. Initial efforts were focused on the formulation cost of alloys by increasing revert content or designing new alloys that require less expensive raw materials. None of these alloys could achieve a solid presence in these markets until very recently. The environment for low cost titanium has been changing favorably to low cost titanium alloys. EB cold hearth melting is recognized as a versatile tool to provide low cost alloys as well as premium grade titanium alloys. The reduction of conversion cost is critical to an overall cost reduction. A combined effort to introduce new titanium alloys by vertical integrated teams with users and suppliers has started to open up the opportunity for low cost titanium alloys in cost sensitive applications. This paper will review and discuss the recent developments in the manufacturing of low cost titanium alloys.

11:20 AM
Direct Production of Ti-6-4 Alloy Plate from Single Melt, Electron Beam Cold Hearth Melted Slab Ingot: *J. R. Wood*[1]; Steven H. Reichman[2]; [1]Allegheny Technologies, Inc., Allvac, 2020 Ashcraft Ave., Monroe, NC 28111-5030 USA; [2]Allegheny Technologies, Inc., Allegheny Ludlum, 500 Green St., Washington, PA 15301 USA

As part of the Metals Affordability Initiative Consortium Program, this study was initiated to produce aerospace quality, standard grade Ti-6Al-4V alloy plate directly from single melt, electron beam cold hearth melted (EBCHM) rectangular slab ingots. It is to be demonstrated that the EBCHM process is capable of producing chemical homogeneity, macro and microstructure and mechanical properties comparable to plate made from conventional double melt vacuum arc refined (VAR) round ingots. It is also to be demonstrated that large EBCHM slab ingots can be rolled directly to plate on conventional equipment with significant cost savings compared to the processing route for conventional round VAR ingots. The lower cost is derived from the use of less costly raw materials, lower melt costs, streamlined processing and improved yields. The progress to date in this multi-year program is reported and discussed.

11:40 AM
Electron-Beam Cold-Hearth Single-Melt Ti-6Al-4V Plate for Armor Applications: *John C. Fanning*[1]; [1]TIMET, Techl. Lab., PO Box 2128, Henderson, NV 89015 USA

Although titanium provides high ballistic mass efficiency and other advantages, widespread use of titanium alloys in ground combat systems has been limited by the relatively high raw material cost. Ti-6Al-4V (and variations thereof) is the preferred alloy for both structural and applique armor applications. Up to the present virtually all of the production of this product has been per aerospace specifications, which typically require multiple vacuum arc re-melting (VAR). During the past several years cold hearth melting has been used in place of one of the vacuum arc melts because of the ability of the process to remove inclusions. Significant cost savings may be achieved by the use of a single melt process. Although single melt heats of commercially pure titanium are now being routinely produced, there is limited production experience for single melt of alloy grades such as Ti-6Al-4V. A recent study by the US Army provided an initial assessment of the product of an electron beam, cold hearth, single melt of Ti-6Al-4V plate for application to Army ground vehicles. This paper provides the results of ballistic and mechanical property testing of additional samples of this type of product.

Hume-Rothery Award Symposium: CALPHAD and Alloy Thermodynamics: Phase Equilibrium Modeling

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Robert D. Shull, NIST, Magnetic Materials, Boyds, MD 20841-9015 USA

Monday AM Room: 204
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, C.&M.S. Direct. (L-353), PO Box 808, Livermore, CA 94551 USA; Suzana Gomez Fries, ACCESS e.V., RWTH-Aachen, Intzestrasse 5, Aachen 52072 Germany

8:30 AM Introductory Remarks

8:35 AM Keynote
Hume-Rothery and CALPHAD Thermodynamics: *Larry Kaufman*[1]; [1]140 Clark Rd., Brookline, MA 02445-5848 USA

H-R's "Atomic Theory for Students of Metallurgy" was a text for undergrads in the '50s. In '66 I was in Geneva for the Battelle's Conference on "Phase Stability in Metals and Alloys" with H-R, Brewer and Kubachewski. H-R invited me to review the subject for vol.14 (1969) of PMS that he edited. We corresponded 30 months till his death in '68. Kuba's Brunel and Sheffield conferences in '71 and Munster '72 spawned the CALPHAD method, Journal and conferences which have grown till today! The Symposium on Computational thermodynamics and Materials Design at the 2001 TMS Annual Meeting illustrated many cases where CALPHAD Thermdynamics affording the broadest description of stable, unstable and metastable phases over wide ranges of composition, pressure and temperature were used to predict useful materials and processes. In this presentation the connection will be made between H-R's work, the CALPHAD method and "Alloy Thermodynamics".

9:30 AM Invited
Phase Diagram Calculations: Contributions of Ab Initio and Cluster Variation Methods: *Catherine Colinet*[1]; [1]LTPCM, ENSEEG, BP 75, Saint Martin d'Heres, Cedex F-38402 France

The treatment of the short-range order and long-range order in solid solutions, and of order-disorder transformations need the use of the cluster variation method (CVM) or of Monte Carlo simulations. These methods need as input the interaction parameters between the atoms, called the cluster interactions. These parameters may be fitted using thermodynamic and phase diagram data, such as enthalpies of formation or order-disorder temperatures. However the procedure is ambiguous since the nature of the most important cluster interactions (pairs, triplets, quadruplets, etc), their range, and their composition dependence are a prior unknown. Several attempts have been made to deduce the cluster interactions from first principle calculations. Two approaches are available for such calculations. In the first one, the energy of the completely disordered solid solution is calculated by the coherent-potential approximation, the effective cluster interactions are calculated by the embedded-cluster method or by the generalized perturbation method. The second approach that is now extensively used consists in performing ab initio total energy calculations of perfectly ordered compounds in order to estimate the values of the cluster interactions. A review of the phase diagram calculations which have been performed using the methods described above will be presented.

10:00 AM Invited
Effect of the Interaction between the Chemical and the Magnetic Ordering on the Phase Equilibria of Iron Base Alloys: *Ikuo Ohnuma*[1]; Ryosuke Kainuma[1]; Kiyohito Ishida[1]; [1]Tohoku University, Dept. of Matls. Sci., Aoba-yama 02, Senday 980-8579 Japan

It is well known that the magnetic properties such as the Curie temperature T_c and the mean magnetic moment β of ordered compounds have different values from those of the disordered solutions. For instance, both T_c and β of the Ni_3Pt ($L1_2$) and NiPt ($L1_0$) and T_c of the CoPt ($L1_0$) and $CoPt_3$ ($L1_2$) ordered compounds are strongly depressed due to the ordering compared with those of the metastable disordered Ni-Pt and Co-Pt alloys. On the other hand, the γ'-$FeNi_3$ ($L1_2$) and the α'-FeCo (B2) ordered compounds have higher T_c and β values comparing with the disordered solution phases, γ (A1) and α (A2), respectively. In consequence, the stability of the ordered phase is depressed or enhanced due to the interaction between the chemical and magnetic ordering caused by the decrease or increase of T_c and β values. The purpose of this study is to investigate the effect of the interaction between the chemical and the magnetic ordering on the phase equilibria in the Fe-X (X=Al, Co, Ni, Rh, Si) binary systems.

10:30 AM Break

10:45 AM Invited
Phase Diagram Calculation: Present State and Applications: *Y. Austin Chang*[1]; W. A. Oates[2]; Shuanglin Chen[3]; F. Zhang[3]; S. Daniel[3]; Xinyan Yan[3]; F.-Y. Xie[1]; R. Schmid-Fetzer[4]; [1]University of Wisconsin, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA; [2]University of Salford, Sci. Rsrch. Inst. M5 4WT UK; [3]CompuTherm, LLC, 437 S. Yellowstone Dr., Madison, WI 53719 USA; [4]University of Clausthal, Inst. of Metall., D-38678, Clausthal-Zellerfeld Germany

In this presentation, we will (1) assess the current state of the phenomenological-model approach in calculating phase diagrams stressing the great stride made in recent decades and the need for continuing improvement in the future and (2) present experimental and computational results obtained on the paths of solidification and microsegregation in multicomponent commercial aluminum alloys, i.e. more than 10 components. The calculated results were obtained by integrating phase diagram calculation (PanEngine) with a modified Scheil model incorporating solid state diffusion, dendritic coarsening and undercooling effects. The experimental results were obtained by directional solidification.

11:15 AM Invited
Chemical Criteria in the Assessment of Alloy Constitutional Properties: *Riccardo Ferro*[1]; Gabriele Cacciamani[1]; [1]Universita' di Genova, Dip. di Chimica e Chimica Industriale, via Dodecaneso 31, Genova I-16146 Italy

It is well known that an accurate prediction of phase equilibria in multi-component systems requires a sound thermodynamic character-

ization of the involved binary and ternary sub-systems. In turn this needs good sets of data precisely determined and critically assessed. Comments and examples about a number of semi-empirical chemical criteria which may be useful in the selection and application of the assessment procedures are reported. Regularities which may be found in considering the general alloying behavior of the metals pertaining to certain groups or blocks of the periodic table (such as, for instance, the alkali metals, the lanthanides, the actinides, etc.) are underlined. The results obtained in the author's laboratory in the study and evaluation of properties of lanthanide alloys (phase equilibria, thermodynamics, crystallochemistry, etc.) are presented and discussed.

11:45 AM Invited
Developments in Thermodynamic and Phase Equilibrium Modelling at the National Physical Laboratory: *John A. Gisby*[1]; Alan Dinsdale[1]; Hugh Davies[1]; Susan Martin[1]; Jim Robinson[1]; [1]National Physical Laboratory, NPL Matls. Ctr., Queens Rd., Teddington, Middlesex TW11 0LW UK

Almost since its foundation in 1900, the National Physical Laboratory in the UK has been involved in the study of the thermodynamic data and phase diagrams. Initially this tended to focus on the accurate measurement of the constitution of binary and ternary alloy systems. From the 1950's onwards due to the pioneering work of Kubaschewski this led to the study of thermodynamic properties through the development and use of key experimental equipment, the critical assessment of thermodynamic properties and their use in the calculation of the change in thermodynamic properties associated with chemical reactions. The natural direction of Kubaschewski's work led to the development at NPL of MTDATA, comprising software and databases utilising the growing availability and power of computers. MTDATA has long been one of the world's foremost thermochemical modelling packages and it has proved itself over many years of use in solving practical problems. It is a powerful and user-friendly software and data package for the prediction of phase equilibria in systems containing large numbers of chemical elements. It has been designed around the high reliability of the NPL Numerical Optimization Software Library. MTDATA is licensed to industrial, government and academic organisations worldwide and training courses are held regularly at NPL and in the US. A range of databases covering a very wide variety of system types is available for use with MTDATA reflecting numerous potential applications in the fields of metallurgy, chemistry, materials science and geochemistry. In this paper NPL's contribution to the critical assessment of thermodynamic data and the calculation of phase equilibria will be reviewed. Particular emphasis will be given to the SGTE database for elements, the development of MTDATA and some of the areas in which MTDATA has been used successfully for the benefit of industry.

International Symposium on Science and Technology of Interfaces in Honor of Dr. Bhakta Rath: Nanostructures and Materials

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Physical Metallurgy Committee, Superconducting Materials Committee, Jt. Mechanical Behavior of Materials, Titanium Committee
Program Organizers: Sreeramamurthy Ankem, University of Maryland, Department of Material & Nuclear Engineering, College Park, MD 20742-2115 USA; I. Ovidko, Russian Academy of Sciences, Institute of Problems of Mechanical Engineering, Laboratory for Theory of Defects in Materials, St. Petersburg 199178 Russia; Chandra Pande, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5000 USA; S. Ranganathan, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India

Monday AM — Room: 617
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Hubert I. Aaronson, Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA; Chandra Shekhar Pande, Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6325, Washington, DC 20375 USA

8:30 AM Opening Remarks

8:40 AM Invited
Grain Rotation and Nanostructure Evolution: New Insights from Atomistic Simulations: *S. G. Srinivasan*[1]; J. Cahn[2]; [1]Los Alamos National Laboratory, T-11 Grp., Theoretl. Div., MS B262, Los Alamos, NM 87545 USA; [2]NIST, Matls. Sci. & Eng., 100 Bureau Dr., MS 8555, Gaithersburg, MD 20899-8555 USA

Bhakta Rath has made seminal experimental contributions that has led to the theoretical understanding of grain boundary (GB) migration [Rath and Hu, Trans. AIME, Vol.245, pp.1577 (1968)]. We revisit this fundamental problem of GB motion and the concomitant grain growth (GG) on the nanometer scale using molecular dynamics simulations in 3-dimensions. We found frequent grain rotation with some surprising and counterintuitive results. For example, bicrystal systems of a circular cylindrical grain, embedded in another with a variety of initial misorientations, can rotate continuously from small angles to large angles, even where their GB specific surface energy increases, as they shrink. Suppression of the grain rotation by using rounded lath shaped cylindrical grains, with straight faces and curved ends, enable the grains to shrink considerably faster than the embedded circular-cylinders with the same initial misorientation. When such grains rotate, the velocity of their grain boundaries is not proportional to curvature. In an array of congruent hexagonal columnar grains, which should have been stable, grain rotation triggers unexpected GG. For small misorientations, many of these surprises can be understood in terms of motion of GB dislocations, but the behavior continues smoothly to higher angles. The implication of these findings for nanostructure stability and GG will be discussed.

9:05 AM Invited
Interfaces and Surfaces in Nanostructured Materials: *Miguel Jose Yacaman*[1]; [1]University of Texas-Austin, Dept. of Cheml. Eng., Austin, TX 78712-1062 USA

The study of metal nanoparticles and their assembling is becoming very important in many fields such as: nanoelectronics, photonics, sensing devices, drug delivery and so on. In these systems the surface atoms are a large proportion of the total number of atoms that produce unique properties. In the present work we discussed the structural changes on nanoparticles as a function of the size, two main cases are discussed: bare particles and particles capped with an organic molecule. It is shown that significant changes on the structure are observed. The coalescence behavior and their self-assembly is also discussed.

9:30 AM Invited
Solid State Alloying of Nanostructured Fe-Zn Binary System: Fei Zhou[1]; *Ye Tsang Chou*[1]; Enrique J. Lavernia[1]; [1]University of California, Dept. of Chem. & Biochem. Eng. & Matl. Sci., 916 Engineering Tower, Irvine, CA 92697-2575 USA

Iron-Zinc binary alloys in solid solution were processed by mechanical alloying (MA) of high-purity metal powders. The MA-induced microstructural evolution was characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC). The nanostructured alloys are maintained in single-phase, body-centered cubic lattice with the zinc content up to 65 at.%. Further addition of zinc, e.g. at 95 at.%, would shift the cubic lattice to hexagonal close packing. An important feature of this binary system is that the solid solutions formed by the MA obey the Vegard's law over the entire range of mixing. The effect of the addition of zinc on ferromagnetic properties was examined and compared with the data obtained from the same alloy system prepared by vapor quenching. Mechanisms underlining the solid state alloying in the Fe-Zn system are also discussed.

9:55 AM Invited
Chemical Reactions at the Gas-Solid Interfaces: Effects on the Electrical Conductivity of Semiconducting Nanomaterials: *Marie-Isabelle Baraton*[1]; Lhadi Merhari[2]; [1]University Limoges, SPCTS UMR 6638 CNRS, 123 Ave. Albert Thomas, Limoges 87060 France; [2]Ceramec, 64 Ave. de la Libération, Limoges France

Improvement of nanosized powder-based sensors requires a careful study of the surface reactions at the origin of the gas detection mechanism. This is a necessary step toward a rigorous industrial fabrication protocol. In this work, chemical reactions occurring at the interface between gases and semiconducting nanopowders are investigated by Fourier transform infrared (FTIR) spectrometry. These surface reactions are compared for several semiconducting materials (tin, indium and tungsten oxides) at different temperatures. Simultaneously, the effects of these surface reactions on the electrical conductivity of the metal oxides are analyzed and discussed in relation with their sensing properties toward oxygen and carbon monoxide. This work is funded by the European Commission under the "Information Society Technologies" programme (contract number IST-12615).

10:20 AM Invited
Interfaces in Zr Based Nanocrystals and Combustion Synthesized Ti Aluminide: *Gautam Kumar Dey*[1]; [1]Bhabha Atomic Research Center, Matls. Sci. Div., Trombay, Mumbai, Maharashtra 400 085 India

The nature of the interfaces associated with intermetallic phases produced in a very fine morphology by crystallization of Zr based bulk metallic glasses and combustion synthesis of TiAl has been examined by high resolution electron microscopy. The nanocrystals produced by the crystallization of $Zr_{52}Ti_6Al_{10}Cu_{18}Ni_{14}$ bulk glass have been found to lie in the size range of 15 to 50 nm and comprised phases isostructural with Zr_2Ni and Zr_2Cu. Various types of interfaces in these phases such as twin boundaries, stacking faults and antiphase domain boundaries have been studied. It has been possible to generate a very fine lamellar microstructure by combustion synthesis of TiAl with widths of the α_2 and γ lamellae in the range of 5 to 20 nm. The interface between the α_2 and γ phases has been investigated in detail in this microstructure. The interfaces between contiguous lamellae of the γ phase have also been examined.

10:45 AM Invited
Effects of Interfaces on the Properties of Nanostructured Magnetic and Optical Films: *Gan-Moog Chow*[1]; [1]National University of Singapore, Dept. of Matls. Sci., Kent Ridge 119260 Singapore

Nanostructured polycrystalline films with grain size less than 100 nm have a significant amount of grain boundaries and interfaces. Control of the grain size,chemistry and structure of the interfaces is essential to control material properties. We have fabricated nanostructured films (NixCo100-x, CoCrPt/Ti and Au/BaTiO3) for magnetic and optical applications. These films were studied using x-ray scattering, anomalous x-ray scattering, extended x-ray absorption fine structure, scanning electron microscopy, transmission electron microscopy, atomic force microscopy, x-ray photoelectron spectroscopy, vibrating sample magnetometry, alternating gradient magnetometry and UV-VIS-IR spectroscopy. In this talk, examples will be given to demonstrate the effects of interphase interface on the structural, magnetic and optical properties of these films. The experimental results of the long-range order and local atomic environment (in plane and out-of plane) and the interphase interfaces of these films will be discussed and correlated with that of thermodynamics modeling.

Magnesium Technology 2002: Magnesium Primary Production

Sponsored by: Light Metals Division, Magnesium Committee, International Magnesium Association
Program Organizers: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA; Gerald S. Cole, Ford Motor Company, Ford Research Laboratories, Dearborn, MI 48121 USA; Rod Esdale; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA; Ramaswami Neelameggham, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Eric A. Nyberg, Pacific Northwest National Laboratory, Materials Processing Group, Richland, WA 99352 USA; Mihriban O. Pekguleryuz, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H9R 1G5 Canada; Bob R. Powell, General Motor Corporation, NAO Research and Development Center, Warren, MI 48090-9055 USA; Allen Schultz, Hatch, Mississauga, Ontario L5K 2R7 Canada

Monday AM — Room: 606
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Howard I. Kaplan, Magnesium Corporation of America, 238 N. 2200 W., Salt Lake City, UT 84116 USA; Neale Neelameggham, Magnesium Corporation of America, 238 N. 2200 W., Salt Lake City, UT 84116 USA

8:30 AM
History Primary Magnesium since WWII: *Byron B. Clow*[1]; [1]International Magnesium Association, 6731 Whittier Ave., McLean, VA 22101 USA

Primary magnesium production just prior to WWII was around 32,000 tonnes with most of it coming from Germany and England with smaller amounts from France and the USA. Peak production during the war was 232,000 tonnes with most of it coming from the 15 plants that had been built in the United States. By 1946 worldwide demand had fallen back to pre-war levels and did not return to war levels until 1980. The paper will identify the various plants built around the world since WWII and the impact of exports from Russia and China beginning in the early 90's.

9:00 AM
Magnesium Market Model Simulation: The Impact of Increased Automotive Interest in Magneisum: *Randall Joseph Urbance*[1]; Richard Roth[1]; Joel Clark[1]; [1]Massachusetts Institute of Technology, Matl. Sys. Lab., 238 Main St., Ste. 319, Cambridge, MA 02142 USA

Increasing energy and environmental concerns are forcing automakers to consider light-weight alternatives to traditional component designs. Magnesium, the lightest engineering metal, has been cited as a potential weight-saving material for automobiles, but has been slowly adopted due to high prices and limited engineering experience. Despite the technical and financial challenges of magnesium design, recent trends in the auto industry show renewed interest in the material. The small size of the magnesium supply base could limit the use of magnesium in the automotive arena if increasing demand leads to material shortages and price volatility. A system dynamics model of the magnesium market was created to investigate the potential problems caused by a small, rapidly expanding supply base coupled with increasing, but very price-sensitive material demand. The model, which simulates supply, demand and price interactions, was used to investigate market stability strategies that will benefit all players.

9:25 AM
An Improved Process for Production of Magnesium: Hans R. Eklund[2]; Per B. Engseth[3]; *Thorvald Mellerud*[1]; Birger Langseth[4]; Oddmund Wallevik[4]; [1]Hydro Magnesium, Ave. Marcel Thiry 83, B-1200 Brussels Belgium; [2]Hydro Magnesium, N-0246 Oslo Norway; [3]Hydro Magnesium, N-3901 Porsgrunn Norway; [4]Norsk Hydro ASA, Rsrch. Ctr., N-3901 Porsgrunn Norway

Norsk Hydro started production of primary Magnesium in Porsgrunn, Norway 1951,using the IG Farben chlorination process for production of anhydrous MgCl2 as feedstock to the electrolysis. Norsk Hydro has continued to develop the IG Farben chlorination process, and an improved version is still in operation at the Porsgrunn plant. In the late 1960's Norsk Hydro started to develop a process for production of anhydrous MgCl2 based on dehydration of MgCl2 brines. The first generation of the process was started in Porsgrunn 1978, when a 15 kt Mg/yr demonstration unit was put in operation. A modified and developed version of the technology was installed in the Norsk Hydro green-field facility at Becancour (Quebec) Canada. The plant produce MgCl2 brine by dissolving of magnesite in hydrochloric acid and started operation in 1989. The new dehydration technology first of all offers an environmentally sound solution with minimal emissions of chlorine and chlorinated hydrocarbons, improved productivity and improved quality of the anhydrous MgCl2 feed, giving significantly improved performance of the electrolysis. The development of the Norsk Hydro dehydration process has continued, and today an improved second generation process is ready for implementation in brown-field expansions or green-field projects. Several elements of the improved process are already installed in the Becancour plant. Compared to previous generation, the improved dehydration process has further improved productivity and reduced energy consumption. A presentation is made of the Norsk Hydro improved dehydration process. Its basic features and performance are described, supporting that large scale operation based on this technology is very competitive compared to other existing and recently presented front end technologies.

9:50 AM
Process Optimization of Magnesium Thermal Reduction: *Henry Hu*[1]; Alfred Yu[1]; *Naiyi Li*[2]; [1]University of Windsor, Mech., Auto. & Matls. Eng., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Ford Motor Company, Mfg. Sys. Dept., Scientific Rsrch. Lab., Rm. 3011, MD 3135, 2101 Village Rd., Dearborn, MI 48124 USA

A numerical model of heat and mass transfer in the vertical retort has been developed to simulate and optimize the process of magnesium thermal reduction. The simulations were run to determine the effect of varying parameters, such as the diameter of retort, thickness of the compound, and slot angle, on the magnesium reduction time. The model predicted the temperature distributions, the heating curves, the recovery ratio of magnesium, and the total process time. The predictions were used to optimize the magnesium reduction process, the dimensions of retort, the shapes of materials, and reaction cycle. The

result shows that the optimizations will significantly improve the Pidgeon magnesium reduction process in vertical retort, increase production capacity and recovery, decrease reduction period, and save energy consumption.

10:15 AM Break

10:35 AM
Solid-Oxide-Oxygen-Ion-Conducting Membrane (SOM) Technology for Primary Production of Magnesium from its Oxides: *Uday B. Pal*[1]; Timothy Keenan[1]; Ajay Krishnan[1]; Christopher Manning[1]; [1]Boston University, Mfg. Eng., 15 St. Mary's St., Boston, MA 02446 USA

The work is aimed at producing Mg metal from oxide ores with oxygen gas as a by-product of the process. Oxygen-ion-conducting yttria-stabilized-zirconia (YSZ) crucibles are used to hold the melt (flux) containing dissolved magnesium oxide. A suitable anode is placed on the outside of the YSZ tube. An inert metal rod is placed in the melt, contained in the YSZ crucible, to serve as the cathode. An electric potential is applied between the cathode and the anode to dissociate the magnesium oxide and produce Mg metal at the cathode, while removing oxygen ions through the zirconia membrane and oxidizing them at the anode; a reducing gas is passed over the anode to getter the oxygen. To minimize the ohmic resistance across the YSZ membrane and obtain high current density, these experiments are conducted at temperatures between 1300 to 1400C. The results obtained are used to model the process.

11:00 AM
Physical and Chemical Investigations of Salt Melt Compositions of KCl-NaCl-MgCl-NaBr and KCl-NaCl-MgCl-CaCl Systems: *Ivan Andreevich Barannyk*[1]; [1]State Titanium Research and Design Institute, Magnesium Production, 180 Lenin Av., Zaporizhzhya 69035 Ukraine

In magnesium production by electrolysis and in magnesium and Mg alloys refining multicomponent salt melts, consisting of four and more alkaline halogens, alkali earth metals and magnesium are used. Integrated study of physical and chemical properties of complex halogen systems is of great interest for a justified choice of optimum salt melt compositions for magnesium production. The most important salt melt physical and chemical properties are as follows: fusibility, density in operating temperature limits(650-750°C), viscosity and conductivity. The results of density and fusibility measurements of some salt compositions of KCl-NaCl-MgCl2-NaBr and KCl-NaCl-MgCl2CaCl2 systems are given in the report. Influence of NaBr, CaCl2 and their mixture compositions on multicomponent halogen systems of salts fusibility and density was determined by experimental data processing. Investigated melts density dependence on the temperature in the limits of (700-800°C) was fixed. Using another physical and chemical salt properties data, taken from the literature, an attempt to substantiate the choice of optimum salt mixtures composition for Mg and Mg alloys melting and refining was made.

11:25 AM
Investigation of Granulation of Potassium Clorides and Cent-Electrolyte: *Andrey Vladimirovich Tarasov*[1]; [1]Gintsvetmet Institute, Metall., 13, Ac. Koroleva str., Moscow 129515 Russia

Potassium chloride is one of the most common mineral fertilizers transported to all continents. Its unstable crystals are attrited to dust and cake together during transportation. Granules of potassium chloride must have a diameter of at least 2mm and withstand forces of up to 2kg. Such properties make it possible to spray them rather uniformly within a band up to 10-12m wide. The research conducted with respect to granulation of fused potassium chloride has indicated that conventional methods of granulation of molten potassium chloride are ineffective and do not provide the desired results, primarily due to the physical properties of potassium chloride. Granules produced by filtration of melt through a sieve do not have the required strength and collapse under a force of 0.7kg. The most common method for potassium chloride granulation is pressing in a roll press with subsequent grinding of the pressed layer and strengthening of the granules produced. The yield of final granules is below 30%. Sorting of final granules of carnallite, potassium chloride by flotation permits separation of a fraction of not more than 1.5mm and the pressed granules produced have sharp-angled irregular form. Granulation tests were carried out on a bench-scale laboratory unit using flat water-cooled rolls. One of the rolls had a matrix of granules. Special material was selected for the matrix roll. The granules produced had a regular semispherical form. Rolled granules had a strength of 2.2-2.5kg. At the same time, tests were carried out to study the effect of addition of alkali metals on the strength of granules. Addition of 2-3% of such additives improved the strength of granules up to 4kg. An engineering design has been developed for a semi-commercial plant for potassium chloride granulation in a roll-type unit with a productivity of 2t/hr. Technical parameters of the plant: Fuel requirement, equivalent fuel per 1t of granules 11kg/t; Thermal efficiency of the melting unit 51-54%; Amount of recycled material 5-7%; Dust content of gas at the outlet 10mg/m3. This unit has not been built due to the lack of financing. Granulation of Cent-Electrolyte: Cent-electrolyte formed in the process of electrolytic magnesium production contains 61-63% of potassium chloride and is sent to further processing. Most commonly, cent-electrolyte is cast into blocks of 250-300kg and after cooling during 24 hours it is ground. This method results in formation of large amounts of fines and is associated with substantial emissions of dust, gas and heat into the ambient environment at workplaces. In order to correct those drawbacks, a method of pneumatic granulation of cent-electrolyte was tested and proven on a laboratory-scale unit with a productivity of 20kg of melt per hour. As a result, the appropriate aerodynamic conditions and the composition of spraying gas were determined. An engineering design has been developed for a shop for cent-electrolyte granulation at a plant with a productivity of 25,000 tpy of magnesium. This shop consists of an electrically heated holding furnace, a granulation chamber, a system for metered feeding of molten cent-electrolyte and a dry dust collection system. The main part of the spraying device is a nozzle of a special design. Granules produced are of irregular sharp-angled form, with an average size of 0.9-1.0mm, with a content of about 5% of size fractions of less than 0.9mm and over 1.0mm. Such a shop is currently in operation at the magnesium plant of the Dead Sea Works in Israel.

Processing and Properties of Lightweight Cellular Metals and Structures
The MPMD Third Global Symposium
Structural and Multifunctional Applications - Session I

Sponsored by: Materials Processing & Manufacturing Division, Jt. Computational Materials Science & Engineering, Powder Materials Committee, Jt. Processing Modeling Analysis & Control Committee, Surface Engineering Committee, Shaping and Forming Committee, Solidification Committee

Program Organizers: Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; T. Dennis Claar, Fraunhofer USA, Newark, DE 19716 USA; T. H. Sanders, Georgia Institute of Technology, Department of Materials Science and Engineering, Atlanta, GA 30332 USA

Monday AM	Room: 205
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Amit Ghosh, University of Michigan, Dept. of Matls. Sci. & Eng., 2066 H.H. Dow Bldg., 2300 Hayward, Ann Arbor, MI 48109-2136 USA; William Clyne, Cambridge University, Matls. Sci. Dept., Pembroke St., Cambridge CB2 3QZ UK

8:30 AM Opening Remarks

8:35 AM
Multifunctionality of Closed-Cell Aluminum Foams: *T. Dennis Claar*[1]; Virgil Irick[1]; Jim Adkins[1]; Kenneth Kremer[1]; [1]Fraunhofer USA, Ctr. for Mfg. & Adv. Matls., 501 Wyoming Rd., Newark, DE 19716 USA

Closed-cell aluminum foams are an emerging class of ultra-lightweight materials that can be tailored to exhibit a broad range of properties. The powder metallurgy-based foaming process is capable of fabricating foams with porosity levels of 40 to 90 vol%. Various geometries are possible, including 3-D molded shapes, foam-filled tubes, and aluminum foam sandwich (AFS) panels. A significant characteristic of aluminum foams is their ability to absorb energy during compressive deformation. Energy is absorbed during crushing at a nearly constant "plateau" stress to approximately 60% strain, before the stress increases significantly. Aluminum foam inserts inside metal tubes increased the specific energy absorption by 35-40% compared to the hollow tubes. Foam inserts also increased the bending stiffness of hollow tubes by 40-45%. Foamed aluminum has demonstrated superior fire resistance compared to solid aluminum plates. During exposure to a gas flame at 850-870°C, a 6061 Al plate experienced melt-through, while an Al foam plate was unaffected. Aluminum foams also improve

the performance of lightweight armor, providing improved projectile defeat, lower dynamic deflection, and enhanced ballistic shock attenuation. AFS panels have very high specific stiffness, allowing significant weight reduction. Applications that take advantage of these multifunctional features of Al foams will be presented.

9:00 AM
Perspectives on High-Volume Automotive Applications of Lightweight Cellular Metals and Structures: *Ray Jahn*[1]; Andrew M. Sherman[1]; [1]Ford Motor Company, FRL Mfg. Sys., MD 3135, 2101 Village Rd., Dearborn, MI 48124 USA

Laminated and cellular materials have been proposed as a way of achieving higher weight reductions in automotive structures than available with high strength steels, aluminum, magnesium and polymer composites. However, it remains to be seen whether the applications in production vehicles predicted for these materials will develop. One reason is the high cost of making the materials and fabricating them into components. Also, although it is easy to show that such materials offer superior properties, e.g. specific stiffness, it is much more difficult to translate this into lighter weight in fully engineered structures. Among the difficulties are that in applications having complex loading the directional properties of these materials may be a disadvantage, the load carrying characteristics of the available joining methods do not permit full utilization of the material's capabilities, or other aspects of the specific application compromise the materials in some way. In this paper we present a cost/benefit analysis of the use of lightweight materials for automotive weight reduction, which helps establish cost and weight target ranges for the use of cellular and laminated metals.

9:25 AM Moved to Tuesday PM
Tailored Component Fabrication using Stabilized Aluminum Foam: *Greg Mills*

9:25 AM
The Stiffness and Weldability of an Ultra-Light Steel Sandwich Sheet Material with a Fibrous Metal Core: *A. E. Markaki*[1]; S. A. Westgate[2]; T. W. Clyne[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK; [2]TWI, Granta Park, Great Abington, Cambridge CB1 6AL UK

A sandwich material, based on a pair of thin stainless steel faces plates separated by a core incorporating stainless steel fibres, has recently been developed. This material has the potential to exhibit an attractive combination of properties, while retaining formability and general handling characteristics similar to those of conventional steel sheet. Three different core structures have been investigated: (a) transversely-aligned fibres bonded to the face plates by adhesive, (b) a sintered fibre mat bonded to the face plates by adhesive and (c) a sintered fibre mat brazed to the face plates. The beam stiffnesses of these three structures have been measured and compared with theoretical predictions. Through-thickness electrical conductances were measured and compared with predictions from simple analytical models. These data were correlated with the results of spot welding tests. It is shown that structure (c) is readily weldable, whereas there are certain difficulties with the adhesively-bonded sheet.

9:50 AM Break

10:10 AM
Constructed Cellular Metals: *David J. Sypeck*[1]; [1]University of Virginia, Matls. Sci. Eng., 116 Engineer's Way, Charlottesville, VA 22911 USA

Cellular solids are made up from interconnected networks of solid struts or plates that form the edges and faces of cells. To date, the main obstacle to obtaining superior properties with materials of this type has involved gaining good control over the distribution of material at the cell level. For many years, it has been known that cellular designs having the highest weight specific stiffness and strength are truss-like when the solid volume fraction is small. Imitating decades of work by structural engineers, cellular solids researchers have now begun to investigate open periodic cell lattices (trusses) with controlled distributions of material at the cell level. These are miniaturized versions of the large engineering type structures (e.g. skyscrapers, bridges, transmission towers, etc.) that have already been made and tested for many years. In this work, efficient truss architectures are identified, methods for affordably making them from metals are presented and properties are assessed.

10:35 AM
Cellular Structure by Direct Metal Deposition: *Jyoti Mazumder*[1]; [1]University of Michigan, Ctr. for Laser-Aided Intelligent Mfg., 2350 Hayward St., 2041 G.G. Brown Bldg., Ann Arbor, MI 48109-2125 USA

The Direct Metal Deposition (DMD) process is creating considerable contemporary interest due to its capability to deliver "Art to Part". DMD can reduce the lead time for a concept to product by eliminating several intermediate steps. The most attractive feature of the process is that not only can it produce functional parts but it can also be interfaced with the Homogenization Design Method (HMD), Heterogeneous Solid Model (HSM) and Computer Aided Design (HCAD) software to produce "Designed Material" with desired properties generally not observed in nature. Closed Loop DMD is a synthesis of multiple technologies including lasers, sensors, Computer Numerical Controlled (CNC) work handling stage, CAD/CAM software and cladding metallurgy. This paper describes the methodology used for designed cellular structures for closed loop DMD.

11:00 AM
Reinforced Alulight for Structural Use: *Frantisek Simaník*[1]; Walter Rajner[2]; Rainhard Laag[3]; [1]Institute of Materials and Machine Mechanics SAS, Ra ianska 75, Bratislava SK-831 02 Slovakia; [2]Alulight Int. GmbH, Ranshofen Austria; [3]Alulight Deutschland GmbH, Wasseralfingen Germany

PM foams can be prepared with pore size gradient and with preferred orientation of pores. There are almost no constraints in the complexity of outer shape and geometry. PM foams are always covered by a dense skin which improves the mechanical properties (e.g. bending stiffness, etc.). However the natural skin of foams has variable thickness and sometimes contains small holes or cracks. Reinforcing of tensile loaded surface skin with metallic or ceramic wires or fibers woven into grids with various mesh size can improve mechanical properties. The possibility to reinforce the foamed part selectively and anisotropically according to the expected load can produce significant improvement in the stiffness, strength, plasticity, energy absorption capacity and damage tolerance of foamed part with only slight weight increase (ca. 20-30%). The reinforcements prevent liquid foam from collapsing on cooling and thus they have also an additional stabilizing effect. Moreover the reinforcements increase the thickness of surface skin, simplify joining of foamed parts (welding is possible), and enable limited shaping after the foaming process.

Processing of Refractory Metals and Alloys - I

Sponsored by: Structural Materials Division, Refractory Metals Committee
Program Organizers: Clyde Briant, Brown University, Division of Engineering, Providence, RI 02912 USA; Pete Lipetzky, Pittsburgh Materials Technology, Inc., Large, PA 15025 USA

Monday AM — Room: 209
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Clyde Briant, Brown University, 182 Hope St., Providence, RI 02912 USA; Pete Lipetzky, Pittsburgh Materials Technology, 811 Rte 51 S., Bldg. #9, Large, PA 15025 USA

8:30 AM Opening Comments

8:35 AM
Refractory Alloy Development: *Pete Lipetzky*[1]; [1]Pittsburgh Materials Technology, Inc., 1801 Rte. 51, Bldg. 9, Jefferson Hills, PA 15025 USA

Over the last 40 years numerous refractory metals and other advanced alloy systems have been investigated for applications ranging from power generation to rocket nozzles to chemical processing equipment. The materials were primarily in the families of Mo-W, Ta-Nb, V-, Zr-, and Re-based. Alloy additions such as silicon were used to perform seminal work in intermetallics and in-situ composites. The fabrication of these alloys were by powder metallurgical methods and AC vacuum arc remelting, often followed by extrusion, forging, swaging and rolling. Microstructural effects on mechanical, electrical, corrosive and oxidation properties were investigated. Furthermore, ultra-high vacuum (UHV) heat treating and mechanical testing were utilized. These UHV facilities were used to demonstrate that creep properties are sensitivity to vacuum level in some alloys. Some successful results will be discussed.

8:55 AM

Processing of Refractory Metals using Radiant Arc-Lamp Heating: *E. K. Ohriner*[1]; C. A. Blue[1]; T. J. Huxford[1]; [1]Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6083 USA

Radiant arc-lamp heating offers the potential for direct production of melted sheet products and foil. A focussed 300 kW arc lamp provides incident radiant heat at a flux of up to 3 kW/cm2. Among the refractory metals the melting of tungsten, rhenium, and tantalum have been demonstrated. The process offers the advantages of: 1) high efficiency of conversion of electrical energy to absorbed incident energy, 2) the ability to transmit radiant energy through a quartz window enabling environmental isolation of processed material, and 3) processing of large areas of material at rates of the order of 10 cm2/s. Current research efforts include the direct production of rhenium metal sheet from sintered powder compacts and include computer modeling of melting and solidification sheet materials.

9:15 AM

The Effects of Initial Grain Size on Pure Tantalum Processed by ECAE: *S. N. Mathaudhu*[1]; K. T. Hartwig[1]; [1]Texas A&M University, Mech. Eng., 319 Eng./Physics Bldg., Spence St., College Station, TX 77843-3123 USA

Bulk VAR tantalum with three different initial grain sizes (as-cast: >5mm, large: 200-800 microns, medium: 20-80 microns)was deformed to plastic strains of 1.16, 2.32, and 4.64 by equal channel angular extrusion (ECAE). Results of hardness measurements and metallography are reported. Recrystallized grain size and morphology were measured for each case. Results indicate that the recrystallization temperature was mostly a function of plastic strain, and was independent of initial grain size. It was also observed that the microstructural morphology was similar after two ECAE passes, and that fine(<20 micron), equiaxed grains were developed after four passes for all three starting grain sizes.

9:35 AM

Tensile Properties and Microstructure of the Electroformed Rhenium: *Alexander Smirnov*[1]; [1]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

Rhenium has high a melting point, high density, ductility and good strength at elevated temperature. A number of manufacturing methods are being applied to rhenium to produce near-net shape products. Electroforming process in molten salts is used by Engelhard-CLAL to produce high-density, porosity-free rhenium coatings for different applications. Electroformed rhenium must meet various requirements and the most impotent of them are tensile properties. It is shown that tensile properties of electroformed rhenium depend on the microstructure of the material. The influence of various conditions of electrolyses, annealing parameters and cold deformation on microstructure of the electroformed rhenium and tensile properties were studied. The stress-strain behavior and the microstructure of electroformed rhenium will be discussed.

9:55 AM Break

10:15 AM

Development, Characterization, and Li Compatibilty of an Electrically Insulating CaO Coating on V-4Cr-4Ti Alloy: *Mehmet Uz*[1]; K. Natesan[2]; [1]Lafayette College, Cheml. Eng. Dept., 341 AHE, Easton, PA 18042 USA; [2]Argonne National Laboratory, Energy Tech. Div., 212/G180, 9700 S. Cass Ave., Argonne, IL 60439 USA

As part of the US Department of Energy's Fusion Reactor Program, an electrically insulating and liquid Li compatible CaO coating is being developed on V-4Cr-4Ti alloy. This alloy is the primary candidate for various fusion reactor structural applications including the first wall structure/blanket. The first wall will be in contact with liquid Li coolant on the outside, and electrical conductivity across the wall will lead to magnetohydrodynamic, pressure losses during flow of the conducting liquid in the magnetic field. Hence, it is essential that a coating be applied to the alloy, and that it be electrically insulating and compatible with a liquid Li environment at temperatures of 500-700°C. This paper deals with the development and characterization of a CaO coating on V-4Cr-4Ti alloy specimens by a vapor transport process with double Ca-deposition/oxidation steps. The specimen surfaces were analyzed after each step during the process, and also after exposure to liquid Li together with their cross sections. The analysis and characterization were done using one or more techniques, including scanning electron microscopy, energy dispersive X-ray analysis, X-ray diffraction, and measurement of electrical resistivity external to the Li. Results are presented with emphasis on the details of the experimental procedure used and the microstructural characteristics of the coated samples. This work has been supported by the US Department of Energy, Office of Fusion Science, under Contract W-31-109-Eng-38.

10:35 AM

Effects of Processing and Prolonged High Temperature Exposure: *Mehmet Uz*[1]; Robert H. Titran[2]; [1]Lafayette College, Cheml. Eng. Dept., 341 AHE, Easton, PA 18042 USA; [2]NASA-GRC, 27146 Winwood Way, Olmsted Falls, OH 44138 USA

A systematic study was carried out to evaluate the effects of thermomechanical processing and prolonged high-temperature exposure on the microstructure of Nb-1 wt.% Zr-0.1 wt.% C alloy. All samples were from sheet stocks fabricated by cold rolling of separate billets that were single-, double-, or triple-extruded at 1900 K with a 4:1 ratio. Samples were annealed for 1 h at 1755 K and/or 2 h at 1475 K and subsequently exposed to 1350 K for up to 34,500 h with and without applied load. A combination of various analytical and metallographic techniques was used to characterize the microstructures of sheet samples, as well as the residue extracted from them after each step. The results show that the carbides, which are relatively coarse orthorhombic Nb2C in the as-rolled samples, transform upon subsequent heat treatment to rather fine cubic carbide of Nb and Zr with various Zr/Nb ratios. Also, the relative amount of cubic carbides and the Zr/Nb ratio increase as the number of extrusions increase before cold rolling. Furthermore, prolonged exposure to 1350 K gives rise to complete transformation of Nb2C to (Zr,Nb)-C regardless of the processing history. These and other observations are presented with the emphasis on the effects of processing on microstructure.

10:55 AM

Undercooling Experiments on the Zr-Ta System from Drop-Tube Processing: *Bernard Vinet*[1]; [1]CEA, DTEN/SMP/LESA, 17 rue des Martyrs, Grenoble 38054 France

Deep undercooling on refractory metals and alloys can be obtained during a few seconds in the 48-m high drop tube facility built in Grenoble. A first class of materials has been considered by alloying hcp Re with bcc W, Ta, Nb and Mo metals. With the support of first-principle calculations, this study was focused on nucleation and phase phenomena involving tcp phases [see PRL 83(1999)1621]. In the course of this new program, undercooling experiments are realized on a new class of materials where the above bcc metals are alloyed with elements from the group IVB. Characteristics results obtained on Zr-Ta alloys will be described including microstructures, exploitation of recalescence profiles and microhardness tests. The undercoolability of pure Zr as well as the formation of the metastable omega phase in solidified samples will be also discussed.

11:15 AM

Anomalous High Content of Strongly Bound Water in Plastically Bent Tungsten Single Crystals: *Oleksandr Illich Dekhtyar*[1]; Victor Ivanovich Patoka[1]; Mykola Yakimovich Shevchenko[1]; [1]Kurdyumov Institute of Metal Physics of National Academy of Science of Ukraine, Div. of Strength & Plasticity of Nonhomogeneous Alloys, 36 Vernadsky Blvd., Kiev, Kiev Region 03142 Ukraine

The chemical composition of tungsten single-crystalline plates with the surface orientation {110} by 1.2-1.5 mm thickness plastically bent in vacuum around axis <112> with the help of electron beam heating at 1170-1270 K temperatures up to 20-25% strain have been studied by secondary ion emission. There was found the anomalous high content of water in the bulk of tungsten. This content is essentially higher than one of usual insert impurities (C, O, N) and other elements and compounds. There was studied the water amount after high temperature annealings. It is found that high water amount conserves up to 2700 K. Such strongly bound state of water molecules is caused by existing of the high excess density of one sign dislocations which conserves up to near tungsten melting point. The water molecules have to be finding way into the dislocation cores as ionized molecules.

Second International Symposium on Ultrafine Grained Materials: Processing and Structure - I

Sponsored by: Materials Processing & Manufacturing Division, Shaping and Forming Committee
Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; Terence G. Langdon, University of Southern California, Department of Mechanical Engineering, Los Angeles, CA 90089-1453 USA; Terry C. Lowe, Technanogy, Newport Beach, CA 92627 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; Michael Jeremi Saran, Case Western Reserve University, Cleveland, OH 44106 USA; S. Lee Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Monday AM Room: 210
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Yuntian T. Zhu, Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; Terry C. Lowe, Metallicum, LLC, 1207 Callejon Arias, Santa Fe, NM 87501 USA

8:30 AM Keynote
Continuum Mechanics Approach in Severe Plastic Deformation: *Vladimir M. Segal*[1]; [1]Honeywell Electronic Materials, STAR Ctr., R&D, 15128 E. Euclid Ave., Veradale, WA 99216-1895 USA

The presentation considers some aspects of severe deformation processing: 1. Early development in 1970's-1980's. 2. Deformation mode and loading history. 3. Simple shear versus pure shear. 4. Structure evolution during simple shear. 5. Processes of simple shear. 6. ECAE: slip line solutions and deformation mode. 7. ECAE: technological opportunities and challenges. 8. Conclusion

8:55 AM Invited
Powder Consolidation by Equal Channel Angular Extrusion: *Ricardo B. Schwarz*[1]; Jose I. Archuleta[1]; Jenya Macheret[2]; Tom Lillo[2]; Gary Korth[2]; [1]Los Alamos National Laboratory, Struct./Prop. Relations Grp., MS G755, Los Alamos, NM 87545 USA; [2]DOE-ID, 850 Energy Dr., MS 1225, Idaho Falls, ID 83401 USA

Equal-channel angular extrusion (ECAE), an innovative deformation process capable of introducing elevated levels of shear while preserving the cross section of the product, has been applied to the consolidation of metal powders. A mixture of spherical copper and silver particles was loaded into square-cross-section copper cans, which were then extruded at 300°C. After three successive ECAE passes (equivalent extrusion ratio of 32), the powder becomes a filamentary Cu and Ag composite. The composites are fully dense and crack free. Their compressive yield strength is 330 MPa and their ductility exceeds 20%. The Young's moduli along and perpendicular to the filaments are 106 and 96 GPa, which equal the values calculated using appropriate averages of the elastic constants of pure copper and silver. The mechanical tests and elastic constant data show that ECAE is a viable method for the consolidation of powders.

9:15 AM
Grain Refinement in Beryllium by Equal Channel Angular Extrusion: *David J. Alexander*[1]; Robert D. Field[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-6, MS G770, Los Alamos, NM 87545 USA

Fine-grained beryllium alloyed with 6 wt% copper is the material of choice for fabrication of targets for the National Ignition Facility. Beryllium is a difficult material to process, because of its limited ductility and its environmental and safety concerns. Equal channel angular extrusion (ECAE) of Be has been performed at elevated temperatures to determine if ECAE is a suitable processing method for production of this material. Be samples 5 mm in diameter and 30 mm long were canned in mild steel to create a billet 10 mm square by 60 mm in length. These billets were extruded through 90° tooling. Several different processing routes were also investigated. Significant grain refinement occurred, even after only a single extrusion pass, with additional refinement for subsequent passes. The microstructures and textures resulting from the different processing routes will be compared, and the suitability of ECAE for production of fine-grained Be and Be alloys will be considered.

9:30 AM
Grain Refinement of Medium Carbon Steel with Controlled Thermo-Mechanical Deformation: Jongmin Park[1]; Dae Hyun Song[1]; Duklak Lee[2]; Wung Yong Choo[2]; Ik-min Park[1]; *Kyung-mox Cho*[1]; [1]Pusan National University, Matls. Sci. & Eng., San 30 Jangjeondong, Kumjungku, Pusan 609-735 Korea; [2]Posco, Ltd., Pohang, Kyungbuk 790-785 Korea

Microstructure of 0.45%C medium carbon steel was tried to control through thermo-mechanical treatment. The purpose is to obtain fine grained microstructure of the cold heading quality steel. With refined microstructure, the heat-treatment time for spheroidization could be reduced greatly. For the thermo-mechanical treatment, deformation temperature, deformation rate and cooling rate were controlled. The steel was deformed in austenite region (above A3) as well as austenite+ferrite region (between A3 and A1) and the resulting microstructure was compared. The grain size of thermo-mechanically treated 0.45%C medium carbon steel up to sub-micro-meter (less than 1 micro-meter) was obtained. The spheroidization heat-treatment time with the refined microstructure was reduced to about 3-5 hours comparing the conventional steel needed 15 hours for spheroidization.

9:45 AM
Heterogeneous Microstructural Evolution and Reactions during Repeated Intense Deformation: *Rainer J. Hebert*[1]; John H. Perepezko[1]; [1]University of Wisconsin, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA

With the recent improvements in severe plastic deformation and cold rolling, deformation processing has become an alternative to rapid-solidification processing for the synthesis of bulk metastable structures. The systematic study of the deformation patterns that develop during repeated rolling of multilayer based on a stacked elemental crystalline array including the analysis of interface areas and the evolution of interface roughening, offers an effective approach to the examination of the mechanism involved in deformation alloying, nanostructure synthesis and amorphization. A key-factor for deformation-induced phase transformations is the occurrence of atomic scale mixing effects at interfaces. The results from cold rolling experiments on Al-Pt and Al-Hf alloys demonstrate that the overall multilayer refinement represents an essential component for the prediction of amorphization by cold rolling in addition to the heat of mixing and a deformation enhanced mixing kinetics at individual interfaces. In light of cyclic crystalline-to amorphous reactions reported for ball-milled Co-Ti and Al-Zr alloys, the effect of cold rolling on the devitrification behavior of Al-based glasses has also been addressed in a systematic study. For marginal glass formers, such as melt-spun Al92Sm8, cold rolling induced a complete crystallization without annealing while for melt-spun Al85Ni10Ce5, a crystalline phase has not been observed under the same deformation conditions. The structural changes are observed to develop heterogeneously throughout the sample over several deformation cycles due to the distribution of microstructural size scales. These developments highlight the importance of the localized microstructural response to the overall sample deformation. The support of the ARO (DAAD19-01-1-0486) is gratefully acknowledged.

10:00 AM
On the Development of Microstructure in a Metal Matrix Composite using Nano-Materials: Vladimir Popov[1]; *Donald Lesuer*[2]; Iouri A. Kotov[3]; Viktor V. Ivanov[3]; Andrej A. Aksenov[1]; Igor Khodos[4]; Galina Klimenko[4]; Oleg Smirnov[1]; Ajdar Murzakaev[3]; Sergei Zayats[3]; [1]Moscow State Institute of Steel and Alloys, Leninsky Prospect 4, Moscow 117936 Russia; [2]Lawrence Livermore National Laboratory, L-342, Livermore, CA 94551 USA; [3]Institute of Electrophysics of Ural Branch Academy of Science, Ekaterinburg Russia; [4]Institute of Microelectronics Technology and High Purity Materials, Chernogolovka Russia

Metal matrix composites (MMCs) containing matrices with nanometer grain sizes provide opportunities for improved properties. In this paper, we report on MMCs prepared from pure aluminum nano-powders (particle sizes of 50-200 nm) with SiC reinforcement (particle size 3-10 μm). The volume fraction of SiC reinforcement was varied between 20 and 40%. The pure aluminum nano-powders were prepared using an exploding wire technique. The particles of the MMC were mixed and then dynamically compacted to high density using an explosive compaction method. The resulting microstructures were evaluated using optical and transmission electron microscopy. Microstructural analysis of the resulting MMC showed a highly uniform distribution of SiC particles with no visible defects or pores and the absence of deleterious phases (such as Al4C3) at the interfaces between the aluminum nano-grains and the SiC particles. The evolution of microstruc-

ture during dynamic compaction has also been studied using TEM and found to progress in three stages. The results of these microstructural evolution studies will be discussed as well as the resulting properties of the MMC. Work performed under the auspices of the US Department of Energy by the Lawrence Livermore National Laboratory under contract No. W-74055-ENG-48.

10:15 AM Break

10:25 AM Invited

Fine Scale Microstructures Produced by Sliding Loads: *Darcy A. Hughes*[1]; [1]Sandia National Laboratories, Matls. & Eng. Sci. Ctr., POB 969, MS9405, Livermore, CA 94551-0969 USA

Frictional contact between metals under large sliding loads is a method for introducing extreme strains, thereby producing nano-scale deformation microstructures within the near surface regions. This microstructure consists of layers of finely spaced deformation-induced boundaries. The boundary spacing coarsens with increasing depth from the surface in correspondence to the decrease in deformation. This graded structure ranges in size from 10nm at the surface to a few μms at a depth of 200μm. The microstructural characteristics are quantitatively analyzed and compared to those developed by rolling. The structures in both cases evolve similarly with strain over this large range of scales. The similarity indicates that the deformation mechanisms controlling microstructural evolution are the same from 10 to 10,000nm. This work was supported by the Office of Basic Energy Sciences of the US DOE, Division of Materials Sciences under contract no. DE-AC04-94AL85000.

10:45 AM Invited

Grain Refining Mechanisms of Ti during Equal Channel Angular Pressing: *Yong-Seog Kim*[1]; Jong Youl Kim[2]; Inyoung Kim[2]; Dong Hyuk Shin[2]; [1]Hong-Ik University, Matls. Sci. & Eng., 72-1 Sangsu Dong, Mapo Ku, Seoul 121-791 S. Korea; [2]Hanyang University, Metlgcl. & Matls. Sci., Sa 1 Dong Ansan Si, Kyunggi-Do 425-791 S. Korea

Microstructural development of titanium during equal-channel angular pressing was investigated to understand the mechanisms of grain refinement and strain accommodation. The samples were processed via three different pressing routes: A, B and C. TEM micrographs of the sample after the first pass showed that {1011} deformation twinning plays a primary role in accommodating the plastic deformation imposed by the pressing. During subsequent passes, plastic deformation was accommodated mainly by dislocation slip and its slip system was dependent on the pressing routes: a slip on prismatic planes when processed via route B, a slip on basal planes and a+c slip when processed via route A, and a slip on prismatic planes and deformation twinning when processed via route C. These differences between the routes were discussed based on the preferred orientation and the CRSS of each slip mode.

11:05 AM

Grain Refinement and Texture Development in Asymmetrically Rolled Aluminum Alloy Sheets: *Dong Nyung Lee*[1]; Su-Hyeon Kim[1]; Jong Kook Lee[1]; [1]Seoul National University, Matls. Sci. & Eng., Shinrim-dong, Seoul 151-742 Korea

Asymmetric rolling, in which the ratio of upper to lower roll diameter is different, has been used to introduce an intense plastic shear strain for the purpose of grain refinement and in turn strengthening and of obtaining higher plastic strain ratios of aluminum sheets due to shear deformation textures through the sheet thickness. Commercial purity aluminum sheets were asymmetrically cold rolled without lubrication and the strengths and microstructures of the sheets were investigated by tensile testing, transmission electron microscopy (TEM), and electron back-scatter diffraction (EBSD) analysis. After several passes of asymmetric rolling, a fine grain structure with large misorientations developed in the sheets and their strengths increased. Unidirectional rolling in which the rolling direction does not change between passes gave rise to more uniformly refined structures than alternating direction rolling in which the rolling direction is rotated through 180º about the rolling direction between passes.

11:20 AM

Effect of Vanadium Addition on Dynamic Gamma to Alpha Transformation during Hot Deformation of Low Carbon Steels: *Hu-Chul Lee*[1]; Jae-Young Cho[1]; Joo-Hee Kang[1]; Dong-Woo Suh[2]; [1]Seoul National University, Matls. Sci. & Eng., Shinrim-dong San 56-1, Kwanak-ku, Seoul 151-742 Korea; [2]National Institute of Materials Science, Matl. Creation Rsrch. Grp., 1-2-1 Sengen, Tsukuba 305-0047 Japan

The effect of 0.06-0.2% vanadium and 0.01wt% nitrogen addition on the dynamic gamma to alpha transformation in a low carbon steel (Fe-0.15wt%C-1.2wt%Mn-0.3wt%Si-0.02wt%S) was investigated. The 0.06-0.2wt%V steel and base steel were heated to 1080°C and 960°C, respectively, held for 5 minutes to produce an austenite grain size of about 100 microns, and deformed for 25%-70% in a single pass at 700°C, just above Ar3 temperature, using laboratory deformation simulator. The dilatation curve was measured for 10 minutes after deformation and normalized by the diameter of specimen just after deformation. The volume fraction of dynamically transformed ferrite was evaluated from dilation curves. The volume fractions of dynamically transformed ferrite were 21% and 52% in base steel and 35 and 54% in 0.2%V steel, respectively, after deformation of 25% and 40%. When the deformation was higher,the effect of vanadium addition was diminished. After deformation of 40% or higher, the base and 0.2wt%V steel show dual grain structure of fine ferrite grain (about 3-4 microns) which are formed and grown during deformation and coarse ferrites (about 7-8 microns) which are formed and grown during isothermal holding at 700°C. These results show that vanadium addition to low carbon steel enhances not only the static but also the dynamic transformation resulting in the refinement of ferrite grain size. The results of TEM analysis on the role of vanadium carbo-nitrides in dynamic transformation is also discussed.

11:35 AM

Nano-Nano Composites of Silicon Nitride and Silicon Carbide: *Julin Wan*[1]; Matt J. Gasch[1]; Amiya K. Mukherjee[1]; [1]University of California, Dept. of Chem. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

This is an effort of bringing the grain size scale of silicon nitride and silicon carbide composites into the truly nanometric range, i.e., with the grain size of both silicon nitride and silicon carbide within 100nm limit. Amorphous Si-C-N bulk materials were first synthesized by a processing route based on pyrolysis of polymer precursor. By controlling the crystallization heat-treatment at relatively low temperature levels, and without oxide sintering additives, silicon nitride/silicon carbide composites with grain size of 30-50nm can be achieved. A second processing method of processing was high pressure sintering. Oxide additives were incorporated into precursor pyrolysis-derived powders, and sintering was conducted at 1400-1600°C under a pressure of 1-2GPa. Although in this case liquid phase sintering was involved to some extent, the combination of high-pressure, low temperature, short duration and the nature of starting powder effectively hindered grain growth during sintering, leading to nano-nano microstructures. This research is supported by the US Office of Naval Research (Grant No. N00014-00-1-0186).

11:50 AM Invited

Grain Refinement and Phase Transformations in Al and Fe Based Alloys during Severe Plastic Deformation: *Sergey Dobatkin*[1]; [1]Moscow State Steel and Alloys Institute, Dept. of Plastic Deformation of Special Alloys, Leninsky prospekt, 4, Moscow 119991 Russia

The present paper shows the possibility of additional refinement of the structure up to nanoscale size in Al and Fe based alloys during severe plastic deformation (SPD) by torsion under high pressure (HPT) due to phase transformations. 1. Al-Cu-Mg alloy. Severe deformation of oversaturated solid solution of Al-Cu-Mg-alloy after quenching allows to overcome the nanoscale of grain size (less then 100 nm) due to strain aging. 2. Al-Mg-Sc alloys. During hot SPD by HPT of Al-Mg-Sc alloy 01570 at T=400°C the UFG structure with grain size of about 150 nm is formed at the expense of decomposition of a solid solution. 3. Low-carbon steels. SPD of the initially quenched 0,09CMnSi and 0,1CMnVTi steels leads to a more dispersed nanoscale structure than that of initially hot rolled steels. The possibility of precipitation and dissolution of carbides are discussed. 4. Austenitic stainless steels. The martensitic transformations in austenitic steels during severe cold deformation by HPT promote the additional refinement of structure.

Third International Sulfide Smelting Symposium - "Sulfide Smelting '02": From Fundamentals to New Projects

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee, Copper, Nickel, Cobalt Committee, Lead and Zinc Committee, Non-Ferrous Metals Committee
Program Organizers: Robert L. Stephens, TeckCominco Metals, Ltd., Trail, British Columbia V1R 4L8 Canada; Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112 USA

Monday AM Room: 607
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Robert L. Stephens, TeckCominco Metals Ltd., PO Box 1000, Trail, British Columbia V1R 4L8 Canada; H. Y. Sohn, University of Utah, Dept. of Metallurgical Eng., Salt Lake City, UT 84112 USA

8:30 AM Plenary
Continuous Coppper Converting–A Perspective and View of the Future: *David B. George*[1]; [1]Kennecott Utah Copper Corporation, 8315 W. 3595 S., PO Box 6001, Magna, UT 84044-6001 USA

In spite of the many well known problems inherent in Peirce-Smith converting, it has remained the dominant copper converting process for nearly 100 years. Continuous copper converting and direct concentrate to blister smelting have been commercially available for over 20 years and now produce roughly 15% of the world's primary copper. The fully commercialized continuous converting processes, the Mitsubishi MI Process and Kennecott-Outokumpu Flash Converting, and several emerging converting concepts are reviewed with view to their potential, limitations, and environmental performance. Some myths regarding these technologies and Peirce-Smith converting are also challenged.

9:05 AM Plenary
Basic Principles of Sulfide Smelting and Converting with Oxygen-Rich Gas: Kimio Itagaki[1]; *Hong Yong Sohn*[2]; Manuel Pérez-Tello[3]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., Katahira 2-1-1, Aoba-Ku 980-8577, Sendai Japan; [2]University of Utah, Dept. of Metallurgical Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA; [3]University of Sonora, Dept. of Cheml. Eng. & Metall., Hermosillo, Sonora 83000 Mexico

Thermodynamics and rate process fundamentals are two of the important principles involved in nonferrous production processes. Process configuration establishes whether thermodynamics or rate process behavior is most relevant in a particular application. In this paper, thermodynamic theory and rate process fundamentals are used to analyze the main characteristics of smelting and converting processes. The effect of increased oxygen potential is examined with emphasis, considering the increased use of highly oxygen-enriched gas in modern smelting processes. The phase equilibrium and the distribution of minor elements between the copper or nickel matte and the FeOx-SiO2 or FeOx-CaO slag are reviewed. The use of oxygen-rich gas has no serious effect on the recovery of valuable copper and silver in the matte phase, while the recovery of nickel and cobalt is adversely affected. The oxygen content in the gas also strongly affect the distribution behaviors of arsenic, antimony and bismuth. The rate processes occurring in the shaft of flash smelting and converting furnaces are analyzed by a three-dimensional computational fluid-dynamics model. The computer model is used to simulate the main features of an industrial flash converting operation. The role of basic principles in the engineering analysis of high-intensity matte smelting processes is discussed.

9:40 AM Cancelled
Considerations on Slag Chemistry of the Noranda Process Reactor: *M. Zamalloa*

9:40 AM
Kinetics of Sulfide Smelting in Mitsubishi Process: *Zenjiro Asaki*[1]; [1]Mitsubishi Materials Corporation, Central Rsrch. Inst., 1-297, Kitabukuro-cho, Saitama 330-8508 Japan

The copper concentrate particles dissolve quickly into the molten matte in the Smelting furnace of Mitsubishi Process, which indicates that the sulfide smelting is essentially gas/liquid reaction. In this paper, theoretical calculation has been made on the rate of oxidation of FeS component in the matte to form FeO by rising gas bubbles using the penetration theory proposed by Higbie. The results showed that all the oxygen in the bubble of 1.2 cm in diameter, which is the maximum stable bubble diameter, is consumed in 3 ms. This time required is sufficiently short, and it results in the actual operation of smelting furnace of 100% oxygen-efficiency. This also indicates that most smelting reactions are completed just under the lance except slag formation because the dissolution rate of silica particles is rather low.

10:05 AM Invited
The Ignition of Sulphide Flotation Concentrates in Flash Smelting: *Frank R.A. Jorgensen*[1]; [1]CSIRO Minerals, PO Box 312, Bayview Ave., Clayton, Victoria 3169 Australia

Flash smelting is used to smelt a large proportion of the world's copper and nickel and some lead/zinc concentrates. Understanding the factors controlling the ignition of these concentrates is an important consideration in designing burners and maximizing the throughput of the flash smelting process. The literature on the subject is briefly reviewed and techniques for measurement of ignition temperature are discussed. The mineralogy of the concentrates and the role of thermodynamics and phase diagrams in interpreting and predicting ignition behaviour are emphasized and applied to interpreting results obtained on a nickel concentrate.

10:30 AM Break

10:45 AM
Gresik Copper Smelter and Refinery–Current Operation and Expansion Plan: *Moto Goto*[1]; [1]PT Smelting Co., Desa Roomo, Kecamatan Manyar, PO Box 555, Gresik, Jawa Timur 61151 Indonesia

PT Smelting, located in Gresik, East Java, is the first copper smelter and refinery in Indonesia. Since the first charging concentrate in December 1998, many equipment troubles have been systematically eliminated, and the present operation is smooth and has accomplished the design capacity of 200,000 tonnes per year of LME, grade-A copper cathode. This paper describes troubles and countermeasures, which were experienced until now.

11:10 AM
Budel Zink Sets a New Standard for NO_x Reduction in a Sulphuric Acid Plant: J. van Driel[1]; B. Giesen[1]; *A. Berryman*[2]; S. Sampat[2]; S. Enevoldsen[3]; H. Jensen-Holm[3]; [1]Budel Zink B. V., Hoofdstraat 1, 6024 AA Budel-Dorplein The Netherlands; [2]Kvaerner Chemetics, A Div. of Kvaerner Canada, Inc., 1818 Cornwall Ave., Vancouver, BC V6J IC7 Canada; [3]Haldor Topsøe A/S, Nymollevej 55, DK-2800 Lyngby Denmark

NO_x is a growing concern for acid plant operators due to recent legislation reducing the allowable stack limits, as well as continually more stringent requirements for product acid quality. NO_x also causes a major problem during maintenance as NO_x rich acid residues fume following exposure to moisture in the air. Until recently, the solution for NO_x related issues was to treat the symptom, rather than the cause. NO_x in product acid was treated using chemicals and the NO_x in the plant stack was generally left untreated. As part of an environmentally driven change in concentrates, Budel Zink installed the first gas phase NO_x destruction process to be incorporated into a commercial scale acid plant. Nitrogen oxides contained in the process gas are reduced selectively with ammonia over a catalyst to form nitrogen and water. As a result, the NO_x reduction is in agreement with the permit requirements.

11:35 AM
An Overview on Operation of Jinlong's Copper Flash Smelting Since Start-Up: Bao Xian Cheng[1]; Song Xiu Ming[2]; *Jun Zhou*[1]; [1]Jinlong Copper Co., Ltd., Prod. Dept., Jinshan Rd., Tongling, Anhui 244021 China; [2]Jinlong Copper Co., Ltd., Tech. Dept., Jinshan Rd., Tongling, Anhui 244021 China

Jinlong's copper flash smelting process was started up in April, 1997. In the early days of production lots of equipment and process problems were encountered. But through continual improvements in equipment and operation, designed productivity was reached two years later, and smelting operation has been greatly upgraded since then. After more than 4 years of production, we have made great achievements, such as high flash furnace availability, high matte grate operation, optimization in concentrate burner's structure and operation, stable operation of converter and anode furnace, and etc. Smelter emission control has reached nation's standard since commissioning. This paper intends to outline Jinlong's operational achievements made after commissioning, analyze problems ever encountered and their remedies. Operational experience is also summarized.

Advances in Metallic Glasses: Glass Formation, Consolidation and Microstructure

Sponsored by: Electronic, Magnetic & Photonic Materials Division,
Program Organizers: K. F. Kelton, Washington University, Department of Physics, St. Louis, MO 63130 USA; A. L. Greer, University of Cambridge, Department of Materials Science & Metallurgy, Cambridge CB2 3QZ UK; Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA

Monday PM Room: 212
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: En Ma, Johns Hopkins University, Dept. of Matls. Sci. & Eng., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218-2689 USA; Krishna Rajan, Rensselaer Polytechnic Institute, Dept. of Matl. Sci. & Eng., Troy, NY 12180-3590 USA

2:00 PM
Structure-Forming Principles for Amorphous Metals: *Kevin L. Kendig*[1]; Daniel B. Miracle[1]; Oleg N. Senkov[2]; W. Sanders[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., AFRL/MLLMD, Dayton, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

After extensive experimental and computational research over the past several decades, there is still a rather poor understanding regarding the structure of metallic glasses. The clearest picture exists for metal-metalloid glasses, yet this still provides only an incomplete understanding. The chemical and topological aspects of these systems differ, sometimes strongly, from many other well-established metallic glass systems, so that the fundamental structure-forming principles which operate in metallic systems are still not established. The purpose of this research is to propose specific ordering principles, which may govern the structural formation of metallic glasses. Earlier concepts of high atomic packing efficiency will be extended and applied to topological systems, which represent most metallic glass alloys. The concept of high packing efficiency has specific implications for both short- and medium-range atomic configurations, which will be presented. Possible atomic configurations will be explored, and consistency with experimental observations will be provided and discussed.

2:20 PM
Atomistic Mechanism of Bulk Metallic Glass Formation: *Takeshi Egami*[1]; Hao Chen[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Eng., LRSM, 3231 Walnut St., Philadelphia, PA 19104-6272 USA

Microscopic mechanism of bulk glass formation is discussed from a very general point of view, focusing on the size factor and interatomic potential. A key for the bulk glass formation is the "strong" glass behavior, as defined by Angel, that keeps the diffusivity of the melt low well above the glass transition temperature. A concept of distributed local glass transition is introduced to explain the strong glass behavior. In a multi-component system each element has a different local topological instability condition that defines the glass transition. This allows calculation of the local glass transition temperature for each component of the system. Widely distributed local glass transition temperatures result in the strong glass behavior. Molecular dynamics simulations to demonstrate this effect will be presented.

2:40 PM
Mechanical Modeling of the Structure of Amorphous Al: *Daniel B. Miracle*[1]; Kevin L. Kendig[1]; Oleg N. Senkov[2]; Robert Smith[3]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [3]Southern Ohio Council of Higher Education, 3171 Research Blvd., Ste. 141, Dayton, OH 45420 USA

A mechanical modeling approach has been applied to explore the structure of amorphous Al alloys. Polyethylene spheres were used to represent atoms within the structure. The number and relative sizes of the spheres were selected to represent typical amorphous Al alloy glass compositions. Arrays of 1000 atoms were used in the models. The spheres were loaded in an elastic container to minimize the influence of the free surface, and to allow introduction of arbitrary shear deformations to randomize the structure. Measurements of free volume for monosized spheres agree very well with accepted values from the literature, providing confidence in the methodology used. The coordinates of each atom in the "amorphous Al glass" arrays were measured, and provided the ability to obtain and study local atomic configurations and to determine partial and total radial distribution functions. The system free volume was also measured and compared with literature values. The results obtained from these models will be presented and discussed.

3:00 PM
Synthesis of $Ti_{34}Zr_{11}Ni_8Cu_{47}$ Bulk Metallic Glass by Warm Extrusion of Gas Atomized Powders: *Daniel Sordelet*[1]; Elena Rozhkova[1]; Matthew Kramer[1]; Paul Wheelock[1]; Matthew Besser[1]; Monique Calvo-Dahlborg[2]; Ulf Dahlborg[2]; [1]Ames Laboratory/Iowa State University, Metall. & Cer./Dept. of Matls. Sci. & Eng., 107 Metals Dvlp., Ames, IA 50014 USA; [2]LSG2M-UMR7584, Ecole des Mines, Nancy France

Bulk metallic glass was prepared by warm extrusion of $Ti_{34}Zr_{11}Cu_{47}Ni_8$ gas atomized powders. After consolidation near 723 K using an extrusion ratio of 5, the material retains between 88% and 98% of the amorphous structure found in the gas atomized powder. The onsets of the glass transition and crystallization temperatures of this extruded material are observed, respectively, at slightly higher and lower temperatures than those of the starting powders. Powders extruded at the same temperature, but using higher extrusion ratios of 9 and 13, exhibit substantial devitrification during consolidation, yet still deform homogeneously. A thin layer of a nanocrystalline phase rich in Ti and Zr was observed at some particle boundaries of extruded samples. Although the gas atomized powders contain approximately 700 ppm by weight O, parallel energy loss spectroscopy did not detect any O within this nanocrystalline phase. This devitrification may be related to localized frictional heating at the particle interfaces during extrusion.

3:20 PM
Consolidation of Metallic Glass Powders with Equal Channel Angular Extrusion: *K. T. Hartwig*[1]; I. Karaman[1]; Jae-Taek Im[1]; J. Robertson[1]; I. E. Anderson[2]; [1]Texas A&M University, Dept. of Mechl. Eng., MS-3123, College Station, TX 77843 USA; [2]Iowa State University, Ames Lab., Ames, IA 50011 USA

Previous research has shown that bulk amorphous metals can be fabricated by warm extrusion of rapidly solidified amorphous particulate. In the present study, the effectiveness of equal channel angular extrusion (ECAE) for consolidation of amorphous Zr-based powder is examined. ECAE subjects the workpiece to uniform simple shear but not area reduction and offers the opportunity for large cross section products. Powder, produced by gas atomization, was vacuum encapsulated in copper cans and extruded at a temperature above the glass transition temperature but below the crystallization temperature where the alloy behaves temporarily as a viscous liquid. The effects of extrusion temperature, strain rate, and strain level on consolidate porosity, degree of crystallinity and microstructure were examined. Results along with property comparisons to cast alloy are presented. Support for this work provided by DARPA under contract no. DAAD 19-01-1-0481.

3:40 PM Break

4:00 PM
Mechanical Properties of ECAE Consolidated Zr-Based Bulk Glassy Alloys: *Ibrahim Karaman*[1]; K. T. Hartwig[1]; J. Robertson[1]; [1]Texas A&M University, Dept. of Mechl. Eng., MS 3123, College Station, TX 77843 USA

Bulk metallic glasses (BMG) have received increasing interest during recent years because they are promising for use in engineering applications. Although many amorphous alloy systems have been known and studied for decades, their use for practical applications has been limited because of high cooling rate requirements and resulting product with at least one small dimension. Consolidation of particulates can circumvent this problem. The method involves viscous flow and atomic diffusivity in the supercooled liquid region with resultant fabrication of full density bulk glassy alloys. In the present work, a severe plastic deformation technique, equal channel angular extrusion (ECAE) is used for consolidation of atomized metallic glass powders. We will present a comparison of the mechanical properties of cast and conventionally extruded metallic glass powders with samples obtained by ECAE consolidation. The materials under study are Zr-based glassy alloys that are chosen for their high glass forming ability. Tensile, compressive and hardness properties are compared.

4:20 PM
Modeling of Thermal Tempering in Bulk Metallic Glasses: *Cahit Can Aydiner*[1]; Ersan Üstündag[1]; [1]California Institute of Technology, Eng. & Appl. Sci./Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA

Bulk metallic glasses (BMGs) have recently been shown to possess good mechanical properties and are suitable candidates for many struc-

tural applications. One of the critical issues that influence their properties is the existence of residual stresses in them. In this case, they originate due to "thermal tempering". In this process, the viscoelastic nature of these materials and the high cooling rates necessary in their production lead to compressive surface residual stresses balanced with mid-tension. We have modeled the development of these stresses starting with a simple instant freezing model and progressing towards a more complicated and data intensive structural model. This talk will compare the predictions of these models. The results suggest the existence of large stress gradients on specimen surface.

4:40 PM
Internal Stresses in Tungsten Fiber Reinforced Bulk Metallic Glass Matrix Composites: *Bjorn Clausen*[1]; Seung-Yub Lee[1]; Ersan Üstündag[1]; Mark A.M. Bourke[2]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, MST-8, PO Box 1663, MS H805, Los Alamos, NM 87545 USA

Bulk metallic glass (BMG)-matrix composites have been shown to increase the ductility of BMGs. However, the matrix-reinforcement interactions have not been fully understood. One important issue in such composites is the presence of internal stresses due to the mismatch in coefficients of thermal expansion between the matrix and the reinforcements as well as their elastic and plastic incompatibility under loading. In the present study, we have used neutron diffraction to measure the thermal residual strains in a series of bulk metallic glass matrix composites with varying volume fraction of tungsten fibers. As the neutron diffraction measurements only yield information about the fibers, finite element modeling has been used to predict the thermal residual strains in the BMG matrix. These composites have then been loaded in compression to study the in-situ mechanical deformation of the reinforcements and to understand their interaction with the matrix.

5:00 PM
Measurement of Residual Stresses in Bulk Metallic Glasses: *Cahit Can Aydiner*[1]; Ersan Üstündag[1]; [1]California Institute of Technology, Eng. & Appl. Sci./Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA

Recent studies have suggested that large residual stresses exist in bulk metallic glasses (BMGs) due to "thermal tempering". This mechanism involves quenching a viscoelastic material from above its glass transition region to room temperature. During this process, compressive residual stresses are generated on the surface while the middle is under tension. While such stresses could be measured conveniently by photolelasticity in silica-based glasses, the opacity of BMGs prevent the application of this method. Similarly, the amorphous structure of BMGs precludes the use of diffraction. We have, therefore, employed mechanical relaxation methods in this investigation. In this presentation, results will be shown from relaxation methods including hole drilling, layer removal and crack compliance. These results will then be compared to modeling predictions.

Advances in Molten Salt Processing Technology: Process Fundamentals

Sponsored by: Light Metals Division, Reactive Metals Committee
Program Organizer: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division, Argonne, IL 60439-4837 USA

Monday PM — Room: 614
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Sean M. McDeavitt, Argonne National Laboratory, Cheml. Tech. Div., Argonne, IL 60439 USA

2:00 PM Opening Remarks

2:05 PM
Determination the Free Energy of Formations of Na2Cr2O4 and Na2CrO4 using Na -b''-Al2O3 Solid Electrolyte: *Vilas D. Tathavadkar*[1]; M. P. Antony[2]; Animesh Jha[1]; [1]University of Leeds, Dept. of Matls., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK; [2]Post-Irradiation Studies Section, Fuel Chem. Div., IGCAR, Kalpakkam, TN 603 102 India

The thermodynamic properties of the Na2O-Cr2O3 mixtures were investigated using a Na -b''- Al2O3 solid electrolyte in the temperature range of 1100 to 1200 K. During the soda-ash roasting of chromite ores, both sodium chromite and sodium chromate are formed depending on the oxygen partial pressure and the amount of sodium carbonate. The experiments were carried out to measure the activity of Na2O in Na2O-Cr2O3 system using the following e.m.f cells: CELL-I :(-) Pt / Ar / Na2Cr2O4 + Cr2O3 || Na-b''-Al2O3 || Na2WO4 + WO3 / Air / Pt (+) CELL-II :(-) Pt / Air / Na2CrO4 + Cr2O3 || Na-b''-Al2O3 || Na2WO4 + WO3 / Air / Pt (+) The free energy of formation of sodium chromite and sodium chromate from solid Na2O and Cr2O3 was calculated using the e.m.f. values of cell I and II, and the activity of Na2O in Na2WO4 + WO3 reference electrode. The derived equations for DGo in the range 1100 to 1200 K for Na2Cr2O4 and Na2CrO4 are -213153 + 7.0172 T J/mole (+ 8 kJ) and -332173 + 64.164 T J/mole (+ 5 kJ) respectively. The relevance of controlling the oxygen partial pressure for the formation of water soluble chromium phases is emphasised. The results are compared with the available thermodynamic data from the literature. The experimental results obtained from the X-ray diffraction confirm the presence of sodium chromite and sodium chromate phases alone with Cr2O3 in the working half cells I and II respectively. The present investigation also compares the liquidus temperatures in the Na2CO3-Na2CrO4 system, which were determined by differential thermal analysis. The measured activities of Na2CrO4 and Na2Cr2O4 confirm the importance of controlling the oxygen partial pressure and soda-to-chromite ratio during the soda-ash roasting of chromite ores.

2:35 PM
Occlusion of Molten Chloride Salt by Zeolite 4A-Nature, Thermodynamics, and Application to Nuclear Waste Treatment and Disposal: *Dusan Lexa*[1]; Irving Johnson[1]; [1]Argonne, Cheml. Tech., 9700 S. Cass Ave., Argonne, IL 60439-4837 USA

Glass-zeolite ceramic waste forms are being developed at Argonne National Laboratory for the disposal of radioactive waste salts from electro-metallurgical treatment of spent nuclear fuel. The major component (70 wt%) of the process salt is the LiCl-KCl eutectic salt (58 mol% LiCl, 42 mol% KCl). In addition, the process salt contains NaCl, at least 16 other fission product chloride and iodide salts, and 2 mol% actinide chlorides, such as UCl3, NpCl3, and PuCl3. The melting temperature of the process salt is ~ 600 K. The selection of an optimal process salt treatment method requires detailed knowledge of the equilibrium ion-exchange zeolite/molten salt separation factors. These have been determined in a number of pseudo-ternary systems, including NaCl-LiCl-KCl, CsCl-LiCl-KCl, RbCl-LiCl-KCl, MgCl2-LiCl-KCl, CaCl2-LiCl-KCl, SrCl2-LiCl-KCl, BaCl2-LiCl-KCl, LaCl3-LiCl-KCl, CeCl3-LiCl-KCl, UCl3-LiCl-KCl, and PuCl3-LiCl-KCl, at 823 K, by chemical analysis of both the zeolite and the molten salt phases.

3:05 PM
Ion Exchange of Fission Products between Zeolite and a Molten Salt: *Mary Lou Dunzik Gougar*[1]; Michael F. Simpson[1]; [1]Argonne National Laboratory-West, PO Box 2528, Idaho Falls, ID 83403 USA

In an electrometallurgical process to treat spent nuclear fuel, a molten chloride salt solution accumulates fission products from the alkali, alkaline earth, lanthanide, and actinide families. One technique that has been identified for removing these fission products and extending the usable life of the molten salt is ion exchange with zeolite A. Salt fills the micropores of the zeolite, and ion exchange of fission products can then occur between the molten salt phase and the solid zeolite phase. Closed-system contact experiments have been performed using both radioactive, fission-product-loaded salt and non-radioactive surrogate salt. The time for the establishment of equilibrium has been determined, and initial data has been obtained in support of the development of a multi-component equilibrium model for the ion exchange of fission products between molten chloride salt and zeolite A.

3:35 PM Break

3:50 PM
Dissolution Kinetics of Silicon-Containing Sodium Fluosilicate in Stirred Bath of Molten Aluminium: Ibrahim Hamed Aly[1]; *Eng Ebrahiem Esmail*[1]; A. M. Omran[2]; [1]Minia University, Cheml. Eng. Dept., Fac. of Eng., Minia 61111 Egypt; [2]Aluminium-Company of Egypt, Matls. Sci. Eng., Nag-Hamady Egypt

In the present study an attempt was made to characterize the mechanism of silicon dissolution from sodium fluo-silicate lumps to molten aluminium. The experiments were carried out under the influences of various process parameters such as temperatures, particle size, Na2SiF6/Al ratio and dissolution time. The obtained results were explained in detailed, they showed that the silicon dissolution rate reached a maximum in the first 5 min., and then maintained constant. Silicon dissolution rate is also increases proportionally with increasing the particle size of sodium fluo-silicate, intensity of mixing and the Na2SiF6/Al

ratio. The analysis of the experimental results suggested that the solute silicon atoms diffuse across a liquid boundary layer that was formed between solid phase (i.e. Na2SiF6 crystals) and liquid phases. Also the silicon dissolution rate is controlled by diffusion which has an activation energy ranged between 2.824 and 1.902 kcal/mole.

4:20 PM
Kinetics of Molten Chloride Salt Sorption into Zeolite-4A: *Michael Forrest Simpson*[1]; Sara Marie Lance[1]; Gregory Lee Moore[1]; [1]Argonne National Laboratory, Eng. Tech. Div., PO Box 2528, Idaho Falls, ID 83406 USA

A critical step in the EBR-II spent fuel treatment process involves blending fission-product loaded chloride salt with anhydrous zeolite-4A. The salt-loaded zeolite is a precursor for the final ceramic wasteform, which is comprised of glass-bonded sodalite. The blending is achieved by heating granular salt and zeolite to 500C in a rotating v-mixer for no more than 24 hours. The salt is ultimately occluded inside of the zeolite's alpha cages due to this process. Numerous salt sorption kinetics experiments using a laboratory-scale v-mixer have been performed in an effort to understand the factors that contribute to the rate of salt occlusion. A kinetic model for salt sorption in the zeolite has also been formulated with kinetic parameters estimated based on the experimental results.

4:50 PM
The Microreversibility of the System CuFeS2/Electrolyte: *Z. D. Stankovic*[1]; M. Rajcic-Vujasinovic[1]; Z. Stevic[1]; V. Fajnisevic[1]; [1]University of Belgrade, Techl. Fac. Bor, 19210 Bor Yugoslavia

In this paper results of the investigation of the microreversibility of the system CuFeS2/electrolyte are presented. The galvanostatic single pulse method has been used for investigation. Namely, the current pulses of the different amplitude and constant duration (40s) have been applied to electrode system composed of CuFeS2/electrolyte. The potential-time response in the form of the rising and decay curves were recorded. From the decay curves the conclusions of the reversibility of the system CuFeS2/electrolyte were evaluated.

5:20 PM
Molten Salt Separation from Uranium during the Processing of Spent Nuclear Fuel: *Brian R. Westphal*[1]; [1]Argonne National Laboratory, PO Box 2528, Idaho Falls, ID 83403-2528 USA

As part of the electrometallurgical treatment project at Argonne National Laboratory, a vacuum distillation process is being employed for the recovery of uranium following an electrorefining process. Distillation of a molten salt electrolyte, primarily consisting of a eutectic mixture of lithium and potassium chlorides with minor amounts of fission product chlorides, from uranium is achieved by a batch operation called cathode processing. The degree of molten salt separation from uranium at the cathode processor is termed distillation efficiency. This paper discusses the efficiency of the cathode processor via the use of representative isotopic tracers of the fission product chlorides. The relationship between distillation efficiencies, chloride vapor pressures, and relative concentrations is also discussed and applied to other chloride species of the system.

Alumina and Bauxite: Bayer Residue Handling

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Jacques M. Mordini, Aluminium Pechiney, Gardanne, Cedex 13541 France; Steve Rosenberg, Worsley Alumina Pty, Ltd., Process Chemistry Group, Collie, West Australia 6225 Australia; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Monday PM — Room: 609
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Steven P. Rosenberg, Worsley Alumina Pty Ltd., Proc. Chem. Grp., PO Box 344, Collie, WA 6225 Australia; Jacques Mordini, Aluminium Pechiney, Direction Industrielle, Boite Postale 54, Gardanne Cedex France

2:00 PM
Behavior of Red Mud Agglomerated with Various Flocculants under Shear Conditions: *Michel Gagnon*[1]; Guy Simard[1]; Mélanie Normandin[1]; Guy Péloquin[2]; [1]Université du Québec à Chicoutimi, Appl. Scis., 555 boul Universite, Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International, Limitée, Jonquière, Québec G7S 4K8 Canada

In the Bayer process, flocculating agents play an important role in the efficiency of the gravity settlers. They are added to the red mud slurry in the feed pipe and in the feed well. The flocculation process is certainly affected by the changing turbulent flow conditions existing in the pipe and at some point in the feed well. To gain a better understanding of the influence of flocculant properties and structure on the shear-flocculation occurring at these levels of the process, the behaviors of three commercially available flocculating agents were studied under shear conditions using concentric rotating cylinder (Couette system). The data obtained are expressed in terms of settling velocity, residual turbidity, and floc size distributions.

2:25 PM
Rheology Aids for Effective Mud Disposal: *Stephen John Adkins*[1]; Dana Therese Smith[2]; [1]Ciba Specialty Chemicals, Extractive Industries, PO Box 38, Low Moor, Bradford, W. Yorkshire BD12 0JZ England; [2]Alcoa World Alumina Atlantic, Point Comfort Operations, PO Box 101, Point Comfort, Texas 77978 USA

The process of red mud slurry stacking is widely used within the alumina industry. The physical and process variables which have a direct influence on this application, including solids content, particle size distribution and physical shear, have been previously investigated and documented. In many instances the physical properties of a final thickener underflow will meet the requirements for effective mud stacking. However, a significant proportion of the yield stress of the thickener underflow arises from the network structure imparted to it by the polymer added during the flocculation and thickening process. The necessity to transport the flocculated mud significant distances to a disposal area can result is a dramatic reduction in its rheological properties, caused by a break down of the network structure, and thereby reducing its ability to stack effectively. The physical and operational variables, together with operational constraints present within an alumina process, mean that it is difficult to maintain control of the physical properties of red mud for optimal disposal. The introduction of a rheology aid to the final mud could negate the impact of these variables, allowing optimum stacking performance to be maintained. This paper demonstrates the effect of such rheology aids in both laboratory and plant situations.

2:50 PM
Revised Application of Synthetic Flocculants in Red Mud Separation: *John Patrick Murray*[1]; [1]Alpart, Techl. Serv., PO Box 529, Arabi, LA 70032-0529 USA

Alumina Partners of Jamaica (Alpart) has used synthetic flocculants in Red Mud Separation since the late 1970s. These polymers were typically supplied to the vessels at low concentrations (0.05 to 0.10vol.%) following popular technical guidelines. During the late 1990s, Alpart was challenged to reduce flocculant consumption. An audit indicated that flocculant activity loss due to excessive degradation in the distribution piping was a major problem. Based on field experimentation, activity loss was eliminated at concentrations in the region of 0.4 to.5vol.%. Furthermore, concentrations on the order of 1.5 to 1.6 vol.% improved flocculant performance on the settlers (thickners). Since late 2000, Operations have been modified to reflect these findings, resulting in: 1) Flocculant usage reduced in excess of 30%, 2) Increased stability of the settlers and washers, 3) Reduced soda losses.

3:15 PM Break

3:35 PM Cancelled
Implementation of Turbidness Automatic Control in Thickener and Washer Tanks at C.V.G. Bauxilum: *Jesus Noya*

4:00 PM
Bauxaline: A New Product for Various Applications of Bayer Process Red Muds: *Valerie Martinent-Catalot*[1]; Jean-Michel Lamérant[1]; Gérard Tilmant[1]; [1]Aluminium Pechiney, Alumina Tech., BP 54, Gardanne 13541 France

Gardanne Pechiney plant produces 650000 tpy of alumina along with about 320000 tpy of red muds presently dumped into a deep submarine hole. For many years important R&D programs had been conducted to find possible ways of valorization of red muds, named as per december 2000 Bauxaline for commercial applications. Two main approaches are presently in progress: one consisting in thickening the muds so as to get a large amount for applications as for example dump rehabilitation, artificial reefs, filler for injection; one taking advantage of coloring properties, this application needs a more sophisticated preparation. The paper describes two particular uses of Bauxaline: arsenic fixation on co-disposal projects and coloring of various materials.

4:25 PM
CVG-Bauxilum Red Mud Neutralization: *Rodrigo R. Carneiro*[1]; Robert E. Keane[1]; Ricardo A. Galarraga[2]; Giao Nguyen[3]; [1]KD Engineering Company, 7701 N. Business Park Dr., Tucson, AZ 85748 USA; [2]CVG-Bauxilum, Ave. Fuerzas Armadas, Zona Industrial de Matanzas, Ciudad Guayana, Venezuela; [3]Atomaer Pty, Ltd., 14 Bell St., Canning Vale 6155 Western Australia

CVG-Bauxilum has been working toward the best alternative to neutralize and dispose the red mud and liquor generated from the alumina Bayer process. This has been done to eliminate any potential environmental impact on the near by Orinoco river. In June 2001, CVG-Bauxilum and ATOMAER-KD Engineering Co., Inc., conducted an on site pilot red mud neutralization study using carbon dioxide and the ATOMAER gas shearing technology. The pilot test program successfully reduced the red mud pulp pH from 12 to 7.60. The metallurgical results indicated that the process employed by CVG-Bauxilum and ATOMAER-KD Engineering Co., Inc., is feasible to neutralize the residue from the Bayer process.

Aluminum Reduction Technology: Environmental

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Martin Segatz, VAW Aluminum AG, D-53117, Bonn Germany; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Monday PM — Room: 6B
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Nancy J. Holt, Hydro Aluminium Metal Products, Tech. Ctr. Årdal, Øvre Årdal N-6882 Norway

2:00 PM
Can the Operator Take the Heat?: *Nancy J. Holt*[1]; Eldbjørg Saksvikrønning[2]; [1]Hydro Aluminium Metal Products, Tech. Ctr. Årdal, Øvre Årdal N-6882 Norway; [2]Hydro Aluminium Metal Products, Hydro Aluminium, Årdal Smelter, Øvre Årdal N-6882 Norway

All workers exposed to a hot and sometimes also humid environment may run the risk of heat stress and especially so if the work is strenuous. Work in the aluminium industry still includes several operations where the workers are exposed to potentially severe heat stress. The most widely accepted method used to evaluate heat stress is the Wet Bulb Globe Temperature (WBGT) index. The last couple of years Hydro Aluminium has gathered information on several work routines where the operator is exposed to heat stress. Although focus has been on potroom operations at several locations, measurements have also been done in the cast house and anode paste plant. This survey has been performed not only to find out if work routines are within international standards on recommended maximum heat stress levels. Another aspect of the measuring campaigns has been to gather information from different smelters with different technological solutions, and then compare these to see if there are possibilities for improvements that can reduce heat stress. Further, when e.g. a current increase section or new routines in anode change has been introduced, some of these have been evaluated with regards to heat stress. Heat stress evaluation is usually a natural part of exposure risk assessments. The information and experience with heat stress measurements have contributed significantly to a revised heat stress evaluation procedure for smelters in Scandinavia.

2:20 PM
Latest Developments on Dry & Wet Scrubbers Dedicated to Treatment of High Amperage Pots: *Andrew Haberl*[1]; Jean-François Langle[2]; [1]Procedair Industries, Inc., Techl. Div., 625 President Kennedy, Montreal, Quebec H3A, 1K2 Canada; [2]Procedair SA, Sales Dept., 25 Blvd. de la Paix, Saint Germain en Laye, Yvelines 78100 France

Major electrolysis technology providers offer today the capability for building large capacity primary aluminium smelters based upon a limited number of very high amperage pots. These smelters feature several particular parameters which are to be considered in the design of relevant modern Dry-Scrubbing Systems (DSS), such as i) requested increase of efficiency on HF, F particulate and total dust removal; ii) limited available area for DSS installation due to restricted distance between potrooms; iii) intensive magnetic field. Depending upon local regulations, wet scrubbers (WS) for SO2 removal together with optional waste treatment may be installed downstream above DSS. The aim of the present paper is to highlight the developments which have been recently implemented on DSS and WS in order to achieve the latest requirements from both environmental authorities and electrolysis plant operators.

2:40 PM
Retrofit of Dry Scrubbers Pays Back: *Geir Wedde*[1]; [1]ALSTOM Power Norway, Boilers & Environ., Ole Deviksvei 10, Oslo 0666 Norway

Since early 1970's the dry scrubbing process became the state of the art emission control and fluoride recovery technology for treating fumes from aluminium reduction lines. Many (most) of these scrubbers are still in operation 20-30 years later. Technology improvements and cost reduction programmes have ensured more efficient dry scrubbers. Retrofitting potlines with the latest dry scrubbing technology give healthy investment returns from reduced operating costs and other important benefits such as operators improved health and safety, reduced emissions and improved availability.

3:00 PM
Analytical and Experimental Study on Fluoride Evolution: Abdelhamid Meghlaoui[1]; Yussuf Ali Mohamed Al Farsi[1]; *Najeeba Hassan Al Jabri*[1]; [1]Dubai Aluminium Co., Ltd (Dubal), Tech. Dvlp. Dept., PO Box 3627, Jebel Ali, Dubai UAE

Aluminium fluoride is consumed within the electrolysis cell by hydrolysis reactions, reactions with alumina impurities and cathode reactions. It is difficult to calculate theoretically the AlF3 losses using physical and chemical equations because of the effect of other factors. Haupin considers the HF gaseous and particulate emissions as major and proposed a model to estimate them. The model is more oriented towards the environmental aspect than controlling the AlF3 additions. Entner proposed a multivariate regression model based on the AlF3 mass balance, which estimates the overall AlF3 losses, but doesn't specify their repartition. In this study, we implemented an AlF3 control strategy based on Entner model and fuzzy logic. Then calibrated Haupin model, using correlation equations from an experimental trial, and used it as soft sensor. A long-term run shows that AlF3 additions based on fuzzy-Entner control strategy is balanced with Haupin total emissions and reactions due to alumina impurities.

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The Influence of Dry Scrubber Alumina and Humidity on COS Smelter Emissions: *S. J. Hay*[1]; M. M. Hyland[1]; J. B. Metson[2]; O-M. Bade[3]; [1]The University of Auckland, Dept. of Cheml. & Matls. Eng., PB 92019, Auckland New Zealand; [2]The University of Auckland, Dept. of Chem., PB 92019, Auckland New Zealand; [3]Alstom Power (Environment), Oslo Norway

Carbonyl sulfide (COS) emissions from aluminium smelting are modest compared to emissions of HF or SO2. However, the potential environmental effects of COS require that COS produced during aluminium smelting be considered. These studies aim to determine the activity of smelter-grade alumina used in dry scrubbing, in reaction with COS. In addition, the effects of humidity on this system have been considered. The major source of COS is from the 2-3wt% sulfur impurity present in anodes consumed during the production of aluminium. While COS is the main sulfur gas to be produced at the anode, it is largely oxidised to SO2 on contact with air in the headspace of the cell and in the ducting. Several groups have monitored COS entering and leaving the dry scrubber, but there are inconsistencies in their conclusions from these measurements, suggesting that local conditions in the dry scrubber affect the concentration of COS. Thermodynamic calculations were completed to predict the stable phases at equilibrium when starting with COS in the presence and absence of alumina and in different gas compositions. Results were compared with laboratory studies, performed by passing COS containing gases through a fluidised bed of alumina and measuring the outlet gas composition. Alumina type was varied, in addition to inlet gas composition, which allowed the effect of humidity to be considered. After reaction, alumina from the fluidised bed was studied using a variety of surface sensitive methods. Experimentally COS was found to be stable in the apparatus when in isolation, although the thermodynamic calculations predicted otherwise, pointing to the importance of kinetic factors. The presence of smelter-grade primary alumina resulted in partial hydrolysis of COS, producing H2S. A sulfur mass balance indicated some adsorption of COS on the alumina surface, which appeared to lead to saturation of the alumina. Secondary alumina from a typical dry scrubber was found to be unreactive towards COS, suggesting that the presence of F and S reduced the activity of the alumina surface. The presence of humidity altered the extent of conversion of COS to H2S and prevented saturation of the alumina surface for the duration of a typical experiment.

3:50 PM
Reduction of CF4 Emissions at the Smelter in Essen: *Martin Iffert*[1]; Regina Ganther[1]; Juergen Opgen-Rhein[1]; [1]Aluminium Essen GmbH, Electrolysis, Aluminiumallee 1, Essen D-45356 Germany

PFC emissions are harmful to the environment regarding their global warming potential and therefore it is a challenge to the aluminium industry to reduce the evolution of these gases. Based on this background we carried out two measurement campaigns of CF4 emissions from our smelter in Essen during spring 2001. Within the first campaign the current level of emissions was fixed and different strategies of automatic anode effect treatment were analysed and compared. We paid special attention to the success rate and the CF4 emission levels of the anode effect quenching strategies. Afterwards the most successful method with the lowest emissions was implemented in all process computers. In the second campaign the improvements were verified. This paper shows the correlation between anode effect over voltage and CF4 emissions that we have found in our campaigns. As well we point out our suggestions and trials about anode effect quenching strategies.

4:10 PM
Interaction Between Aluminium Process and Powder Technology: *Morten Karlsen*[1]; Are Dyroey[2]; [1]Hydro Aluminium, Tech. Ctr. Årdal, PO Box 303, Øvre Årdal 6882 Norway; [2]Tel-Tek, POSTEC, Kjoelnes Ring, Porsgrunn 3918 Norway

Historically, the development of the aluminium industry has been focused on the improvement of the electrolysis process. However, the largest volumes of handled materials are alumina and petrol coke, which are bulk solids. The interfaces between the pots and the fume treatment facilities often present challenges to which the application of powder technology may provide feasible and good solutions. The dusting problems of the open Søderberg pots represent a severe limitation with respect to fulfilling future environmental requirements. Based on correlations between dust and alumina fines content, promising test rig results initiated the development of a continuous de-dusting unit. This unit separates the alumina fines and reroutes this fraction into the feed of the dry scrubber section serving a prebake pot line. Increasingly strict environmental demands inevitably cause old fume treatment facilities to be replaced or retrofitted. If a retrofit is possible the harvest time of the initial investment may be prolonged for 5 to 10 years, giving the opportunity to plan and design a possible new expansion. By modifying the alumina injection points into the gas reactor, the cleaning efficiency of the dry scrubber can be improved. Implementation of the new injection technique has reduced the outgoing concentration by 72%.

4:30 PM
New Aerated Distribution (ADS) and Anti Segregation (ASS) Systems: *Bernt Nagell*[1]; Gisle G. Enstad[2]; Peter Hilgraf[3]; Morten Karlsen[4]; Are Dyroey[2]; [1]Norsk Hydro, Hydro Karmoey Aluminium, Haavik 4265 Norway; [2]Tel-Tek, POSTEC, Kjoelnes Ring, Porsgrunn 3918 Norway; [3]BMH, Claudius Peters Buxtehude, Schansenstrasse 40, Buxtehude 21614 Germany; [4]Hydro Aluminium, Tech. Ctr. Årdal, PO Box 303, Øvre Årdal 6882 Norway

Two tons of alumina is required to produce one ton of Aluminium. The logistics of alumina therefore represents an important part of the Aluminium production, including unloading of ships, internal transport and storage; fume treatment and finally distribution into the electrolysis pots. Over the last 20 years several automated distribution systems utilizing pneumatic conveying have been developed. These systems are closed, resulting in less dusting. However, the erosive properties of alumina are better countered by the use of low velocity conveying. Hence the development of an air-slide based system was implemented. Also low velocity and low-pressure drop reduces the ability of secondary alumina to form scales. We present a new system utilizing low conveying velocity air-slides with adequate capacities. The Aerated Distribution System (ADS) has been installed and tested in regular production and shows almost eliminated need for maintenance. The system also includes a separation unit to remove the scales, eliminating scaling problems. The difference in maintenance frequency, the lifetime cost and the "up-time" strongly favours low velocity systems. Another advantage of distribution systems is that several pots fed by these systems, more easily may be treated as one unit instead of several individual pots. When introducing automated distribution systems, improved homogeneity of the ingoing alumina is usually required to ensure stable logistic flow. A system to reduce segregation when filling silos and A-frames has been developed and operated in production, demonstrating a homogenizing factor of 1.47 on the alumina put through the system.

Automotive Alloys 2002 - I

Sponsored by: Light Metals Division, Aluminum Association,
Program Organizer: Subodh K. Das, Secat, Inc., Lexington, KY 40511 USA

Monday PM — Room: 611
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Subodh K. Das, Secat Inc., 1505 Bull Lea Blvd., Lexington, KY 40511 USA

2:00 PM
Assuring Continued Recyclability of Automotive Aluminum Alloys: Grouping of Wrought Alloys by Color Sorting and Chemical Composition-Based Sorting: *Adam J. Gesing*[1]; Tim D. Good[1]; F. Scott Costello[1]; Larry Berry[1]; Richard B. Wolanski[1]; Paul B. Schultz[2]; [1]Huron Valley Steel Corporation, R&D, 41000 Huron River Dr., Belleville, MI 48111 USA; [2]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069-0001 USA

Although automotive aluminum is currently completely recycled, recycling technologies need to be improved to assure continued, high-value recyclability of all present and future automotive alloys–particularly wrought alloys, as the use of these alloys increases in the aluminum intensive vehicle. The Automotive Aluminum Alliance has designed a program to demonstrate the ability of new scrap sorting technologies to deliver high-value recyclability of aluminum scrap from end-of-life vehicles. Huron Valley Steel Corporation, the developer of these sorting technologies, is participating in this demonstration program. In this paper, we compare the alloy groupings for wrought scrap obtained using two techniques: color sorting of particles tinted by etching in an NaOH solution, and chemical composition-based alloy sorting.

2:30 PM
Filler Alloy Selection for Aluminum Welding: *Tony Anderson*[1]; [1]AlcoTec Wire Corporation, 2750 Aero Park Dr., Traverce City, MI 49686-9263 USA

When considering the welding of aluminum alloys, and the development of welding procedures one of the main considerations must be that of filler alloy selection. Typically there are a variety of filler alloys available which may be used to weld any given base alloy combination. However there are a number of variables associated with selection of the most suitable filler alloy to be used. These variables are largely based on product application and are as follows: Strength of the weld, Ductility, Corrosion Resistance, Sustained Temperature Service, Color Match After Anodizing, and Post Weld Heat Treatment. The understanding of these variables is a significant aspect in ensuring the correct design and development of a successful welding procedure.

2:55 PM
Effect of Quasi-Static Prestrain and Eddy Currents on Limit Strains in Electromagnetic Pulse Assisted Stamping of Two Aluminum Alloys: *Vincent Joseph Vohnout*[1]; Glenn S. Daehn[1]; [1]The Ohio State University, Matl. Sci. & Eng., 2041 College Rd., Watts Hall 177, Columbus, OH 43210 USA

The integration of electromagnetic pulse energy with conventional stamping shows promise as a method capable of overcoming the well known forming limit problems of stamped aluminum parts. The fundamental viability of this combined, hybrid process was demonstrated by conducting a series of experiments with 0.8 mm thick coupons of two aluminum alloys, 6111-T4 and 5754-0. The coupons were first quasi-statically strained to various levels terminating a few percent below the conventional forming limit. Each coupons was subsequently expanded rapidly, with an electromagnetic pulse, to failure. Records of the failure strains generated by the combined processes show a consistent increase in limit strains for this hybrid process when compared to either the conventional or high speed process applied singly. The effects of material type and expansion velocity on the extended formability limit was investigated as was the effect of eddy current joule heating of the coupon.

3:20 PM
The Influence of Surface Roughness on the Deforming Friction Behavior of Al-Mg Alloys: *Mark R. Stoudt*[1]; [1]NIST, Matls. Perfor. Grp., 100 Bureau Dr., MS 8553, Gaithersburg, MD 20899-8553 USA

The inhomogeneous plastic flow exhibited by numerous aluminum alloys presents significant challenges to their use in automotive appli-

cations. The size and distribution of surface asperities influence the friction between mating die surfaces resulting in accelerated die wear and progressively reduced shape accuracy in the metal stampings. Finite element predictions of friction are often inconsistent with that observed in real measurements. This suggests additional studies are required to improve the understanding of the fundamental relationships between surface roughness and friction under deforming loads. Friction measurements typically draw metal sheets over a mandrel making evaluation of the material behavior under the deforming loads difficult. A measurement technique was developed to determine the frictional coefficients and to enable detailed assessments of the material response to the deforming loads. Measurements were performed on thin Al-Mg alloy specimens with controlled levels of initial surface roughness under varying normal forces. The results will be presented and discussed.

3:45 PM Break

4:00 PM
Using the Method of an In-Situ Thermal Analysis Array in a Cast Section of an Engine Block Casting to Diagnose the Cause of Shrinkage Porosity: *M. Djurdjevic*[1]; R. I. Mackay[1]; J. H. Sokolowski[1]; D. Cusinato[2]; A. Mueting[2]; [1]NSERC/Ford-Nemak/University of Windsor Industrial Research Chair in Light Metals Casting Technology, Mechl., Autom. & Matls. Eng., Rm. 203, Essex Hall, 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Nemak Corporation of Canada, Windsor Aluminum Plant, 4600 G. N. Booth Dr., Windsor, Ontario N9C 4G8 Canada

Hot spots are undesirable occurrences during the cast solidification process since they lead to a higher level of porosity in the cast structure. Many techniques have been developed to identify the propensity of hot spots and they include using thermocouple arrays in cast moulds. This work was done in order to determine the specific solidification kinetics, which occurred in the cast section of interest that yielded the observed shrinkage porosity. The observation of, what will be described in this work as, a latent heat generated hot spot phenomenon was made. This phenomenon resulted in cooling in rates for the primary a-Al dendritic phases and for the Al-Si eutectic to change from one cast section to another. This was the result of the accumulative heat extraction by the mould wall and the partially solidified metal and the latent heat release of the solidifying Si crystals.

4:25 PM
Formability Performance of 5XXX Series Aluminum Alloys Produced with Twin-Roll Casting Technique: *Murat Dundar*[1]; Abdullah Soner Akkurt[1]; Chris Romanowski[2]; [1]ASSAN Aluminum, E5 Karayolu 32. Km., Tuzla, Istanbul 81700 Turkey; [2]FATA-Hunter, 6147 RiverCrest Dr., Riverside, CA 92507 USA

The ultimate goal of the present study was to produce twin roll cast 5XXX series alloys and related processes suitable for products involving severe press shop operations, with similar, or preferably better, properties than the corresponding DC-cast material. In connection with the previous paper of the authors that has involved microstructural and mechanical characterization of twin roll cast AA5052, AA5754 and AA5182 alloys aiming to automotive applications, formability performance of these materials were investigated by constructing their Forming Limit Diagrams (FLD) in the present study. Tendency of the materials to the localized necking and its plastic anisotropy were correlated with the FLD results through the material properties of n and r values. These results were compared with those of similar alloys produced by the DC casting route. Influence of microstructural features, that were either developed during processing route or intrinsically existing from casting, on failure limits of the alloys were elucidated by utilizing different characterization techniques. Contribution of grain structure and intermetallic particles on surface cracking and strain inhomogenities that could be developed by centerline segregation during biaxial loading were investigated.

4:50 PM
Effect of Second Phase Particles on Bendability of Al-Mg-Si Alloy Sheet: *Hidetoshi Uchida*[1]; Mineo Asano[1]; Hideo Yoshida[1]; [1]Sumitomo Light Metal Industries, Ltd., R&D Ctr., 3-1-12 Chitose, Minato-ku, Nagoya, Aichi 455-8670 Japan

Recently Al-Mg-Si alloy sheets are applied to automotive closure panel for good paint bake response and better recyclability. Improvement of bendability on Al-Mg-Si alloy sheet requires for hemming performance. In this study, effect of second phase particles on bendability of Al-Mg-Si alloy was investigated. Solution heat treatment time and quenching rate were changed to obtain several conditions of second phase particles. In regard to quench rate, fast rate is better for bendability because less the second phase particles on grain boundary. In regard to the solution heat time, level of crack after bending was becoming worse in less than 90 s and more than 300 s the level was becoming better. The reason of the phenomena was considered that combination of formation of microband by matrix deformation and size of the second phase particles.

5:10 PM
Effect of Casting Defects on Mechanical Properties of Magnesium Alloy Castings: *A. M. Gokhale*[1]; G. R. Patel[1]; [1]Georgia Institute of Technology, Sch. of Matl. Sci. & Eng., Atlanta, GA 30332-0245 USA

Die cast AM60 and AZ91D magnesium alloys show a large variation in tensile strength and ductility within a group of castings cast under the same average process conditions and chemistry. It is, therefore, important to understand the factors that affect such variability in the mechanical properties. In an earlier contribution, we presented quantitative microstructural and fractographic data on AM60 alloy castings and their correlations with the variability in the mechanical properties. In this contribution, we present our quantitative data on variability in mechanical properties of AZ91D magnesium alloy castings to establish quantitative correlations with microstructural and fractographic parameters. For this purpose, the fracture surfaces of the tensile test specimens and the bulk microstructures have been quantitatively characterized. In these cast microstructures, porosity (shrinkage and gas and/or air) is a dominant type of defect. A method to quantify porosity on the fracture surface and in the bulk microstructure has been developed and quantitative relationships between porosity on the fracture surface and the tensile ductility as well as UTS are established. A comparison is made between these data and the earlier data obtained on AM60 alloy castings.

Carbon Technology: Anode Raw Materials and Anode Property Testing

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Don T. Walton, Aluminum Company of America, Wenatchee Works, Malaga, WA 98828-9728 USA; Les Edwards, CII Carbon, Chalmette, LA 70044 USA; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Monday PM	Room: 602-603
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Robert H. Wombles, Koppers, Pittsburgh, PA 15238 USA; J. Tom Baron, Koppers, Pittsburgh, PA 15238 USA

2:00 PM
Production of Low-PAH-Pitch for Use in Söderberg Smelters: *Winfried L. Boenigk*[1]; Gord H. Gilmet[2]; Dirk Schnitzler[1]; Jens Stiegert[1]; Mike Sutton[2]; [1]RUETGERS VFT AG, Kekuléstr. 30, Castrop-Rauxel 44579 Germany; [2]VfT, Inc., 725 Strathearne Ave. N., Hamilton, Ontario L8H 5L3 Canada

The legal understanding of PAHs differs from its scientific definition. Therefore the composition of low-PAH-pitches may vary from country to country. Strategies to produce low-PAH-pitches using coal tar and petroleum feedstocks will be discussed. The quality characteristics of low-PAH-pitch depend on the applied production strategy. Advantages and disadvantages of several production options will be pointed out. Industrial use of low-PAH-pitches for Söederberg applications has started.

2:25 PM Cancelled
Average Molecular Weight Measurement of Binder Pitch by Thermogravimetric Analysis(TGA) Method: *Kwang Eui Yoon*

2:50 PM
Alcan Characterization of Pitch Performance for Pitch Binder Evaluation and Process Changes in an Aluminum Smelter: *Amir A. Mirchi*[1]; Gaby Savard[1]; Jean-Pierre Tremblay[1]; Michel Simard[2]; [1]Alcan International, Ltd., Reduction Tech., 1955 Mellon Blvd., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada; [2]Alcan Primary Metal Group, Info. Tech., 1955 Mellon Blvd., PO Box 1500/1640, Jonquiere, Quebec G7S 4L2 Canada

Coal tar pitch is the main raw material used as a binder for the anode fabrication. The characteristics of the pitch binder affected the paste plant operation and anode performance during electrolysis. Over the last two decades, the coke oven design and technology have been improved to minimize the environmental emission and to increase the

coke oven productivity. The properties of the coal tars used as raw materials for pitch production have been affected by this change. The pitch producer also uses more and more different tar sources. In the same period, the aluminum industry increased its requirement in terms of anode quality to improve the cell control, productivity and to increase the cell amperage. The impact of the pitch property evolution on the paste and electrode performance has been studied. A new experimental device has been developed by Alcan to measure the pitch wetting capability for pitch binder evaluation.

3:15 PM
The Characterization of Pre-Baked Carbon Anodes using X-Ray Computed Tomography: *Angelique N. Adams*[1]; [1]The Pennsylvania State University, 401 Academic Activities Bldg., University Park, PA 16802 USA

Carbon anodes represent about 25% of the production cost of aluminum, therefore quality control is important to the smelting operation. Typical quality control methods involve testing cores from production and scrap anodes. One parameter of particular interest is density. The aim of this research is to determine the density profile of carbon anodes through non-destructive analysis. X-ray computed tomography (CT) is a technique that can determine the density of materials and represent the information visually at high resolution. A bench-scale green anode was scanned using X-ray CT, over the length of the anode. The anode was baked in an industrial laboratory and re-scanned at the same registration. The density data were converted into a series of images. The density difference between the green and baked sample is apparent. The presence of additional features such as butts and localized areas of inadequate amounts of binder were also observed.

3:40 PM Break

3:50 PM
New Method for Measuring Thermal Conductivity of Coarse Grain Carbon Materials: *Peter S. Gaal*[1]; Heng Wang[1]; Silviu P. Apostolescu[1]; [1]Anter Corporation, 1700 Universal Rd., Pittsburgh, PA 15235 USA

Thermal conductivity and thermal diffusivity are two very important physical properties of carbon materials that can substantially impact upon the life cycle of anodes. The two properties are interrelated through density and specific heat capacity. The role of thermal conductivity in the performance of anodes is well known, while the role of thermal diffusivity, a dynamic heat transport parameter, is largely unknown. This is partly due to the complexity of measuring it, and the historically very small samples used in laser flash measurements, which in case of coarse grain materials, may not always be representative. This paper describes work aimed at extending the inherently fast flash technique to measure these thermal properties of carbon anode materials, by using larger samples (up to 30 mm diameter x 8 mm thick). Data is presented comparing results obtained with using the new larger size samples and conventional small samples. The results are also compared to those obtained using conventional steady-state heat flow methods.

4:15 PM
The Use of Image Analysis for the Optimization of Pre-Baked Anode Formulation: *Angelique N. Adams*[1]; [1]The Pennsylvania State University, 401 Academic Activities Bldg., University Park, PA 16802 USA

Carbon anodes represent about 25% of the production cost of aluminum. The direct cost is that of the consumable raw materials. The indirect cost is electrode resistance, which contributes to energy consumption. It has been well established that anode life and electrical resistivity are optimized when the binder quantity is optimized. The aim of this research is to determine if semi-automated image analysis can be used to determine the appropriate binder quantity for a given coke particle size distribution. Samples of anode mix were taken from a bench-scale anode production process, and characterized using a microscope with semi-automated image analysis. The thickness of the pitch film around coarse coke particles was determined. The results show good agreement between the quantity of pitch measured using image analysis and the amount added in the production process.

4:40 PM
Air Reactivity Factors Affecting Anode Performance: Frank R. Cannova[1]; *Bernard C. Vitchus*[1]; [1]BP, 300 Oceangate, Long Beach, CA 90802 USA

Anode reactivity is used to monitor anode quality and explain anode performance. Data will be presented which shows how anode air reactivity varies in anodes made from the same calcined coke but with different reactivities due to sodium content and air permeability properties. These reactivity results were obtained on cores taken from the center of commercial anodes. However, the air and CO2 reactivity of the anode occurs at the anode surface. The reactivity of the anode surface can be different than a core taken from the center of the anode. Air reactivity data is presented for surface anode and butt specimens. These data provide insight of how anode baking, effect of soft butts and cryolite from cells can affect the surface reactivity of anodes.

5:05 PM
Gas Reactivity Inside Industrial Anodes: *Marianne Aa. Engvoll*[1]; Harald A. Øye[1]; Morten Sørlie[2]; [1]Norwegian University of Science and Technology, Inst. of Chem., Trondheim N-7491 Norway; [2]Elkem Aluminum Research, PO Box 8040 Vaagsbygd, Kristiansand N-4675 Norway

Bath compounds, especially cryolite, sodium fluoride and cryolite, act as strong catalysts towards the air and CO2 gasification reactions. To prebaked anodes, considerable amounts of fluorides are introduced via the addition of butts. Dust accumulation on the anode top and penetration of NaAlF4 gas through the open porosity appear to be important contamination sources in Söderberg anodes. By use of electron microscopy, this work examines the extent of catalyzed gasification inside industrial prebaked and Söderberg anodes. The results are related to the gas permeabilities, contamination profiles and laboratory reactivity experiments.

Cast Shop Technology: Modeling II

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: David H. DeYoung, Alcoa Technical Center, Alcoa Center, PA 15069 USA; John F. Grandfield, CSIRO Australia, Preston, Victoria 3072 Australia; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Monday PM — Room: 6A
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Yogesh Sahai, Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; Bjørn Rune Henriksen, Elkem ASA, PO Box 8040 Vaagsbygd, Kristiansand 4675 Norway

2:00 PM Keynote
Mathematical Modeling in Aluminum Casting and Molten Metal Treatment: *James William Evans*[1]; W. Kinzy Jones[2]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]Motorola, Adv. Products Tech. Ctr., 8000 W. Sunrise Blvd., Ft. Lauderdale, FL 33322 USA

The paper reviews the mathematical models that have been developed in recent years, at Berkeley and elsewhere, to simulate the DC and electromagnetic casting of aluminum, along with models for other cast shop operations such as melting or gas fluxing. Wherever possible the predictions of the mathematical models are compared with experimental measurements, either on actual plant operations or on physical models.

2:30 PM
Inverse Analysis of Space and Time Variations in the Boundary Heat Flux during Water Film Cooling: *Einar Arne Sørheim*[1]; Dag Mortensen[1]; Steinar Benum[2]; Christian Stette[2]; [1]Institute for Energy Technology, Matls. & Corr. Tech., PO Box 40, Kjeller N-2027 Norway; [2]Hydro Aluminium R&D Materials Technology, PO Box 219, Sunndalsøra N-6601 Norway

An inverse problem of recovering the heat flux, in a situation where an aluminium block is exposed to a sudden spray of water, is presented. The block is isolated on all sides but one; the water cooled as cast surface. The objective is to gain insight into the water film cooling mechanisms. Several thermocouple sensors are located in the interior of the block and their time history is recorded for several different water qualities and spray nozzle characteristics. A solution to the inverse problem is developed, by using a finite element model and the adjoint functions method combined with a conjugate gradient algorithm. Several different regularization strategies are applied and a connection between the resolution of the recovered heat flux and regularization, sensor locations, and number of sensors is discussed.

2:55 PM
Modelling of Grain Refinement in Directional Solidification: *A. L. Greer*[1]; T. E. Quested[1]; J. E. Spalding[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

The modelling of grain refinement due to Bunn et al. [Light Metals 1998, pp. 963-968] was based on the Maxwell-Hellawell assumption that the melt remains spatially isothermal. A new model is presented for grain refinement in directional solidification where this assumption cannot apply. A simple heat balance is used evaluate the undercooling in the quasi-isothermal solidifying zone. This undercooling determines the grain size through the free-growth criterion in which the potency of an inoculant particle is assumed to be related only to its size. The predictions of the new model are compared with those from the isothermal-melt model and with measured grain sizes in TP-1 and Bridgman samples. Good predictions are obtained for the dependence of grain size on refiner addition level, solute content and cooling rate. The influence of temperature gradient on grain-refiner effectiveness is considered.

3:20 PM Break

3:35 PM
Modelling of Air Gap Development and Associated Surface Macrosegregation in DC Casting of Aluminium Sheet Ingots: *Mohammed M'Hamdi*[1]; Dag Mortensen[2]; Hallvard Fjær[2]; Asbjørn Mo[1]; [1]SINTEF Materials Technology, PB 124 Blindern, N-0314 Oslo Norway; [2]Institute for Energy Technology, PB 40, N-2007 Kjeller Norway

The interaction between thermally induced deformation and heat transfer in DC casting of aluminium often results in the formation of an air gap between the mould and the ingot. The metallostatic head can then force interdendritic liquid through the mushy zone, past the original casting surface, and into the air gap. Such exudation of liquid leads to a solute rich surface layer. A two dimensional mathematical model that quantifies surface macrosegregation due to exudation and solidification shrinkage has been presented elsewhere. A critical parameter of this model is the position of the air gap, and a thermomechanical calculation is needed for an accurate prediction of the development of this gap. For this purpose, the heat transfer calculation being input to the surface segregation model is carried out with an advanced model in which the air gap development is also taken into account by a model addressing the thermally induced deformations in the ingot. Periodic variations in the surface macrosegregation along the casting direction have been reproduced by the models simulating the DC casting of an AA5182 sheet ingot. These variations reflect oscillations in measured mould temperatures that are related to the periodic solidification/melting of the surface shell.

4:00 PM
Thermo-Mechanical Modeling to Predict Shrinkage, Shape and Mold Openings for DC-Cast Rolling Ingots: *Werner E. Droste*[1]; Gerd-Ulrich Grün[1]; Wolfgang Schneider[1]; Jean-Marie Drezet[2]; [1]VAW Aluminium AG, R&D, Georg-von-Boeselager-Str. 25, Bonn, NRW D-53117 Germany; [2]Calcom SA, Parc Scientifique, PSE-EPFL, Lausanne CH-1015 Switzerland

The traditional way to calculate mold openings of DC-cast rolling ingots is based on measured values of shrinkages and interpolation methods. Difficulties arise, when there are no measured values close to the shape and casting speed desired, as extrapolation on this basis does not work. On the other hand todays thermo-mechanical simulation tools for DC-casting are based on physical models. After validation using a given ingot size and casting speed such a model should be capable to predict the correct shrinkage even far away from the validation point at quite different ingot shapes and casting speeds. Here, a 3-D thermo-mechanical model based on the general purpose software Abaqus, which takes into account relevant material behavior, was tested for its ability to predict the ingot shrinkage for large and small ingot sizes and different casting speeds after evaluating the model for a medium size ingot. Resulting ingot shapes are compared with measured data and the influence of ingot size, casting speed and cooling conditions on the accuracy are discussed.

4:25 PM
Microporosity and Other Mushy Zone Phenomena Associated with Hot Tearing: Mohammed M'Hamdi[1]; *Asbjørn Mo*[1]; [1]SINTEF Materials Technology, PB 124 Blindern, N-0314 Oslo Norway

A two-phase continuum model addressing the two main mechanisms associated with hot tearing formation during solidification and subsequent cooling, namely melt feeding of solidification shrinkage and thermally induced deformation, has recently been formulated for the aluminium direct chill (DC) casting process. This mathematical model is in the present paper extended to include also the formation of microporosity because this phenomenon is often considered as related to hot tearing. The modelling equations have been solved for simplified one-dimensional conditions having some relevance for the DC casting process. Modelling results reveal that thermally induced deformations strongly affect the porosity level, and correspondence is pointed out between modelled porosity levels and hot tearing susceptibility in casting trials presented elsewhere.

4:50 PM
The Effects of Water Ejection and Water Incursion on the Evolution of Thermal Field during the Start-Up Phase of the Dc Casting Process: *J. Sengupta*[1]; D. M. Maijer[1]; M. A. Wells[1]; S. L. Cockcroft[1]; A. Larouche[2]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, British Columbia V6T 1Z4 Canada; [2]Alcan International Ltd., Arvida R&D Ctr., 1955 Mellon Blvd., PO Box 1250, Jonquière, Québec G7S 4K8 Canada

The control of the thermal cooling conditions at the start-up phase of the Direct Chill (DC) casting process for aluminum ingots is difficult, and is critical from the standpoint of defect formation. Firstly, boiling water heat transfer governs the secondary cooling experienced by the ingot surfaces as they emerge from the mould. This results in varying rates of heat transfer from the ingot faces as the surface temperature of the ingot changes with time during the start-up phase. Moreover, if the ingot surface temperature at locations below the point of water impingement is high enough to promote film boiling, the water is ejected away from the surface. This can result in a sudden decrease in heat transfer and the formation of local hot spots. Also, the chill water may enter into the gap formed between the ingot base and the bottom block with the evolution of the butt curl. This water incursion alters the heat transfer from the base of the ingot, and in turn affects the surface temperature of the ingot faces. This paper outlines the development of a 2D thermal model using the FEM code ABAQUS™ to study the effects of secondary cooling, water ejection, and water incursion on the evolution of the thermal field in the ingot during the start-up phase. Model predictions have been compared to measurements made on an industrial DC casting machine for validation.

Charles J. McMahon Interfacial Segregation and Embrittlement Symposium: Grain Boundary Segregation and Fracture in Steels

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing & Manufacturing Division, Jt. Mechanical Behavior of Materials, Jt. Computational Materials Science & Engineering, Texture & Anisotropy Committee
Program Organizers: Vaclav Vitek, University of Pennsylvania, Department of Materials Science and Engineering, Philadelphia, PA 19104 USA; Clyde Briant, Brown University, Division of Engineering, Providence, RI 02912 USA; Harvey D. Solomon, General Electric Company, Research & Development Center, Schenectady, NY 12309 USA

Monday PM — Room: 307-308
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Clyde Briant, Brown University, Div. of Eng., Providence, RI 02912 USA; Vasek Vitek, University of Pennsylvania, Dept. of Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

2:00 PM Keynote
Embrittlement Phenomena: Where Have We Been, and Where Are We Going?: *Charles J. McMahon*[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

During the past four decades, I have been blessed with a large number of excellent collaborators: graduate students, post docs, and visiting scientists. Here I want to review some highlights of their work in the following areas: Deformation and brittle fracture of iron, Embrittlement of alloy steels, Hydrogen embrittlement of steels, Surface and interface segregation, Ti-Al alloys, Dynamic embrittlement of steels, Cu-based alloys, and Ni-based alloys. There is still work to be done on the dynamic-embrittlement problem, and I will describe our plans for the next few years.

2:35 PM Invited

Industrial Experiences in Relation to Intergranular Cohesion of Steels: *Hiroo Ohtani*[1]; Masaaki Igarashi[1]; [1]Sumitomo Metal Industries, Ltd., Corp. Rsrch. Labs., 1-8 Fuso-cho, Amagasaki, Hyogo 660-0891 Japan

The demands for high strength low alloy (HSLA) steels are increasing from the viewpoint of social needs, especially environmental conservation. There are, however, various types of intergranular embrittlement encountered during processing, fabricating, and also in service. Some examples of decohesion of boundary related to microstructural characteristics and impurity segregation are presented. (1) Intergranular cracking of cast steel: Surface cracking along austenite (γ) grain boundary of continuous cast slab is mainly due to large γ grain strengthened by alloy-carbonitrides with precipitation free zone or with thin ferrite transformed along the boundary. Rapid cooling followed by re-heating to refine γ grain size, or proper selection of the chemistry to avoid eutectic region whose γ grain size is largest is applied. Impurity segregation enhancing the weakness of grain boundaries is typically observed, for example, in cast 9%Ni steel for LNG storage tank. (2) Grain boundary embrittlement on tempering: Quench-temper is a conventional heat treatment to obtain high strength and toughness. Low temperature embrittlement is believed due to continuous precipitation of cementite and re-distribution of impurities along prior γ grain boundaries. In contrast to martensite structure, low temperature bainite is free from this type of embrittlement, and also shows good resistance to delayed fracture. High temperature temper embrittlement can be essentially eliminated by controlling the chemistry, impurities, and heat treatment. It is worth to know the rapid cooling after tempering causes another problem; dimensional change of the plates after cold forming due to residual stress. The processes that are less sensitive to such kind of embrittlement are presented. (3) Embrittlement due to center segregation of continuous cast slab: Hydrogen induced cracking has been seen in line pipe for transportation of natural gas and crude oil, and in petrochemical plants. Cracking path is usually along the boundary between ferrite and low temperature transformation product such as martensite. In addition to reduction of inclusions and shape control of them, TMCP process has been successfully applied to obtain uniform bainitic structure. (4) Grain boundary cohesion in heat resistant steels: Long-term creep embrittlement and the cracking due to reheating for stress relieving of the weldment have not fully been understood yet in heat resistant steels. They are considered to attribute to decrease in grain boundary strength due to combination of precipitation and growth of carbides formed along the boundary, and the resultant enhancement of impurity segregation into re-formed boundaries. Some evidences of improving these kinds of embrittlement by the alloy elements will be discussed in the presentation. (5) Progress in steel making and analyzing technology: The precise control of chemistry and reduction of impurities in steel making together with the progress in the analyses of trace elements and microstructure of the steels will be presented.

3:10 PM Invited

Segregation Intergranular Fracture and Limits to Formability in Re-Phosphorised Steels: *David Embury*[1]; Kevin Boyle[1]; [1]McMaster University, Dept. of Matls. Sci. & Eng., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

Re-phosphorised steels represent an important method of producing formable steels for automotive applications. Under some conditions they can exhibit cold work embrittlement and this represents the basis of this presentation. The study has employed TEM and EBSP methods to examine the degree of P segregation as a function of heat treatment and the character of grain boundaries. Tests have been performed over a range of temperatures and prestrains to determine the conditions for intergranular fracture. A new model of the fracture process has been developed and applied to forming operations such as creep drawing. The presentation will cover both the fundamental aspects of intergranular fracture and the application of the fracture model to the complex strain paths used in forming operations.

3:45 PM Invited

Effects of Impurity-Element Segregation on Fracture Toughness and Fatigue-Crack Propagation in a 2.25Cr1Mo Steel: *John Frederick Knott*[1]; *Aminul Islam*[1]; Paul Bowen[1]; [1]The University of Birmingham, Sch. of Eng., Elms Rd., Birmingham, Edgbaston B15 2TT UK

The paper describes a number of effects produced by the segregation of phosphorus to grain boundaries. Reversible temper embrittlement is exhibited in fracture toughness tests carried out at both 153K and 77K, due to phosphorus segregation. As-quenched and one-step embrittlement are affected by both the pre-segregation of phosphorus in austenite and by the development of carbides during tempering, concentrating phosphorus by the carbide rejection mechanism. Stress-relief cracking and crack growth at approx. 775K are associated with the stress-driven segregation of sulphur to the region of high triaxial stress ahead of a stressed crack tip. Intergranular facets are observed during fatigue crack propagation in air at room temperature. It is suggested that this is a result of hydrogen uptake from the water vapour present in the air, but that the entry of hydrogen is facilitated by the prior segregation of impurity elements to grain boundaries.

4:20 PM

Quench Embrittlement: A Recently Recognized Mechanism of Intergranular Embrittlement in As-Quenched High-Carbon Steels: *George Krauss*[1]; David K. Matlock[1]; [1]Colorado School of Mines, Adv. Steel Rsrch. Ctr., Golden, CO 80401 USA

A number of intergranular embrittlement phenomena, including tempered martensite, temper, and hydrogen embrittlements, have long been recognized in hardened medium carbon steels. However, gradual characterization of intergranular fracture of as-quenched steels containing 0.5 mass pct carbon has now established another form of embrittlement, termed by the authors, in view of the fact that tempering is not required, as quench embrittlement. The mechanism of this embrittlement has been related to phosphorous segregation and cementite formation at austenite grain boundaries during austenitizing or during quenching after austenitizing. The embrittlement develops even in very low P-containing steels, and persists through tempering temperatures into the first stage of tempering, below temperatures that cause tempered martensite embrittlement. This presentation will describe the mechanisms, thermodynamics, and effects of steel chemistry associated with quench embrittlement, and identify processing approaches which are used to minimize the effects of quench embrittlement.

4:40 PM

Anomalous Fracture Behavior of AISI 4340 Steels Treated at Different Solution Annealing Temperatures: *Jaroslav Pokluda*[1]; [1]Brno University of Technology, Inst. of Physl. Eng., Technicka 2, Brno CZ-616 69 Czech Republic

Values of initial fracture toughness KIc and impact absorbed energy of martensitic ultra-high strength low alloy steels AISI 4340 were found to be inversely dependent on the prior austenite grain size (the solution annealing temperature). The increase in KIc values with increasing grain size can be explained in terms of the crack driving force reduction caused by the intergranularly induced crack tip branching in coarse grained steel grades. The paper shows the results of both the analytical and numerical calculations of this shielding effect and the HRAES and X-ray studies of impurity concentration at grain boundaries. These results are used to elucidate quantitatively the anomalous behavior. Moreover, the occurrence of a dimple intergranular fracture mode in samples treated for the temper embrittlement is qualitatively explained in terms of the Mo, P and C diffusion kinetics.

5:00 PM

Boronizing on Impurity Controlled Steels: C. Bindal[2]; *A. H. Ucisik*[1]; [1]Sakarya University, Dept. of Metlgcl. & Matls. Eng., Adapazari-Sakarya Turkey; [2]Bogazici University, Dept. of Matls. Inst. of Biomed. Eng., Saribal S. 40, Ortakoy-Istanbul 80840 Turkey

Boronizing being one of the simplest methods used for boride type ceramic coating has extensively applied by many researchers on commercial alloys to increase surface hardness, to obtain a layer resisting wear and chemical attacks. In this study, boronizing process was applied on impurity controlled steels of which all embrittlement problems have been solved by Professor Charles J. McMahon, Jr. Alloys with and without Mn and Cr had 0.02% P. Mn and Cr levels were 0.5, 1.0 and none. Only one alloy had both 0.5% Mn and 0.5% Cr. Boronizing bath consisted of Borax, Boric Acid and Ferro-Silicon. All alloys were boronized at 940°C for between 0.5hrs and 10hrs. After boronizing, each group of alloy was examined by using classical metallographic techniques, X-ray diffraction, hardness measurements, fracture toughness measurements, Scanning Electron Microscopy and Corrosion tests. It was revealed that hardness level on the layer depends on alloy and boronizing time and coated material are divided into three parts; (i) layer having borides, (ii) the region where boron makes solid solution, (iii) matrix. In addition to iron borides mangenese and chromium borides formed on the surface depending on alloying elements. Mangenese is the only element increasing fracture toughness. In severe conditions all boride layers showed good corrosion resistance.

Computational Modeling of Materials, Minerals & Metals Processing: Monday PM Plenary Session 2:00 PM – 2:45 PM

Sponsored by: Materials Processing & Manufacturing Division, *Program Organizer:* Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Monday PM Room: 619-620
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chair: Vaughan Voller, University of Minnesota, Saint Anthony Falls Lab., Mississippi River at Third Ave., Minneapolis, MN 55414-2196 USA

Keynote

Modelling and Process Optimization for Functionally Graded Materials: *Dan Tortorelli*[1]; [1]University of Illinois-Urbana, Dept. of Mechl. & Indl. Eng., 350 MEB, MC 244, 1206 W. Green, Urbana, IL 61801 USA

We optimize continuous quench process parameters to produce functionally graded aluminum alloy extrudates. To perform this task, an optimization problem is defined and solved using a standard nonlinear programming algorithm. Ingredients of this algorithm include 1) the process parameters to be optimized, 2) a cost function: the weighted average of the precipitate number density distribution, 3) constraint functions to limit the temperature gradient (and hence distortion and residual stress) and exit temperature, and 4) their sensitivities with respect to the process parameters. The cost and constraint functions are dependent on the temperature and precipitate size which are obtained by balancing energy to determine the temperature distribution and by using a reaction-rate theory to determine the precipitate particle sizes and their distributions. Both the temperature and the precipitate models are solved via the discontinuous Galerkin finite element method. The energy balance incorporates nonlinear boundary conditions and material properties. The temperature field is then used in the reaction rate model which has as many as 105° of-freedom per finite element node. After computing the temperature and precipitate size distributions we must compute their sensitivities. This seemingly intractable computational task is resolved thanks to the discontinuous Galerkin finite element formulation and the direct differentiation sensitivity method. A three-dimension example is provided to demonstrate the algorithm.

Computational Modeling of Materials, Minerals & Metals Processing: Track A - Optimization & Novel Methods

Sponsored by: Materials Processing & Manufacturing Division, *Program Organizer:* Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Monday PM Room: 619
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chair: Dan Tortorelli, University of Illinois-Urbana, Dept. of Mechl. & Indl. Eng., Urbana, IL 61801 USA

2:45 PM

Lattice Boltzmann Methods for Metallurgical Process Simulation: *Christian Redl*[1]; [1]Mining University Leoben, CD-Lab. Appl. Comptnl. Thermofluid-dyn., Franz-Josef-Strasse 18, Leoben 8700 Austria

The Lattice Boltzmann Method (LBM) solves fluid dynamics problems based on a discrete mesoscopic approach. It is based on the Lattice Boltzmann equation which is a special discretisation of the continuous Boltzmann equation. Its principle advantage is that it can handle complex physical phenomena (like interaction between different components and surface effects), complicated boundary and initial conditions very well, free from many gridding and stability constraints that plague conventional numerical methods used for fluid flow simulations. The LBM algorithm has a simple structure and acts locally, which is favourable for parallel computing. Complex structures like porous media can be resolved directly. Its potential for metallurgical process simulations is demonstrated in the present study which is concerned with the simulation of the flow in a copper winning electrolysis cell. The electrolyte is modelled as the carrier fluid and the oxygen is considered via a so called active scalar equation. The modelling of buoancy, momentum exchange and the free surface requires special effort. The results of the simulation are validated against experimental data obtained by Laser-Doppler-Anemometry. Despite the simplifications made, good agreement was found.

3:10 PM

Viscosity Estimation Model for an Oscillating Cup Viscometer: *Deming Wang*[1]; R. A. Overfelt[1]; [1]Auburn University, Dept. of Mechl. Eng., 201 Ross Hall, Auburn University, AL 36849 USA

Viscosity measurements of molten alloys become more and more important in modern metallurgy engineering. Lack of the viscosity data of many new alloys hampers many CAD computer codes to apply in casting manufactures. The oscillating cup viscometer has become a dominant technique to measure the viscosities of high temperature molten metals. Unfortunately, the viscosity estimation model from the observed logarithmic decrement and period of the oscillation is very complicated. There are still large discrepancies of viscosity estimation values between using different measurement facilities or using different viscosity estimation models for a same molten metal. The purpose of the paper is to evaluate the accuracy of an oscillating cup viscometer in Auburn University. Two well known viscosity estimation models, Roscoe's and Torklep's equations, are discussed and compared viscosity for different alloys. The theoretical literature for the fluid flow inside an oscillating cup is reviewed and a more accurate working equation for Auburn oscillating cup viscometer is developed. Some design parameters of the oscillating cup viscometer which also directly affect the accuracy of viscosity estimation by using the working equation are discussed. In addition, applications and experimental measuring data are presented in the paper for several different commercial alloys, such as aluminum alloys: A319, A356 and A201, nickel-base super alloys: In713 and In718, and casting irons, C40 Gray Iron and Ductile Iron.

3:35 PM Break

3:50 PM

Numerical Optimization of Magnesium Reduction in a Modified Pidgeon Process: *Alfred Yu*[1]; *Henry Hu*[2]; [1]Nanjing Welbow North America Office, 601-969 Felix Ave., Windsor, Ontario N9C 4C7 Canada; [2]University of Windsor, Mechl. & Matl., 401 Sunset, Windsor, Ontario N9B 3P4 Canada

A numerical model of heat and mass transfer in the retort was set up to simulate and optimize the reduction phenomena in the process of Pidgeon magnesium reduction. The simulations were run to determine the effect of varying processing parameters on the magnesium reduction time. The model predicted the temperature distributions, the heating curves, the recovery ratio of magnesium, and the total process time. The predictions were used to optimize the magnesium reduction process, the dimensions of retort, the shapes of materials, and reaction cycle. Demo operation shows that, with application of the optimizations, significantly production capacity increases in the same furnace, reduction period decreases, energy consumption decreases.

4:15 PM

Deterministic and Regression Models of Nickel Oxide Reducing Roasting Process: V. M. Paretsky[1]; *A. V. Tarasov*[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., 129515 Moscow Russia

A mathematical model of the low-temperature (soft) nickel oxide reduction (SNOR) in a tubular kiln has been developed including an equation describing changes in the nickel oxide content of the solid material along the length of the kiln; an equation describing the changes in the content of reagents ensuring nickel reduction (i.e., hydrogen and carbon monoxide) in the gas phase along the kiln length; as well as the temperature along the kiln length. The kinetic relationships used for the development of the model had been determined based on experiments conducted specially for this purpose. Model investigations were conducted to determine the distribution of such parameters along the length of the kiln as the contents of metals oxides in the solids, partial pressure of hydrogen and carbon monoxide, variations of the temperature depending on the SNOR process conditions, including the kiln rotation speed. Based on the data obtained, a simplified regression model of the reducing roasting process was developed.

4:40 PM

Integrating Computational Mechanics and Numerical Optimization for the Design of Material Properties in Electronic

Packages: *Stoyan Stoyanov*[1]; C. Bailey[1]; M. Cross[2]; [1]University of Greenwich, Sch. of Compg. & Mathl. Sci., Greenwich, London SE10 9LS UK; [2]The University of Greenwich, Ctr. for Numl. Modlg. & Proc. Analy., Old Royal Naval College, Greenwich, London SE10 9LS UK

Silicon components containing transistor circuitry are at the heart of electronic products such as computers, mobile phones, etc. These components are connected to printed circuit boards (PCB's) using solder material that acts as the conductor for both electricity and heat. During the lifetime of a product it will undergo many thermal cycles where the chip becomes hot during product operation and then cools when the product is switched off. At the product design stage engineers undertake thermo-mechanical simulations to predict the thermal stress in the solder joints due to this thermal cycle and to ensure that the stress magnitude will not result in early fatigue-type failure. The design engineer's aim is to identify component parameters which ensure that the solder will survive beyond a certain number of thermal cycles. The aim of integrating computational mechanics and optimization tools together is to speed up dramatically the design process. From viewpoint of a design engineer in the electronics manufacturing sector, these tools can be used to quickly estimate key design parameters (i.e. material properties, product dimensions, etc.) that will guarantee the required product performance. In this paper a modeling approach coupling computational mechanics techniques with numerical optimization is presented and demonstrated. The integrated modeling framework is obtained by coupling the multi-physics framework–PHYSICA–with the design optimisation tool–VisualDOC. Thermo-mechanical simulations are presented that predict the creep strains in solder material. Different numerical optimization procedures: Direct and Response Surface based optimization plus Design of Experiments, are tested as a part of this modeling framework.

5:05 PM
Computer-Aided Modeling and Control of Autogenous Copper Smelting Process: *A. V. Tarasov*[1]; V. M. Paretsky[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., 129515 Moscow, Russia

Based on theoretical and experimental studies into the specific features of the mechanism, kinetics, aerodynamics and heat transfer parameters of the flame-type oxidation of metal sulfides in a stream of technical-grade oxygen, a three-dimensional deterministic mathematical model of an oxygen-flame smelting furnace has been developed on the basis of the zonal method for computation of heat exchange. The model is presented in the form of equations taking into consideration the radiation component of the selective radiation, the convection component of mass transfer between the space and surface zones, heating of charge particles due to heat conductivity, accretions formation on a multi-layer refractory lining with inserted cooling elements. The adaptation of the model for a full-scale furnace has demonstrated the possibility for its use for computation of main smelting process parameters. Multifactor model investigations were conducted and the results obtained were applied using statistical methods for development of a simplified computational regression model describing the interrelation of the main output parameters: temperatures of flame, lining, slag and off-gas; heat fluxes to the lining; thickness of accretions; heat removed by cooling elements; copper content of matte. On the basis of this model, a block diagram of the algorithm was developed for control of the thermal conditions of the furnace, as well as the control algorithm itself.

Computational Modeling of Materials, Minerals & Metals Processing: Track B - Melting & Solidification - I

Sponsored by: Materials Processing & Manufacturing Division, *Program Organizer:* Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Monday PM — Room: 620
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Vaughan Voller, University of Minnesota, Saint Anthony Falls Lab., Minneapolis, MN 55414-2196 USA; Matt Krane, Purdue University, Dept. of Matls. Eng., W. Lafayette, IN 47907 USA

2:45 PM
A Marker Chain Front Tracking Method for Modelling Meniscus Dynamics in the Al Ingot Casting Process: *Fionn Iversen*[1]; Jon Arn Bakken[1]; Stein Tore Johansen[1]; [1]Norwegian Institute of Science and Technology (NTNU), Matls. Tech. & Electrochem., A. Getz vei 2B, Trondheim N-7491 Norway

In conventional direct chill (DC) hot-top casting of aluminium extrusion ingot using 'gas-slip', poor surface quality of the cast ingot may appear. It is believed that these defects are related to instabilities such as periodic oscillations or folding of the meniscus (the interface between liquid metal and gas in the mould). The object of this work is to develop a stable and reliable model for simulation of the meniscus dynamics. A new 2D cylinder symmetric front tracking model has been developed for the meniscus propagation, and is implemented in a general Navier Stokes solver. The model is based on a finite volume cubic spline marker chain technique. The advantages of the model is its applicability to cases with large density ratios and its ability to calculate independent velocity fields for phases on separate sides of an interface, thereby making it possible to model the dynamics of thin films with a thickness scale smaller than the typical grid size. Effects of surface tension and wetting are also easily applied in the marker chain model. The model is applied to the DC casting process. Results are compared with data from plant test runs and suggestions are made on how to improve the casting process.

3:10 PM
Computational Modeling of Heat Mass and Solute Transport in Directional Solidification Processes: *Mohammed El Ganaoui*[1]; Patrick Bontoux[2]; [1]Université de Limoges, Phys./Numcl. Mod., 123 Albert Thomas, Limoges 87000 France; [2]CNRS, Numcl. Mod., IRPHE, Marseille, 13451 France

During directional solidification, absorption or rejection of latent heat or solute by the solidification front induce convective flows in the liquid phase. These convective motions affect heat and mass transfer in the vicinity of the solidification interface and is subject to many studies. In this work a numerical approach is presented. To avoid remeshing needed by front tracking methods a time-dependent homogeneous formulation is considered to verify implicitly thermal and solutal Stefan conditions at the interface. The numerical solution is based on finite volume approximation. The previous work show that the present method describe accurately the hydrodynamic transition and the interaction with the solid liquid interface in the case of pure material. This study focuses on the occurrence of solutal convection in gradient freezing applications. In a first step only the fluid phase is investigated and the results are validated with respect to spectral ones. In a second step the full solid/liquid model is investigated. A linear approximation of the equilibrium phase diagram is considered for establishing relations between mass fraction and temperature fields to close the set of conservation equations. The present work shows that the method is able to describe with accuracy close to spectral one complex phenomena occurring in reduced configuration to fluid phase. The global model with solid phase account correctly the interface displacement and its interaction with solutal field.

3:35 PM Break

3:50 PM
Computational Modelling of Freeze Layers in Smelting Processes: *Andrew P. Campbell*[1]; Koulis A. Pericleous[1]; Mark Cross[1]; [1]University of Greenwich, Ctr. for Numl. Modlg. & Proc. Analy., 30 Park Row, Greenwich, London SE10 9LS UK

The use of computational modelling in examining process engineering issues is very powerful. It has been used in the development of the HIsmelt® process from its concept. It is desirable to further water-cool the HIsmelt® vessel to reduce downtime for replacing refractory. Water-cooled elements close to a metal bath run the risk of failure. This generally occurs when a process perturbation causes the freeze and refractory layers to come away from the water-cooled element, which is then exposed to liquid metal. The element fails as they are unable to remove all the heat. Modelling of the water-cooled element involves modelling the heat transfer, fluid flow, stress and solidification for a localised section of the reaction vessel. The complex interaction between the liquid slag and the refractory applied to the outside of the water-cooled element is also being examined to model the wear of this layer. The model is being constructed in Physica, a CFD code developed at the University of Greenwich. Modelling of this system has commenced with modelling solidification test cases. These test cases have been used to validate the CFD code's capability to model the solidification in this system. A model to track the penetration of slag into refractory has also been developed and tested.

4:15 PM
Mathematical Modeling of Heat Transfer and Microporosity Formation in Die Cast A356 Wheels: P. Vo[1]; *D. Maijer*[1]; S. L. Cockcroft[1]; M. A. Wells[1]; C. Hermesmann[2]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., Vancouver, British Columbia V6T 1Z4, Canada; [2]Canadian Autoparts Toyota, Inc., 7233 Progress Way, Delta, British Columbia V4G 1E7, Canada

Die cast aluminum wheels are one of the most difficult automotive castings to produce because of stringent cast surface and internal quality requirements. A mathematical model has been developed to predict heat transport and porosity formation in die cast A356 wheels as part of a collaborative research agreement between researchers at the University of British Columbia and Canadian Auto Parts Toyota, Incorporated. The heat transfer model, employing the commercial finite element code ABAQUS, is a three-dimensional, 30° slice of the wheel and die that describes forced air cooling, natural convection of the die to the surrounding environment, and interfacial heat transport between the wheel and die sections. Extensive temperature measurements in the die and in the wheel taken over several cycles in the casting process were used to fine tune and validate the model. Preliminary work on predicting porosity formation focused on using the Niyama parameter as a measure of the probability of porosity. The latest version of the model incorporates a new more fundamentally based porosity criterion, which takes into account the effect of hydrogen and inclusion content. The development of this model together with some early results will be presented.

4:40 PM
Modelling Filters in Metal Casting Processes: *Mark R. Jolly*[1]; Jean-Christophe Gebelin[1]; [1]The University of Birmingham, IRC in Matls., Edgbaston, Birmingham, W. Midlands B15 2TT UK

A number of different types of so-called filters are used on the metal casting industries to impart some cleaning effect and flow control on the liquid metal as it passes through them. The filters range from simple planar meshes through extruded channels to reticulated foam structures. It is most common that software packages used in the industry model the filters by a simple pressure drop associated with some area fraction and permeability parameters. Recent experimental work at the IRC in Birmingham has shown that filters of the same type can behave very differently depending upon the casting process in which they are employed. Modelling filter geometries for a range of different casting processes has indicated that the flow of metal and heat losses through the filters are rather complex and should be considered when using filters in the casting processes. This paper will present a number of cases of different types of filters modeled and different processes and indicate some of the sensitivities of the processes to boundary conditions imposed by the process.

5:05 PM
Computer Heat Transfer Model for Directionally Solidified Castings: *Deming Wang*[1]; R. A. Overfelt[1]; [1]Auburn University, Mechl. Eng. Dept., 201 Ross Hall, Auburn University, AL 36849 USA

Thermal transfer control is very important in directionally solidified (DS) castings. This paper presents a simple and efficient computer-aided heat transfer simulation method to predict the thermal characteristics of an alloy sample in a special furnace for directional solidification. A two-dimensional transient heat transfer by radiation combined with conduction is developed to calculate the energy exchange between the symmetric furnace and the sample. A control volume technique is used to obtain a set of highly efficient finite difference equations for heat conduction and heat radiation with changeable view factors. The model well simulates the transient process of DS castings. The simulation results are verified by a few measurable experimental results. Using the two-dimensional computer simulation model, many thermal properties of the samples can be obtained, such as temperature distribution, solidification velocity, the shapes and positions of the liquid/solid interface and thermal gradient at the inter faces. These are very important to analyze microstructure of DS casting alloy, avoid casting defects and control the quality of DS castings.

Computational Phase Transformations: Atomic Level Modeling of Interface Motion and Phase Transformations

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Jt. Computational Materials Science & Engineering, Thermodynamics & Phase Equilibria Committee
Program Organizers: Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; Mark Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; James Aaron Warren, NIST, CTCMS and Metallurgy Division, Gaithersburg, MD 20899-8554 USA

Monday PM — Room: 201
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Jeff Rickman, Lehigh University, Matls. Sci. & Eng., #5 Whitaker Lab., 5 E. Packer Ave., Bethlehem, PA 18105-3195 USA

2:00 PM
Impurity Effects on Interface Migration: *David J. Srolovitz*[1]; Mikhail I. Mendelev[1]; [1]Princeton University, Princeton Matls. Inst., Princeton, NJ 08544 USA

The migration of interfaces is central to all first order phase transformations and many types of coarsening phenomena. Even in high purity materials, the presence of impurities can dramatically effect how interfaces move. In this presentation, we will discuss the effects of impurities on moving interfaces both from the perspectives of a discrete, Ising-like model and continuum theory. We begin by reviewing the classical continuum theories of Cahn and Lücke and Stüwe and compare their predictions with the results of computer simulations. While these theories capture much of the physics, they do not lead to quantitatively reliable predictions. This is not surprising since the fundamental events associated with interface migration occur via atomic-scale mechanisms, which the impurities can modify. A more quantitative theory is presented based on the details of the atomic scale mechanism. Finally, we examine the effects of co-segregation on interface mobility. A number of new phenomena have been observed, including the identification of conditions under which increasing the impurity concentration can increase the interface mobility.

2:45 PM
Phase Transitions and Dynamical Behavior in Perovskite Ferroelectrics by Molecular-Dynamics Simulation: *Simon R. Phillpot*[1]; Marcelo Sepliarsky[1]; Marcelo Stachiotti[2]; Ricardo Migoni[2]; [1]Argonne National Laboratory, Matls. Sci. Div., Bldg. 212, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Instituto de Fisica Rosario, CONICET-UNR, Rosario Argentina

We use molecular-dynamics simulations to explore two aspects of the phase behavior and dynamical behavior of ferroelectric perovskites. In superlattices consisting of equal thickness layers of a perovskite ferroelectric (KNbO3) and a perovskite paraelectric (KTaO3) we find that the behavior is determined by the strength of the coupling between the KNbO3 layers, mediated by induced polarizations in the KTaO3 layers. Our results are consistent with recent experiments on this system. We also use molecular-dynamics simulations to explore the atomic-level details of the dynamics of polarization reversal in the tetragonal phase of a monodomain order-disorder perovskite ferroelectric. We find that the transition from an [001] orientation to an [00-1] orientation involves polarization rotation through intermediate states with polarization parallel to [011] and [01-1], accompanied by a strong transient piezoelectric response. This work was supported by the US Department of Energy, Office of Science under Contact W-31-109-Eng-38, by the DOE S&P Center Project on Nanoscale Phenomena in Perovskite Thin Films, and by CONICET-Argentina.

3:15 PM
Dynamical Self-Organization in Alloys Driven by Irradiation or Plastic Deformation: *Pascal Bellon*[1]; Jiwen Liu[1]; Raul A. Enrique[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA

The microstructure of alloys driven away from equilibrium by ion irradiation or by plastic deformation can undergo spontaneous organization. In particular, as a result of the competition between external driving forces and internal relaxation processes, the composition field can develop patterns with a characteristic, finite length. In order to determine the conditions required to trigger such a self-organization, and to understand the wavelength selection process, we have developed kinetic Monte-Carlo simulations and continuum kinetic models. The main results is that the external driving force has to introduce a new length scale, different from that of the thermal relaxation process for patterning to take place, and that the structure of the external noise (external fluctuations) can play an important role in the wave length selection. Examples will be given on alloys that would undergo phase separation (e.g., Cu-Ag) or phase ordering and phase separation (e.g., Ni-Al) at equilibrium.

3:45 PM Break

4:00 PM
Atomistic Computations of Thermodynamic and Kinetic Properties of Solid-Liquid Interfaces: *Jeff J. Hoyt*[1]; Mark Asta[2]; Alain Karma[3]; [1]Sandia National Laboratories, PO Box 5800, MS 1411, Albuquerque, NM 87185-1411 USA; [2]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [3]Northeastern University, Phys., 360 Huntington Ave., Boston, MA 02115 USA

The shape and velocity of a dendrite formed during solidification of an alloy depend critically on several thermodynamic and kinetic properties of the solid-liquid interface. In this talk we will review several molecular dynamics and Monte Carlo simulation techniques which can accurately determine many of the important interfacial properties. In each case the embedded atom method is used to model the interatomic potential and results are reported for Ag, Au, Cu, Ni and Pb and the alloys Al-Cu and Cu-Ni. The solid-liquid interfacial free was determined using the fluctuation spectrum method, a technique which is capable of resolving the very small anisotropy. The individual excess entropy and excess energy contributions to the interfacial energy will also be reported. The kinetic coefficient was found by monitoring the velocity of a planar solid-liquid interface as a function of undercooling for the three low index growth directions 100, 110 and 111. Simulation results will be compared to the crystallization rate model of Burke, Broughton and Gilmer. For the binary alloys we have computed the relative absorption of solute to the solid-liquid boundary using Monte-Carlo and thermodynamic integration techniques. Finally, the segregation coefficient as a function of velocity for Al-Cu will be presented and the results will be discussed in terms of various theoretical models and experiment.

4:30 PM
Monte-Carlo Simulations of Phase Separation in Binary Alloys: Peter Fratzl[1]; *Richard Weinkamer*[1]; Himadri Gupta[2]; Joel L. Lebowitz[2]; [1]Austrian Academy of Sciences and University of Leoben, Erich Schmid Inst. of Matls. Sci., Jahnstrasse 12, Leoben 8700 Austria; [2]Rutgers University, Busch Campus, Depts. of Math. & Phys., New Brunswick, NJ 08903 USA

Using three-dimensional atomistic Monte-Carlo simulations, we have studied phase separation in binary alloys with and without lattice misfit between precipitates and matrix. The elastic interactions are modeled by springs connecting the atoms. Our investigations concentrated on the influence of the misfit on the morphology of the precipitates, their size distribution and growth kinetics. Introducing a slight elastic inhomogeneity into the model, an additionally applied external stress resulted in directional coarsening ("rafting") of the precipitates. Neglecting elastic interactions, we have also studied the influence of vacancies and their effective interaction with the different atomic species on the phase separation kinetics. We found that when the vacancy is preferentially attracted to the precipitates, coarsening does not proceed via the usual evaporation-condensation mechanism, but according to a coagulation mechanism of moving precipitates.

5:00 PM
A Semi-Empirical Atomic Potential for Atomistic Approach to Phase Transformation: *Byeong-Joo Lee*[1]; [1]KRISS, Matls. Evaluation Ctr., Yusong PO Box 102, Taejon 305-600 Korea

Atomistic Simulation can be a useful tool to analyze and predict phase transformations, crystallographic changes and mechanical properties of metals. In order to obtain reliable results within reasonable computing time, it is important to use a well-assessed semi-empirical atomic potential for elements and alloy systems. In the present presentation, a recently developed atomic potential model, the second nearest-neighbor modified embedded atom method (MEAM), will be introduced. Various physical properties of elements (mainly bcc transition metals), including elastic constants, structural properties, point defect properties, surface properties and thermal properties will be calculated and compared with experiments or high level calculations. Some examples for description of alloy systems, and an atomistic approach to recrystallization will also be presented.

Creep Deformation: Fundamentals and Applications: Fundamental Behavior - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing & Manufacturing Division, Jt. Mechanical Behavior of Materials, Powder Materials Committee
Program Organizers: Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA; Sai V. Raj, NASA Glenn Research Center at Lewis Fields, Cleveland, OH 44135 USA

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February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: M. E. Kassner, Oregon State University, Dept. of Mech. Eng., 414 Rogers Hall, Corvallis, OR 97331 USA

2:00 PM Invited
Rate Controlling Processes for Five Power-Law Creep: *M. E. Kassner*[1]; S. C. Bergsma[2]; [1]Oregon State University, Dept. Mechl. Eng., Rogers Hall, Corvallis, OR 97331 USA; [2]Northwest Aluminum Company, 3313 W. Second St., The Dalles, OR 97058 USA

The mechanisms for recovery-controlled-creep in pure and Class M (pure metal behavior) alloys at elevated temperature, usually within the five-power-law creep regime, will be discussed. Specifically, the roles of the dislocation substructure and the so-called internal stresses on the rate controlling mechanisms will be described in terms of recent TEM with convergent beam electron diffraction, in-situ TEM and dislocation modeling techniques.

2:25 PM Invited
Creep Behavior at Very Low Stresses: *Farghalli A. Mohamed*[1]; [1]University of California, Cheml. Eng. & Matls. Sci., 916 Engineering Tower Bldg., Irvine, CA 92697-2575 USA

This paper reviews recent information on two Newtonian deformation processes, Nabarro-Herring creep and Harper-Dorn creep, that may control the creep behavior of materials at very low stresses. In particular, the paper focuses on discussions and results related to the requirements for the occurrence of both deformation processes during the high-temperature deformation of materials.

2:50 PM
The Effects of H_2SO_4 on the Mechanical Behavior of Ice Single Crystals: *Ian Baker*[1]; Y. L. Trickett[1]; X. Li[1]; D. Iliescu[1]; P. M.S. Pradhan[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

Tests were performed in compression at -20°C (0.93 T_m) at a variety of strain rates on three different orientations of ice singles with and without sulfuric acid (a naturally-occurring contaminant). Unlike the effect of impurities on most materials, the H_2SO_4 (as little as 100 p.p.b.) dramatically decreased both the peak stress and the subsequent flow stress of the ice single crystals at all strain rates. The stress exponent was determined to be ~2 and was unaffected by the dopant. The peak strength softening was shown to be proportional to the square root of the concentration of sulfuric acid. Synchrotron x-ray topography showed that the doped crystals had a grown-in dislocation density of 1.0 X 10^{10} m^{-2}. Tests were also performed at -10°C and -30°C for a single orientation and on ice from Byrd Station, Antarctica. Research supported by NSF grant OPP-9980379 and ARO grant DAAD 19-00-1-0444.

3:10 PM
A Framework for Modeling Creep: *Holger Brehm*[1]; *Michael J. Mills*[1]; *Glenn S. Daehn*[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA

We propose a method of modeling creep that couples traditional equations for dislocations overcoming obstacles in a thermally activated manner with those for diffusion-controlled microstructural coarsening, and microstructural refinement due to plastic flow. We find that with this model, over a wide range of input parameters, we can re-

cover: a steady-state stress exponent near 5, a constant structure stress exponent which is much higher, a steady-state creep activation energy that is very close to that of diffusion, and the steady-state inter-obstacle spacing (which scales with subgrain size) which is nearly inversely proportional to the applied stress. One of the attractive features of this model is that it allows interrogation of the separate phenomena (slip, refinement and coarsening) that are essential to the creep process.

3:30 PM
Departures from Model Predictions for Creep in Pure Metals at Low Stresses Near their Melting Temperature: *Kevin Robert McNee*[1]; Vivek Srivistava[1]; Geoffrey Winston Greenwood[1]; Howard Jones[1]; [1]University of Sheffield, Dept. of Eng. Matls., Mappin St., Sheffield S1 3JD UK

Early experiments that verify the diffusional creep rate predictions of Nabarro and Herring were undertaken just below the melting temperature and on small diameter wires and thin foils with through thickness grains. Results were shown to give reasonable agreement with predictions at sufficiently long test durations but specimens of other geometries and grain shapes have shown more variability. Recently some of these results have been re-interpreted and the applicability of the Nabarro-Herring creep mechanism to them has been questioned. Further experiments have been carried out in the present work on a number of pure metals to determine possible sources of the experimental diversity and to re-evaluate operative mechanisms.

3:50 PM Break

4:00 PM Invited
Deformation Anomalies during Creep of Titanium Alloys: Matthew Brandes[1]; Thirumalai Neeraj[1]; Michael F. Savage[2]; *Michael J. Mills*[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]National Institute of Standards and Technology, Metall. Div., Matls. Perfor. Grp., Gaithersburg, MD 20877-8550 USA

Titanium alloys are attractive structural materials because of their high specific strength and excellent corrosion resistance. It has been recognized for some time, however, that Ti alloys must be employed conservatively because of their tendency to creep significantly at room temperature, even at stresses well below the macroscopic yield strength. This presentation will describe two additional, anomalous characteristics of these alloys under these conditions: (a) a distinct tension/compression (T/C) asymmetry and (b) dramatic recovery of strain hardening at room temperature. We will show that a T/C asymmetry is associated with deformation via a-type dislocations, and attempt to rationalize this finding via detailed examination of the core structure of these dislocations using high resolution TEM investigation. The room temperature recovery phenomenon appears to be correlated with the presence of dislocation pile-ups in the alpha-phase of these alloys, as revealed by diffraction-contrast TEM studies. The possible implications of these effects with respect to designing with Ti alloys will be discussed.

4:25 PM Invited
On the Mechanisms Controlling Steady State Deformation of Metals and Alloys: *Erik Nes*[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., N-7491, Trondheim, Norway

The fundamental problems of work hardening and steady state deformation relate to: (i) How to calculate the flow stress at a constant microstructure? (ii) How to define the mechanism of athermal storage of dislocations? And (iii) how to treat dynamic recovery? These are difficult questions, which have stimulated extensive research efforts over many decades. In an effort to reach an improved understanding, a new approach towards these problems has recently been proposed by the present author and co-workers. The model presented is based on a statistical analysis of athermal storage of dislocations. By combining the solution for the dislocation storage problem with models for dynamic recovery of network dislocations and sub-boundary structures, a general internal state variable description is obtained. The model includes effects due to variations in: stacking fault energy, grain size, solid solution content, and particle size and volume fraction. The result is a work hardening model, which in principle is capabl e of providing the stress-strain behavior for a given metal or solid solution alloy under condition ranging from deformation in the ambient temperature range to high temperature creep. In this presentation, the basic model concepts will be discussed and contrasted in relation to alternative interpretations found in the creep literature.

4:50 PM
Recent Advances in Modeling Creep Deformation and Damage: *James C. Earthman*[1]; [1]University of California-Irvine, Chem. & Biochem. Eng. & Matl. Sci., CBEMS, Irvine, CA 92697-2575 USA

Recent research accomplishments in the area of modeling concomitant creep processes and associated damage will be reviewed. Numerical models will be discussed which simulate different creep mechanisms for a range of materials and conditions. Experimental evidence will also be discussed which is particularly insightful for modeling the development of creep damage. For example, data for specimens tested under multiaxial stresses will be presented that are indicative of the principal damage mechanism. Results will be compared and contrasted in order to identify new insight and future research directions that will lead to better methods for predicting creep failure and improved materials performance at elevated temperatures.

5:10 PM
Equation for High Temperature Creep Rate of Pure Metals and Solid Solutions with Complete Structural Factor: *Alexander Illich Dekhtyar*[1]; [1]Kurdyumov Institute of Metal Physics of National Academy of Science of Ukraine, Div. of Strength & Plasticity of Inhomogeneous Alloys, 36 Vernadsky Blvd., Kiev, Kiev Region 03680 Ukraine

The complete structure factor has found for equation of high temperature creep rate of pure metals and dilute solid solutions with FCC and BCC lattice. This factor involves the structural parameters of different structure scales: electronic and atomic structure, inside structure of individual dislocations, dislocation structure including dislocation distribution, grain structure. The structure factor includes the fundamental values of metals, such as atomic mass, melting point, shear modulus. The expression for the structure factor was deduced on the basis of experimentally found for a lot of metals and alloys strong dependence of creep rate on the widening of the reciprocal lattice poles. The reciprocal lattice pole widening is determined by X-ray diffraction investigations with high statistics as reflex widening in azimuthal direction. Obtained equation is proved both for primary and steady state creep stages.

5:30 PM
Interaction Between Dislocations during High Temperature Creep Process: *Oleksandr Illich Dekhtyar*[1]; [1]Kurdyumov Institute of Metal Physics of National Academy of Science of Ukraine, Div. of Strength & Plasticity of Nonhomogeneous Alloys, 36 Vernadsky Blvd., Kiev, Kiev Region 03142 Ukraine

Interaction forces between pairs of straight dislocations in BCC lattice have calculated for the case of high temperature creep deformation. It was done for different combinations of screw, edge and mixed dislocations with different Burgers vectors in different slip planes. These forces were compared with the forces acting under external stresses on the dislocation straight parts determined experimentally by the electron transmission microscopy in single crystalline molybdenum undergone creep deformation. It is found that screw dislocations do not accumulate in subboundaries and all of dislocations do not intersect during creep process at low applied stresses. At high applied stresses dislocations might mutually intersect to create the jog concentration enough for realization the mechanism of intensive collective climb within subboundaries.

David L. Davidson Symposium on Fatigue: Fatigue Thresholds and Life Prediction

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Jt. Mechanical Behavior of Materials, High Temperature Alloys Committee
Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; Thomas Zogas, Carpenter Technology Corporation, Reading, PA 19612-4662 USA

Monday PM Room: 208
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Peter K. Liaw, University of Tennessee, Matls Sci. & Eng., Knoxville, TN 37996-2200 USA; Robert O. Ritchie, University of California-Berkeley, Matls. Sci & Eng., Berkeley, CA 94708-1238 USA

2:00 PM Keynote
High-Cycle Fatigue in Turbine Engine Alloys: *R. O. Ritchie*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94708-1238 USA

High-cycle fatigue (HCF) is a prime cause of military aircraft turbine engine failures. It results from fatigue-crack growth in blades and disks, initiated at small defects often associated with fretting or foreign object damage. Due to the high frequencies (>1 kHz) involved, design based on a HCF threshold would appear to be a preferred approach. In this work, the nature of the fatigue threshold is examined under representative high frequency and high load-ratio conditions, in a Ti-6Al-4V blade alloy with bimodal and lamellar microstructures, with emphasis on behavior following foreign-object damage (FOD) and under mixed-mode (modes I + II) loading conditions. It is shown that for all crack sizes, large or small, but of dimensions large compared to microstructural size-scales ("continuum-sized" cracks), a worst-case fatigue threshold can be defined (at load ratios where R approaches unity such that crack closure is minimized) which represents a lower-bound stress intensity for fatigue-crack growth. This holds for mixed-mode loading (studied over a wide range of mode-mixities at load ratios from 0.1 to 0.8), provided the threshold is characterized under worst-case mode I conditions in terms of the strain energy release rate. However, for crack sizes comparable with microstructural dimensions, i.e., < 10 microns in this alloy, as can be found in the vicinity of damaged regions due to FOD impacts, fatigue thresholds can be a factor of two lower. In such instances where the critical condition for HCF must be defined in the presence of such microstructurally-small cracks, the Kitagawa-Takahashi diagram is more appropriate, where the limiting conditions are defined in terms of the smooth-bar fatigue limit (at microstructurally-small cracks sizes) and the "worst-case" fatigue threshold (at larger, "continuum-sized" crack sizes).

2:30 PM Invited
Step Loading, Coaxing and Small Crack Thresholds in Ti-6Al-4V under High Cycle Fatigue: *Ted Nicholas*[1]; [1]US Air Force Research Laboratory, AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

The conventional method for determining fatigue strength at high cycle counts is to either extrapolate S-N data from lower cycle counts or to generate S-N data in the high cycle count regime using specialized high frequency test machines. As an alternative, a step-loading method may be employed. One question that arises from a step-loading test is any effect due to coaxing, a phenomenon that causes an elevation of the fatigue stress due to prior cycling below the fatigue limit. Data have been obtained which validate the step-loading methodology. Applicability of high frequency testing to the determination of fatigue crack growth thresholds will also be discussed. It will be shown that fatigue limits for small cracks are not drastically altered from uncracked material when using stress rather than stress intensity as the governing parameter with which to characterize threshold behavior.

3:00 PM Invited
A Damage Tolerance Approach for Predicting the Threshold Stress for High Cycle Fatigue in the Presence of Supplemental Damage: *Stephen J. Hudak*[1]; [1]Southwest Research Institute, Matls. Eng. Dept., 6220 Culebra Rd., San Antonio, TX 78238-5166 USA

The occurrence of foreign object damage (FOD) in fan and compressor blades and fretting-fatigue at blade-to-disc attachments can significantly reduce the high cycle fatigue (HCF) resistance of advanced turbine engines. Thus, both of these damage processes need to be explicitly accounted for in design and structural integrity assessments. The high stress concentrations associated with these damage processes often lead to the initiation of microcracks early in the fatigue life, particularly under service loading where low cycle fatigue is also present. Consequently, a fracture mechanics based threshold approach has been developed for treating the growth and arrest of microcracks within the steep gradients that exist at FOD notches and edge-of-contact regions in fretting fatigue. The basis of the methodology will be described and the viability of the approach will be assessed by comparing model predictions with available HCF threshold stress measurements on Ti-6Al-4V specimens containing notches, simulated FOD, fretting, and fretting-fatigue. The consequences of treating the crack growth threshold stress intensity factor as a crack-size-dependent property will be discussed. Key similarities and differences between the FOD notches and edge-of-contact regions will also be highlighted. This work was supported under US Air Force Contract No. F49620-99-C-0007 monitored by Dr. Jeff Calcaterra of AFRL/ML and managed by Dr. Joe Gallagher of UDRI.

3:30 PM Break

3:40 PM Invited
Fatigue Life Predictions in Small Volume Components: *William W. Gerberich*[1]; [1]University of Minnesota, Chem. Eng. & Matls. Sci., 151 Amundson Hall, Minneapolis, MN 55455 USA

Over the years there has been a gradual recognition that the one to one correspondence between crack advance per cycle and fatigue striation spacing broke down at low *K. In an attempt to resolve whether this might apply to small volumes, small diameter wires were evaluated in tension-tension fatigue. Fatigue striations on the order of 20 nm per cycle were able to be resolved at 100,000 and 200,000X magnifications at *K = 4MPa-m1/2 and R>0.5 for both Ta and MP35N wire 250 μm in diameter. From similar tests using runouts in the 10 million cycle regime, thresholds were established. From the fatigue crack propagation tests a Paris Law exponent of two was found and a threshold-dependents algorithm allowing for zero growth as *K approached threshold was established. Using standard integration procedures, it is shown that the fatigue life of sharply notched wires is divided into regimes where initiation is no greater than about half of the total fatigue life for samples with *K > 3MPa-m1/2. The tentative conclusion is that fewer grains traversing the crack front and more uniform stress states combine to allow fcp analysis to afford a reasonable first approximation of failure life in small volume components.

4:10 PM
Mixed-Mode Fatigue Crack Growth Thresholds of Ni-Based Single Crystals: *Kwai S. Chan*[1]; Jim H. Feiger[1]; Yi-Der Lee[1]; Stephen J. Hudak[1]; [1]Southwest Research Institute, Matls. Eng. Dept., 6220 Culebra Rd., PO Drawer 28510, San Antonio, TX 78228-0510 USA

This investigation examines the effects of stress state and crystal orientation on the near threshold fatigue crack growth (FCG) response of PWA 1484 single crystals. Mixed mode FCG on (010) and (111) planes was characterized in air at 1100°F, using an asymmetric four-point bend test technique. Stress intensity solutions of the crack specimen were obtained using anisotropic fracture mechanics. The influence of mode mixity on the FCG threshold was determined as a function of stress state, ranging from pure Mode I to pure Mode II. Progress in applying the analytical and experimental results to develop a fracture mechanics approach for treating mixed-mode FCG in single crystal materials will be discussed. Work supported by AFRL through Contract No. F49620-99-C-0007.

4:30 PM
High Frequency FCP Thresholds in Nickel-Base Superalloys: *Walter W. Milligan*[1]; Amit Shyam[1]; Santo A. Padula[2]; [1]Michigan Technological University, Matls. Sci. & Eng., Houghton, MI 49931 USA; [2]NASA-Glenn Research Center, 21000 Brookpark Rd., MS 49-3, Cleveland, OH 49931 USA

High temperature, high frequency fatigue crack propagation thresholds were determined for a nickel-base turbine disk alloy. Effects of frequency, temperature, microstructure, and load ratio will be discussed. Relationships between thresholds and fractography and deformation mechanisms are explored. Sponsored by AFOSR MURI on High Cycle Fatigue.

4:50 PM
Cyclic Deformation Behavior and Damage Mechanism of HASTELLOY X Superalloy under Fatigue and Creep-Fatigue Loading: Lijia Chen[1]; *Peter K. Liaw*[1]; Gongyao Wang[1]; Robert L. McDaniels[1]; Kevin Liaw[1]; Scott A. Thompson[2]; James W. Blust[2]; Paul F. Browning[2]; Jose M. Aurrecoechea[2]; Rodger R. Seeley[3]; Dwaine L. Klarstrom[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Solar Turbines, Inc., 2200 Pacific Hwy., PO Box 85376, MZ R-1, San Diego, CA 92186-5376 USA; [3]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The fatigue deformation behavior of a nickel-based superalloy, HASTELLOY X, was investigated at 816C and 927C in laboratory air. It was noted that at both temperatures of 816C and 927C, the alloy exhibited initial cyclic hardening, followed by a saturated cyclic stress response or cyclic softening under fatigue loading condition. For creep-fatigue tests, the alloy showed either cyclic hardening or cyclic stability, which is closely related to the test temperature and the duration of the strain hold time. It was also observed that the fatigue life of the alloy considerably decreased due to the introduction of strain hold times. In addition, the fracture surfaces of the fatigued specimens were observed using scanning electron microscopy to determine the crack initiation and propagation modes. The fatigue cracks was usually found to initiate in a transgranular mode, while both transgranular and intergranular crack growth were observed.

Flyash: Generation, Treatment, Metal Recovery and Disposal - II

Sponsored by: Extraction & Processing Division, Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; I. Gaballah, Laboratoire Environmental et Mineralurgie, Associated to CNRS, ENSG-LEM, Vandoeuvre les Nancy 54501 France; David G. Robertson, University of Missouri-Rolla, Department of Metallurgical Engineering, Rolla, MO 65409-1460 USA

Monday PM — Room: 604
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: R. N. Singh, National Environment Engineering Research Institute, Nagpor India; Hideki Yamamoto, Kansai University, Dept. Cheml. Eng., Osaka 564-8680 Japan

2:00 PM Invited
Manufacture of Energy Efficient and Eco-Friendly Alinite Cements using Flyashes and Other Waste Materials: *Pradip*[1]; Maneesh Singh[1]; Anup Lakare[1]; P. C. Kapur[1]; [1]Tata Research Development and Design Centre, 54B Hadapsar Indl. Estate, Pune 411 013 India

We have been able to successfully convert a wide variety of fly ashes and other industrial waste materials into excellent quality hydraulic cements based on alinite cement chemistry. We present in this paper our recent results on the effect of various process variables optimized during our efforts on producing good quality alinite cements from a representative sample of chloride rich municipal incinerator fly ash obtained from South Korea. Most important process parameters include raw mix composition (represented in terms of lime index, silica modulus, iron modulus and chloride content), clinker grinding fineness and the clinkering schedule. It is possible to utilize up to 30% by weight of fly ash in the raw mix needed for the production of these cements. The quality of alinite cements thus produced are comparable in quality (in terms of physical properties and 1:3 cement/sand mortar cube compressive strengths) to portland cements and in fact, meet the Indian standard specifications for ordinary portland cements. Clinkering is carried out at 1150C for successfully achieving the desired proportion of alinite phase in the final cement. X-ray diffraction studies on these cements indicate alinite as the major phase with relatively minor quantities of other phases such as belite and calcium aluminochloride (11 CaO.7Al2O3.CaCl2). Addition of gypsum during clinker grinding is found to have a beneficial effect on the quality of cement produced by this low temperature clinkering route. This process thus offers an economically attractive and eco-friendly method of recycling chloride rich municipal incinerator ashes of considerable nuisance value.

2:30 PM Invited
Recycling Study of Korea's Fly Ash in Municipal Solid Waste Incineration Ashes for Cement Raw Material: *Ji-Whan Ahn*[1]; Hwan Kim[2]; [1]Korea Institute of Geoscience and Mineral Resources(KIGAM), PO Box 111, Yusung, Science Town, Taejon Korea; [2]Seoul National University, Sch. of Matl. Sci. & Eng., Seoul 151-742 Korea

The removal of chloride from fly ash in MSWIA (Municipal Solid Waste Incinerator Ash) by water-washing was investigated. The content of chloride in fly ash was 25-30% and the chloride compounds in fly ash were mainly KCl, NaCl, CaClOH and Friedel's salt (3CaO•Al2O3•10H2O). The effect of time and temperature in washing was surveyed. As a result, we found that the content of chloride in fly ash could be diminished to the content of approximately 2.0% by water washing on condition that washing time is 30 minutes, agitation speed, 300rpm and liquid/solid (v/w) ratio is 10. The removal of the chloride in fly ash can be used to improve the potential application of fly ash including cement raw material and building material and so on.

3:00 PM
Recovery of Lead and Zinc from Fly Ash from Municipal Incineration Plants by Means of Alkaline Leaching and Solvent Extraction: *Katsutoshi Inoue*[1]; Seham Nagib Tawfic[2]; [1]Saga University, Dept. of Appl. Chem., Honjo 1, Saga 840-8502 Japan; [2]Central Metallurgical R&D Institute Egypt

Fly ash from municipal waste incineration (MWI) plant is regulated as a hazardous waste because it contains considerable amounts of heavy metals and hazardous organic materials such as dioxin. Primary fly ash contains 0.8wt.% Zn and 0.12wt.% Pb, 1.37wt.% Fe while secondary fly ash contains 40.18wt.% Zn, 10.7wt.% Pb and 2.12wt.% Fe in addition to large amount of sodium and potassium chlorides. Therefore, it should be treated for detoxification or for the recovery of these metals as secondary resources. The recovery of by acid leaching using different kinds of acids such as sulfuric, hydrochloric and acetic acid was carried out. It was found that sulfuric acid leaching is effective where most of Zn was dissolved and also hydrochloric or acetic acid leaching was effective where most of Pb and Zn were dissolved. However, the acids dissolve also some impurities such as Ca, Fe and Al together with Pb and Zn from the fly ash. Therefore, alkaline leaching using NaOH as leachate was carried out; but, alkaline leaching was found to be suffered from low solubility of Zn. In order to improve Zn solubility, washing the solid residue using 2 or 5wt.% HCl was carried out which achieved the dissolution of almost all Pb and about 68wt.% Zn from secondary fly ash. For the purpose of separating and recovering Pb and Zn from the alkaline leach liquor, solvent extraction behavior of Kelex100 and D2EHPA for these metals from alkaline solution and from acetic acid media, respectively, were investigated.

3:20 PM
Recovering Metals in Fly Ash Derived from Heavy Oil by Combination of Leaching and Ion Exchange Processes: *Hideaki Tokuyama*[1]; Susumu Nii[1]; Fumio Kanaizumi[1]; Katsuroku Takahashi[1]; [1]Nagoya University, Dept. of Cheml. Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

Oil fly ash contains such metals as V, Ni and Fe. Recovery of these metals makes the fly ash less poisonous with the result that the ash becomes a valuable resource. The purpose of present study is to develop a process which can selectively recover V and Ni from the fly ash. Experiment of leaching with water, HCl, H2SO4 and NaOH leads us to propose a two-step leaching process as an effective and mild operation. The first step is leaching with water for removal of Ni, Mg, Al and Zn and the second step is leaching with acidic solution for the recovery of V. Then the metals V and Ni are separated from other metals in each leached liquor by ion exchange. Further advantage of this separation process is that the metal recovery by ion exchange permit to reuse acidic leachant, which makes for saving separation energy.

3:40 PM Break

3:55 PM
Heavy Metals Recovery in Vitrification Process of MSW Residues: *Takuya Shinagawa*[1]; Keisuke Nakahara[1]; Hiroshi Yamamoto[1]; Tetsuo Akashi[1]; [1]NKK Corporation, Eng. Rsrch. Ctr., Keihin Bldg. 6F, 1-1 Minami-watarida-cho, Kawasaki-ku, Kawasaki-shi, 210-0855 Japan

Vitrification is considered a principal process for future waste disposal because the treatment can render harmless even wastes that include harmful substances, and it can recover resources in the waste. The products from vitrification, such as slag and metal, can be used as construction material and weights, and moreover, NKK developed a

heavy metal recovery process in which zinc and lead in the dust are reclaimed as a raw material for smelting by making the best use of the characteristics of the dust. The NKK electric-resistance ash-melting furnace can volatilize the zinc and lead in residues at high rates using reduction melting and then concentrate these metals in the dust. The concentration of zinc and lead in the dust after washing is sufficiently high for the dust to be used as a raw material for the zinc and lead industry. The NKK electric-resistance ash melting furnace provides high performance in separating heavy metals and can produce dust advantageous for reuse as a raw material for smelting and safe slag containing less heavy metals. This system can ideally fulfill the function of vitrification.

4:15 PM
Spent Pot Lining (SPL) Treatment and Fluoride Recycling Process: *Ken Mansfield*[1]; [1]Portland Aluminium, PB 1, Portland, Victoria, 3305 Australia

Spent Pot Lining (SPL) is a hazardous waste product from the smelting of aluminium due to its fluoride and cyanide content. Disposal of SPL is a major problem for the aluminium industry due to disposal by landfill increasingly being banned. Between 1992-94 Portland Aluminium, Alcoa and Ausmelt conducted trials in Ausmelt's demonstration furnace to treat SPL. From these trials, and work with CSIRO to produce aluminium fluoride from the furnace offgas, Portland Aluminium and Alcoa authorized AUD$24,000,000 in 1995 to construct an SPL treatment facility. Output products from the process are aluminium fluoride, which has been successfully tried in the aluminium smelting process instead of imported material, and a vitreous granulated slag having leachability qualities acceptable to the EPA for specific end uses. The 'SPL Treatment and Fluoride Recycling Process' at Portland, Australia is successfully treating SPL and converting it into useful products.

4:35 PM
Improving Concrete Properties using Flyash: *Harovel G. Wheat*[1]; [1]University of Texas at Austin, Mechl. Eng., TX Matls. Inst., Austin, TX 78712 USA

It has been shown that the incorporation of flyash into concrete as a replacement for part of the cement can be beneficial. In some cases, the strength of the concrete can be increased and the permeability can be significantly decreased. This latter property can lead to a reduction in the migration and/or penetration of aggressive species into reinforced concrete. Some of these species, such as chlorides from deicing salts or marine environments, can subsequently result in accelerated corrosion of the steel reinforcement and a loss of structural integrity of the concrete. Care has to be taken so that the appropriate compositions of the components of the flyash are achieved in order to ensure maximum benefit to the concrete structures. This will be discussed as it relates to flyash generation and treatment.

Fundamentals of Advanced Materials for Energy Conversion: Magnets & Thermal Energy

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee
Program Organizers: Renato G. Bautista, University of Nevada-Reno, Department of Chemical and Metal Engineering, Reno, NV 89557-0136 USA; Dhanesh Chandra, University of Nevada-Reno, Metallurgical & Materials Engineering, Reno, NV 89557 USA

Monday PM — Room: 613
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Ricardo Schwarz, Los Alamos National Laboratory, Los Alamos, NM USA; Ramana G. Reddy, University of Alabama, A129 Bevill Bldg., PO Box 870202, Tuscaloosa, AL 35487 USA

2:00 PM Plenary
Magnetic Materials for the Conversion of Magnetic Energy to Cooling and Heating: *Karl A. Gschneidner*[1]; Vitalij K. Pecharsky[1]; [1]Iowa State University, Ames Lab., Ames, IA 50011-3020 USA

The application of a magnetic field to a magnetic material near its magnetic ordering temperature aligns the magnetic moments causing the material to warm up. When the field is removed the moments randomize thus cooling the magnetic material. This is analogous to a gas compressor which has been utilized for about 100 years to cool or heat. Recently it has been shown that magnetic refrigeration is a highly efficient technology, reaching 60% Carnot. Proof-of-principle apparati have achieved some notable firsts for magnetic cooling: a cooling power of 600 watts, a coefficience of performance approaching 15 and a temperature span of 38°C. It has been estimated that for large cooling devices, >50 kwatt, magnetic cooling is ~25% more efficient than conventional gas compression technology. Initial capital costs, however, are higher and it takes ~5 years to recover the higher costs. The status of magnetic heating and cooling will be summarized, including home air conditioners and refrigerator/freezers.

2:35 PM Plenary
Progress in Photovoltaics: A Success Story in Crystal Growth and Materials Science: *Thomas Surek*[1]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA

Photovoltaics (PV) is solar electric power-a semiconductor-based technology that directly converts sunlight into electricity. The manufacture and sale of PV has grown into a $2 billion industry worldwide, with nearly 300 megawatts (MW) of PV modules shipped in 2000. The majority of today's modules are based on crystalline silicon, building on advances in the growth of single crystal and polycrystalline ingots, as well as innovations in techniques to grow silicon sheets and ribbons directly from the melt. Some 25 years of research has led to the discovery and development of new PV materials and devices based on thin-film semiconductors and related alloys such as amorphous silicon, cadmium telluride, and copper indium diselenide. These materials can be deposited on low-cost substrates, such as glass, stainless steel, or plastics, by a number of potentially scalable, low-cost processes. These thin-film technologies are now entering the first-time manufacturing and commercialization stage. Very high efficiency devices have resulted form crystal growth advances of various III-V compounds, usually in multijunction device configurations where different portions of the solar spectrum are absorbed in successive layers of the device. Ongoing research is continuing to innovate new materials and device concepts which hold the promise of significantly higher solar conversion efficiencies and/or much lower costs. Based on the experience of the past 25 years, the successful development of these concepts will rely on continuing advances in crystal growth and materials science.

3:05 PM Invited
Bulk Ferromagnetic Glasses for Use in Energy Conversion Devices: *Ricardo B. Schwarz*[1]; T. D. Shen[1]; Ulrich Harms[1]; [1]Los Alamos National Laboratory, Struct./Prop. Relations Grp., MST Div., MS G755, Los Alamos, NM 87545 USA

The efficiency of transformers and motors can be increased significantly by making the cores from amorphous (glassy) ferromagnetic materials rather than crystalline Fe-Si alloys. Thin ferromagnetic foils, typically 30-50 microns thick, have been commercially available since the 1980s but their use has been limited by the thinnest of gauge, post-anneal brittleness, and stress sensitivity. The recent development of bulk ferromagnetic glasses solves some of these problems. We discuss the synthesis and magnetic properties of bulk ferromagnetic glasses of the type Fe-(Co,Cr,Mo,Ga,Sb)-P-B-C. The new bulk ferromagnetic glasses have a large supercooled region, T_x-T_g, ranging from 35 to 61 K, within which the glass can be shaped under a relatively small applied load. These glasses have coercivity as low as 0.004 Oe and hysteresis losses at 50 Hz of 0.04 W/kg.

3:35 PM Invited
Advanced Thermoelectric Materials and Devices for Energy Conversion: *Jeff Snyder*[1]; Thierry Caillat[1]; Jean-Pierre Fleurial[1]; [1]Jet Propulsion Lab, JPL-Caltech 277-207, Pasadena, CA 91109-8099 USA

This talk will focus on the recent advances made in thermoelectric materials and devices, particularly at JPL, with an emphasis on power generation. Thermoelectric materials convert heat directly into electrical power. New thermoelectric materials, particularly the skutterudites and zinc antimonide developed at JPL, have higher figure of merit and therefore higher conversion efficiency than state of the art materials. These materials, when combined into a segmented thermocouple can achieve 15% conversion efficiency from temperature gradients found in automobile exhaust. New concepts for power generation utilizing automotive or other sources of waste heat, or radioisotope heat sources in space, will be discussed.

4:00 PM Break

4:10 PM
Thermoelectric Properties of $IrSb_3$ and Related Ternary Skutterudite System: *Sung Wng Kim*[1]; Yoshisato Kimura[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Eng., Interdisciplinary Grad. Sch. of Sci. & Eng., 4259 Nagatsuta, Midori-ku, Yokohama 226-8502 Japan

The binary antimonide compounds with the skutterudite structure have a potential for thermoelectric applications because of the high carrier mobility, high thermoelectric power, and relatively low thermal conductivity due to the complex crystal structure. However, the room temperature thermal conductivity of these skutterudite materials is as high as about 10W/mK with an estimated 80-90% contribution from the lattice thermal conductivity. Because the lattice thermal conductivity of related ternary skutterudite compounds can be substantially reduced due to the additional phonon scattering, a study of thermoelectric properties of these compounds is of interest. The binary $IrSb_3$, ternary $Ir_{1-X}Ru_XSb_3$, and $Ir(M_XSb_{1-X})_3$(M=Ge, Sn) compounds were prepared by hot pressing of pre-reacted powders. We have measured the electrical resistivity, thermoelectric power, and thermal conductivity, in a temperature range from room temperature to 973K. The binary $IrSb_3$ compound is found to have P-type electrical properties exhibiting a good Seebeck coefficient(up to 225μV/K around 773K), electrical resistivity($5*10^{-5}$Ohm M around 933K), and relatively low thermal conductivity(6W/mK around 950K). The lattice thermal conductivity, k_{lat} was calculated from the Wiedemann-Franz relationship.

4:35 PM

Thermal Stability of Ionic Liquids: *Zhijing Zhang*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. of Metlgcl. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487 USA

Long-term thermal stability of ionic liquids is essential for reliable performance of ionic liquids in any engineering applications. TGA studies were carried out on a series of ionic liquids with different cations and anions such as 1-butyl-3-methylimidazolium chloride ($[C_4mim]Cl$), 1-hexyl-3-methylimidazolium chloride ($[C_6mim]Cl$), 1-hexyl-3-methylimidazolium hexafluorophosphate ($[C_6mim]PF_6$), 1-octyl-3-methylimidazolium hexafluorophosphate ($[C_8mim]PF_6$), and 1-butyl-3-methylimidazolium bis (trifluromethanesulflonyl) imide ($[C_4mim][Tf_2N]$). The short-term thermal stability of ionic liquids was evaluated by measuring the onset temperature of decomposition of ionic liquids, and the long-term thermal stability of ionic liquids was also evaluated by weight loss of ionic liquids over a long period of time at various temperatures. The experimental results showed that the long-term thermal stability of ionic liquids decreased with increasing temperature and was significantly lower than the onset temperature of decomposition of ionic liquids.

5:00 PM

Solid State Thermal Energy Storage in Tris (Hydroxymethyl) Aminomethane, Neopentylglycol and their Solid Solutions: John Hansen[1]; *Dhanesh Chandra*[2]; [1]University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA; [2]University of Nevada-Reno, Chem. & Metall. Eng., Mackey Sch. of Mines, Reno, NV 89557-0047 USA

A temperature-composition equilibrium phase diagram of the tris (hydroxymethyl) aminomethane (Tris)-neopentylglycol(NPG) binary system is developed to obtain various thermal energy storage materials with different transition temperatures. There are several space-related and other applications for this type of isothermal energy storage system. These organic crystalline materials undergo solid-solid phase transitions, and this property is used to store energy. The low temperature crystal structure of pure Tris is orthorhombic (α phase) and that of pure NPG is monoclinic (β phase). The high temperature "Plastic" (γ or γ') phases are cubic. The energy is stored in the γ or γ' phases primarily due to molecular motion of the O-H...O bonds. Guinier X-ray diffraction and differential scanning calorimetric data showed that there is virtually no solubility of Tris in NPG or visa-versa, below~43°C. A peritectic was observed at 150°C (L + γ —> γ') and also a eutectoid at 127°C (γ —> α + γ'). It is interesting to note that in certain regions of the phase diagrams, the energy is stored in two (γ + γ') plastic phases. Thermodynamic and crystal structure of solid solutions and components will be discussed.

5:20 PM

Phase Equilibria in Thermal Energy Storage Materials: Pentaerythritol and 2-Amino-2-Methyl-1,3-Propanediol: Renee Russell[1]; *Dhanesh Chandra*[2]; Wen-Ming Chien[2]; [1]Pacific Northwest National Laboratory, PO Box 999, MS K6-24, Richland, WA 99352 USA; [2]University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA

Organic molecular plastic crystals such as polyalcohols and amines reversibly store large amounts of thermal energy during solid-state phase transitions, which have many applications. A binary phase diagram for two such energetic plastic crystals, Pentaerythritol (PE) and 2-amino-2-methyl-l,3- propanediol (AMPL) is proposed. The crystal structure of AMPL was determined by single crystal method. This diagram was determined by high temperature Guinier x-ray diffraction and differential scanning calorimetric methods. The phase diagram in this study is rather complex with two eutectoids at 84°C and 147°C and a peritectic at 184°C. The low temperature phases of PE and AMPL are in equilibrium between 20°C and 84°C in the composition range of 12 to 98 mol% AMPL. The solubility of AMFL in PE is very high, up to 45 mol% AMFL in PE at 147°C. The solubility of PE in AMFL is very low, only up to 10 mol% PE in AMFL at 84°C. The structural and thermodynamic results, along with the amount of energy stored, will be presented.

Fundamentals of Structural Intermetallics: Nb and Mo Based Silicides

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials

Program Organizers: Young-Won Kim, UES, Inc., Materials & Processing Division, Dayton, OH 45432 USA; Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Vijay K. Vasudevan, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Monday PM — Room: 615-616
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Ian Baker, Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755-8000 USA; John Perepezko, University of Wisconsin-Madison, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA

2:00 PM Invited

A Review of Very High-Temperature Intermetallic Based Composites: *Bernard P. Bewlay*[1]; Melvin R. Jackson[1]; J.- C. Zhao[1]; P. R. Subramanian[1]; [1]GE-CRD, PO Box 8, Schenectady, NY USA

This paper will review recent progress in the development of intermetallic based composites for very high temperature applications. Emphasis will be given to Nb-silicide based in-situ composites, but there will also be discussion of systems based on other intermetallics, such as Laves phases and Mo-based silicides. Nb-silicide based in-situ composites contain high-strength silicides that are toughened by a ductile Nb-based solid solution. Simple composites are based on binary Nb-Si alloys, more complex systems are alloyed with Ti, Hf, Cr and Al. In higher order silicide-based systems, alloying elements have been added to stabilize intermetallics, such as Laves phases and boron rich T-2 phases, for additional oxidation resistance. In general, alloying schemes have been developed to achieve an outstanding balance of room temperature toughness, high temperature creep performance, and oxidation resistance. This paper will review recent progress in the development of composite processing-structure-property relatio nships in Nb-silicide based in-situ composites and the methodologies that have been employed for Nb-silicide composite property optimization. The Nb-silicide composite properties will be compared with those of advanced Ni-based superalloys, and other intermetallic-based systems.

2:30 PM Invited

Processing, Microstructures and Properties of Nb-Base and Mo-Base Metal/Silicide Multiphase Alloys: *Madan G. Mendiratta*[1]; Sarath K. Menon[1]; Young-Won Kim[1]; [1]Air Force Research Laboratory (AFRL), MLLM, UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA

Mo + Mo_5SiB_2+ Mo_3Si and Nb + Nb_5Si_3 + Cr_2Nb systems are being developed for high temperature structural applications. These alloys are being processed using different methods. The Mo-based alloys are being made using powder metallurgy approach; the prealloyed powders are consolidated by cold isostatic processing + sintering in H_2 atmosphere + hot isostatic pressing + hot-extrusion. Prealloyed powders made by plasma rotating electrode process are being consolidated by hot-extrusion. The Nb-based alloys are being made by induction skull melting and casting into ingots and thin plates. The cast ingots are further processed by hot-extrusion. The microstructures at various stages of processing and heat treating are being characterized. Tensile properties and fracture toughness will be determined, as a function of temperature and failure mechanisms will be correlated with microstructures.

3:00 PM
Computational Design and Property Optimization for Niobium-Based In-Situ Composites: *Kwai S. Chan*[1]; David L. Davidson[1]; [1]Southwest Research Institute, Matls. Eng. Dept., 6220 Culebra Rd., PO Drawer 28510, San Antonio, TX 78228-0510 USA

Niobium-based in-situ composites show potential for high-temperature structural applications, but they need simultaneous improvements in oxidation, creep, and fracture resistance. This paper describes the development of a material-science-based methodology that aids the design of alloy composition and microstructure of in-situ composites for optimized properties of oxidation resistance, yield strength, creep strength, and fracture resistance. Computational and analytical models are developed for describing properties of interest in terms of composition, microstructure, and properties of constituent phases in the in-situ composites. Using the property goals as the constraints, the range of constituent properties, volume fractions, and microstructures that would satisfy the imposed constraints are computed. Application of the proposed methodology to optimizing the oxidation, creep, and fracture resistance of Nb-based in-situ composites is presented and the results are compared against available experimental data in the literature. Work supported by the US Air Force Office of Scientific Research through Contract No. F49620-01-C-0016, Dr. Craig S. Hartley, Program Monitor.

3:30 PM Invited
High Temperature Properties of Laves Phase Two Phase Alloys: Dan J. Thoma[1]; *Robert J. Hanrahan*[1]; John D. Wittenberger[2]; [1]Los Alamos National Laboratory, TA 3, MS G770, Los Alamos, NM 87544 USA; [2]NASA, Glenn Rsrch. Ctr., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

An alloy design strategy has been developed and used to explore the utilization of Laves phases for high-temperature structural applications. NbCr2 was chosen as the base intermetallic constituent based upon existing high-temperature strength properties. Titanium was chosen as an effective addition to broaden the single-phase range of homogeneity and to optimize the deformation behavior of the NbCr2. Dual phase alloys consisting of a Nb/Ti bcc phase and the C15 (Nb,Ti)Cr2 phase were processed. The C15 phase, Nb20Ti20Cr60, had the optimum combination of properties in the ternary single-phase region, and all two-phase alloys examined were on the same tie-line with Nb20Ti20Cr60. 23Nb-34Ti-42Cr (at.%), has a maximum flow stress over 1GPa up to 1000°C, and accommodates compressive strains over 20% from room temperature to 1200°C. Additional studies were conducted of the oxidation and high temperature creep of the same alloys at 2E-4 to 2E-8/s and 900 to 1400 K in air.

4:00 PM
Microstructure Evolution, Thermal Stability and their Effect of Properties in a Multicomponent Nb-Si-X Alloy: *Young-Won Kim*[1]; Dennis M. Dimiduk[2]; Madan Mendiratta[1]; [1]UES, Inc., Matls. & Proc. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., Ste. 1, Wright-Patterson AFB, OH 45433-7817 USA

The microstructural evolution and thermal stability was investigated for a multicomponent, hypereutectic NbSi-X alloy, 45.5Nb-22Ti-2Hf-7Cr-20Si-2Al-1.5Sn (at%), was investigated for. Alloy material was produced in cast-plate forms using the induction skull melting technique (ISM). The cast material consisted of large-size, primary silicides (Nb_5Si_3 type) which were surrounded by eutectic-structure regions. The eutectic structure was a mixture of relatively fine secondary silicides (Nb_5Si_3 type) and dendritic beta phase having small amounts of interdendritic Laves-phase particles. When annealed at 1400°C for 50h and then HIP'ed at 1450°C for 6h, the material showed significant microstructure changes; that is, transformation of the eutectic structure to a fine mixture of slicides and beta grains, partial decomposition of large Nb_5Si_3 type silicides to Ti_5Si_3 type silicides, and fine decoration of Laves phase particles along beta-grain boundaries. This process resulted in fairly uniform, refined microstructures. These microstructures have been tested for their thermal instability and/or phase redistribution happening when exposed to temperatures lower than 1400°C, which is near the upper limit of the desired use temperature. Measurements of yield stress and oxidation resistance are underway for selected microstructural forms, and their interrelationships will be analyzed.

4:30 PM
Development of High Temperature Coating Strategies for Mo-Si-B Alloy: J. S. Park[1]; R. Sakidja[1]; *J. H. Perepezko*[1]; [1]University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA

The development of protective coatings and the oxide scales on Mo-Si-B alloys were examined. Two types of approaches are employed; stable oxides and molybdenum disilicide (MoSi2) phase. The oxide coatings were produced through spray coatings, whereas the disilicide deposits were synthesized through the pack cementation process. Following oxidation in air at 1000-1200C for times up to 100 hrs, the substrate formed oxides composed of borosilicate, MoO2 and the metalloid-depleted zone with silica dispersions. With the oxide coatings, due to constricted oxygen diffusion, improved oxidation resistance was observed as reflected by the reduced thickness of the layered oxides. With disilicide coatings on the other hand, no layered oxides were observed. Instead, limited layered structures composed of Mo5Si3 and borosilicide phases were observed between disilicide coating and the substrate. The growth kinetics of layered structures and the oxides are presented. The support of ONR (N00014-92-J-1554) and AFOSR (F49620-00-1-0077) is gratefully acknowledged.

4:50 PM Invited
Cyclic Fatigue-Crack Growth and Fracture Behavior of Mo-Mo3Si-Mo5SiB2 Silicides at Ambient and Elevated Temperatures: *Heeman Choe*[1]; Joachim H. Schneibel[2]; Robert O. Ritchie[1]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., 463 Evans Hall, #1760, Berkeley, CA 94720-1760 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831-6115 USA

The quest for structural materials that can operate at higher and higher temperatures remains a persistent challenge in materials science. Among the potential candidates for higher-temperature advanced engine systems are multiphase Mo-Si-B intermetallics due to their high melting temperature (> 2000°C) and relatively good oxidation resistance. In this study, we investigate the damage-tolerant properties, i.e., fracture toughness and fatigue-crack propagation behavior, of several such multiphase Mo-Si-B intermetallics. Specifically, the alloys Mo-12Si-8.5B, Mo-16.8Si-8.4B, and Mo-10Nb-12Si-8.5B (at.%), consisting of alpha-Mo, Mo3Si, and Mo5SiB2 (T2) as their primary phases, are examined at temperatures from ambient to 1300°C, with the objective of discerning salient toughening mechanisms. The alloy with 16.8 at.% Si contains less alpha-Mo than that with 12 at.% Si. It is more brittle, but at the same time more oxidation resistant. It is found that Mo-12Si-8.5B (at.%) alloy in particular displays relatively high intrinsic (crack-initiation) toughness both at ambient and elevated temperatures, which is attributed to a crack-trapping mechanism at coarse alpha-Mo particles. Moreover, both fracture toughness and fatigue-crack growth properties in this alloy actually improve as temperature increases up to 1300°C. This is principally due to the enhanced ductility of alpha-Mo phase at elevated temperatures, which promotes some degree of extrinsic toughening, specifically crack bridging, which in turn results in rising resistance-curve behavior at 1300°C. This work was sponsored by the US Department of Energy, through the Office of Science, Office of Basic Energy Sciences, Materials Sciences Division of the US Department of Energy under Contract No. DE-AC03-76SF00098 (for HC and ROR), and the Office of Fossil Energy, Advanced Research Materials (ARM) Program, under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory, managed by UT-Battelle, LLC (for JHS).

5:20 PM
High Temperature Mechanical Behavior of A15 Mo_3Si Single Crystals: *Isai Rosales*[1]; Joachim H. Schneibel[1]; Lee Heatherly[1]; Lorenzo Martinez[2]; Bernardo F. Campillo[2]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831 USA; [2]National University of Mexico, Fac. of Chem., Mexico D.F. Mexico

Mo_3Si single crystals were grown in an optical floating zone furnace, specimens with <111>, <110> and <100> directions were fabricated, and high temperature compression test were performed at 1400°C in vacuum (10-6 Torr). High stress values ranging from 600 to 1100 MPa were observed. Depending on the strain rate and stress, localized recrystallization occurred. Analysis of the slip traces on the surfaces of the deformed specimens was carried out in order to identify the active slip planes, which are {010} for the specimens with <110>, and {012} for the specimens with <111> compression axis. No slip traces were observed for the <100> orientation confirming that the resolved shear stress was zero. TEM study of the slip planes is in progress. Differences in cracking behavior were observed after conical indentation with a load of 0.5 kg. The preferential cleavage planes were {211} for the specimens indented on (111) and (110) surfaces, and {110} for the (110) surface. This work was sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, US Department of Energy, under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory managed by UT-Battelle, LLC.

5:40 PM
Reducing the Thermal Expansion Anisotropy of Mo_5Si_3-Based Molybdenum Silicides: *Joachim H. Schneibel*[1]; Chong Long Fu[1]; Claudia J. Rawn[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831 USA

The intermetallic phase Mo_5Si_3 is of interest because of to its high melting point (2180°C) and potential oxidation resistance. However, its coefficient of thermal expansion (CTE) is highly anisotropic. Theoretical considerations suggest that this anisotropy can be reduced by stretching the Mo-chains in the c-direction of the tetragonal crystal structure, or by partial substitution of the chain atoms with smaller atoms. High temperature x-ray diffraction experiments confirm indeed that additions of atoms which are larger or smaller, respectively, than Mo reduce the CTE anisotropy from a value of 2 for Mo5Si3 to values as low as 1.3 for ternary $(Mo,Nb)_5Si_3$ compounds. The results are discussed in terms of the site occupation of the ternary alloying elements and the underlying electronic structure. The lower anisotropy is accompanied by a dramatic reduction in the density of microcracks in the material. This work was sponsored by (a) the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, US Department of Energy, and (b) the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program. ORNL is operated by UT-Battelle, LLC, for the US Department of Energy under contract DE-AC05-00OR22725.

General Abstracts: Nickel and Molybdenum

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA

Monday PM — Room: 209
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Carl Cady, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

2:00 PM
Double Serpentine Diffusion Paths in Ternary Fe-Ni-Al System: *Mysore A. Dayananda*[1]; Junghan Kim[1]; [1]Purdue University, Sch. of Matls. Eng., MSEE Bldg., W. Lafayette, IN 47907 USA

Solid-solid diffusion couples assembled with (bcc) Fe-Ni-Al alloys were investigated for the development of uncommon diffusion paths. Selected couples were isothermally annealed at 1000°C for 2 days and analyzed for concentration profiles by electron microprobe analysis. Profiles of interdiffusion fluxes were determined and the couples were examined for the development of zero-flux planes and regions of uphill interdiffusion for the individual components. Double-serpentine diffusion paths with two crossings of the straight line joining the terminal alloy compositions on the Fe-Ni-Al isotherm were observed for several couples. These unusual paths were characterized by the development of separate regions of up-hill interdiffusion on both sides of the Matano plane for at least one component. Diffusional interactions among the components were assessed at the compositions of the zero-flux planes developed by the various components. The paths are discussed in the light of interdiffusion and thermodynamic data for Fe-Ni-Al alloys.

2:25 PM
Ternary Interdiffusion and Accumulation of Al in Columnar-Grained $MoSi_2$: *Edward J. Ciecko*[1]; Mysore A. Dayananda[1]; [1]Purdue University, Matls. Sci. & Eng., 1289 MSEE Bldg., W. Lafayette, IN 47907 USA

Materials characterized by columnar structures (long, narrow grains) differ from those characterized by large, equiaxed grains because of the abundance of grain boundaries and the presence of relatively large number of triple junctions. Interdiffusion in such materials was investigated by means of a vapor-solid diffusion couple involving columnar-grained $MoSi_2$ in contact with Al vapor at 1000°C. The formation of the hexagonal (C40 type) phase $Mo(Si,Al)_2$ was observed. In addition, the development of relative maxima in the Al concentration profiles was observed to occur in the columnar-grained $MoSi_2$, indicating an accumulation of Al within the diffusion zone. The relative accumulation of Al is examined on the basis of the phenomenology of bulk ternary diffusion and is discussed in terms of diffusional contributions from grain boundaries and triple junctions.

2:50 PM
Pre-Precipitates in Ni-Rich Ni-Ti Alloys: *Gernot Kostorz*[1]; Michael Kompatscher[1]; Bernd Schoenfeld[1]; [1]ETH Zurich, Angewandte Physik, Zurich CH 8093 Switzerland

The stable precipitate phase forming in Ni-rich f.c.c. Ni-Ti solid solutions is the hexagonal η-phase, but metastable coherent precipitates are found at intermediate temperatures. In-situ small-angle scattering on single crystals of Ni-(10-12) at.% Ti show a complex sequence of decomposition for ageing temperatures around 600°C. An initial stage with precipitates containing about 17 at.% Ti is followed by an increase of the Ti content in the particles to about 22 at.% Ti. These conclusions stem primarily from the variation of the integrated small-angle scattering intensity as a function of ageing time. Further confirmation is obtained from transmission electron microscopy and diffuse scattering near the fundamental Bragg peaks and the superstructure peaks of the precipitates (L_{12} structure). A comprehensive view of the various stages of decomposition, the degree of order, the shapes and interfacial details of the precipitates, and the coherency strains due to elastic mismatch between particles and matrix emerges.

3:15 PM Break

3:40 PM
Fracture Toughness and Tensile Properties in Nanocrystalline Nickel Thin Sheets: Reza A. Mirshams[1]; *Parviz S. Razi*[2]; C. H. Xiao[2]; [1]University of North Texas, Eng., Denton, TX 76203 USA; [2]Southern University and A&M College, Mechl. Eng., Baton Rouge, LA 70813 USA

The potential engineering applications of nanocrystalline materials need more detailed study on deformation and fracture mechanisms at room and elevated temperatures under tensile loading. This paper reports results of a series of experiments carried out on nickel and carbon doped nanocrystalline nickel with different carbon concentrations from 500 to 1000 ppm at room temperature to 300°C. Grain growth was observed in nanocrystalline nickels as the testing temperature increases. A fast grain growth was noticed at 300°C. Pure nanocrystalline nickel experienced an abnormal grain growth at 500°C and its tensile properties reduced to a very low level. The addition of carbon exerted a potential effect to enhance the stability of the microstructure in nanocrystalline nickel at intermediate temperatures. However, carbon doped nickels exhibited lower tensile properties. Nanocrystalline nickels displayed a conventional Hall-Petch relationship. The results are discussed in relation to microstructural characteristics by using TEM and SEM.

4:05 PM
Measuring the Fracture Toughness of TZM and ODS Molybdenum Alloys using Standard and Sub-Sized Specimens: *Brian V. Cockeram*[1]; [1]Bechtel-Bettis, ZAP 08D/MT, PO Box 79, W. Mifflin, PA 15122 USA

Oxide Dispersion Strengthened (ODS) and TZM molybdenum have excellent creep resistance and strength at high temperatures in inert atmospheres. The fracture toughness and transition temperature for brittle behavior must be known to evaluate the use of molybdenum for structural applications. Fracture toughness (Kic) testing in accordance with ASTM E399 methods was performed over a range of temperatures to characterize 0.25" thick plate material of ODS and TZM molybdenum. The transition temperature for brittle behavior of ODS molybdenum was below room-temperature. The transition temperature for TZM molybdenum in the longitudinal direction was slightly above room-temperature, while the transition temperature for the transverse direction was 150C. The use of sub-sized specimen geometries produced fracture toughness results that were comparable to standard-sized specimens.

4:30 PM
Molecular Dynamics Simulation and Experimental Proof of Hydrogen-Enhanced Dislocation Emission in Nickel: *Z. J. Li*[1]; J. X. Li[1]; H. Liu[1]; L. J. Qiao[1]; W. Y. Chu[1]; [1]University of Science and Technology-Beijing, Dept. of Matls. Phys., Beijing 100083 China

The molecular dynamic simulations indicated that hydrogen solubilized in nickel decreased the critical stress intensity for dislocation emission from $K_{Ie}(\theta = 45^0)=1.0MPam^{1/2}$ or $K_{Ie}(\theta = 70°)=0.82MPam^{1/2}$ to $K_{Ie}(\theta = 45°)=0.9MPam^{1/2}$ or $K_{Ie}(\theta = 70°)=0.7MPam^{1/2}$, respectively. Therefore, hydrogen could enhance dislocation emission. On the other hand, hydrogen decreased critical stress intensity for cleavage of a Griffith crack along the only slop plane in the quasi three-dimension model from $K_{IG}(\theta = 0°)=1.03MPam^{1/2}$ to $K^*_{IG}(\theta = 0°)=0.93MPam^{1/2}$ and

then the surface energy from γ_{111}=2.2J/m^2 to γ_{111}=1.78J/m^2, resulting in facilitating dislocation emission. A constant deflection device designed for use within a transmission electron microscope (TEM) was used to study the change in dislocation configuration ahead of a loaded crack tip before and after charging with hydrogen. In situ observation in TEM showed that hydrogen enhanced dislocation emission and motion before initiation of hydrogen-induced crack.

General Abstracts: Aluminum and Titanium

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA

Monday PM Room: 310
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chair: George T. Gray, III, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

2:00 PM
Deformation Microstructure Evolution in Single Crystal Aluminum Alloys: *Michael F. Savage*[1]; Donald E. Kramer[1]; Lyle E. Levine[1]; [1]NIST, Metall. Div., 100 Bureau Dr., MS 8553, Gaithersburg, MD 20899-8553 USA

Localized deformation and the formation of complex dislocation structures play a significant role in the room temperature flow behavior of high stacking fault energy FCC metals. In this study, the slip line evolution and underlying deformation microstructures of high purity single crystal Al and Al solid solution alloys have been investigated. Atomic force microscopy investigations of surface slip bands were performed during deformation of Al single crystals with an evolved dislocation structure. These results have been compared with the deformation microstructures observed in thin foils ex-situ, by Transmission Electron Microscopy. Relationships between the observed surface structures and the presence of Geometrically Necessary Boundaries (GNB's) and Incidental Dislocation Boundaries (IDB's) will be discussed. Effects of crystallographic orientation on the slip line evolution and the nature of these dislocation structures will also be presented. The impact of these results will be discussed with respect to an ongoing modeling effort of plasticity in dislocation cell-forming FCC metals.

2:25 PM
Fatigue Crack Growth in Al-7.5Mg Bulk Nanocrystalline Materials: *Peter S. Pao*[1]; Steve J. Gill[1]; Harry N. Jones[1]; Jerry C. Feng[1]; [1]Naval Research Laboratory, Washington, DC 20375 USA

The fatigue crack growth of bulk nanocrystalline Al-7.5Mg was investigated. Nanocrystalline particulates were prepared by cryo-milling. These particulates were then compacted and extruded into rods. The fatigue crack growth rates of nanocrystalline Al-7.5Mg are significantly higher than those of ingot 7050-T7451. The fatigue crack growth thresholds of the nano alloys are significantly lower than that of 7050-T7451. Post-fatigue SEM investigation has revealed that the fracture surfaces of nano alloys are very smooth and the higher fatigue crack growth rates and lower thresholds may be attributed to the much smoother fracture surface morphology and lower roughness-induced crack closure in nano alloys. The fracture toughness of the nanocrystalline Al-7.5Mg is significantly lower than that of 7050-T7451. The reason for the lower fracture toughness in the bulk nano alloys is not understood yet but may be related to the lack of dislocation activities for energy dispersal in the fracture processes.

2:50 PM
Evaluation of the StressWave Fatigue Life Enhancement Process for Fastener Holes in Aluminum Alloys: *Brian D. Flinn*[1]; Christopher Meyer[1]; Eric T. Easterbrook[2]; [1]University of Washington, Matls. Sci. & Eng., Box 352120, Seattle, WA 98195 USA; [2]StressWave, Inc., 12932 S.E. Kent-Kangley Rd., MS 528, Kent, WA 98031 USA

The fatigue life improvement of the StressWave process was compared with existing split sleeve cold expansion technology and untreated specimens using aluminum 2024-T3 specimens 0.190" (4.82 mm) and 0.063" (1.6mm) thick. The StressWave method (patents pending) overcomes many of the limitations of the split-sleeve and split-mandrel cold working processes. Constant amplitude fatigue tests were conducted on zero load transfer, open hole specimens to evaluate the performance of StressWave-processed holes relative to holes treated with other methods. StressWave produces residual compressive stresses and fatigue performance comparable to, or better than, those produced by the existing processes.

3:15 PM
Fatigue Life Improvement of Fastener Holes in Titanium with the StressWave Process: *Brian D. Flinn*[1]; Scott Llitteras[1]; Milton Siegelmann[2]; Eric T. Easterbrook[2]; [1]University of Washington, Matls. Sci. & Eng., Box 352120, Seattle, WA 98195 USA; [2]StressWave, Inc., 12932 S.E. Kent-Kangley Rd., MS 528, Kent, WA 98031 USA

The fatigue life of titanium 6-4 open hole specimens was measured after treatment with the StressWave™ fatigue life enhancement process. The StressWave™ process induces beneficial compressive residual stresses in the material surrounding the fastener hole via plastic deformation prior to machining the hole. Specimens were tested at three StressWave™ process conditions. Fatigue life, crack initiation and growth are compared with untreated specimens. The StressWave™ process increased the fatigue life by four to ten times that of untreated holes. This life improvement is comparable to that of split-sleeve and split mandrel cold expansion processes.

3:40 PM Break

4:05 PM
Aluminum Titanate Formation and Densification under an External Electric Field: Influence of Precursor Powders: *Lia Stanciu*[1]; Joanna R. Groza[1]; Vladimir Kodash[1]; Carmen Plapcianu[2]; [1]University of California at Davis, Dept. of Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]National Institute for Materials Physics, Str. Atomistilor, 105 bis PO Box MG-776900, Bucharest 776900 Romania

Present work reports the results obtained by FAST (Field Activated Sintering Technique) sintering of three types of powders: cogelified alumina-titania powders, mechanical mixtures of individual alumina and titania gel powders, and alumina-titania precipitates obtained by a flotation method. The use of sol-gel cogelified alumina-titania powders led to a 98% formation of aluminum titanate, high density, and a grain size of 1-2 microns, at 1100°C. Mechanical mixture of individual alumina and titania powders, as well as the powders obtained by flotation method resulted in less aluminum titanate at this temperature and lower density. The sample obtained by sintering the powders obtained by flotation method has also a larger grain size, of about 5 microns. It has been concluded that sol-gel cogelified powders offer the best results in terms of grain size and composition of final ceramic due to the high homogeneity and initial nanometric particle size.

4:30 PM
Fatigue Behavior of Ti-64 Forged Alloy at Ambient and Elevated Temperatures: *Chih-An Yin*[1]; Malcolm C. Thomas[1]; [1]Rolls Royce Corporation, Matls. & Proc. Dept., PO Box 420, MC S-52, 2001 S. Tibbs Ave., Indianapolis, IN 46241 USA

Designing against fatigue failure involves so many uncertainties that it is usually necessary to test full-scale structures prior to service to prove their reliability. Such tests should be simple, short and inexpensive. At the same time, it is necessary to insure that the fatigue response of the laboratory test reflects the actual performance that can be expected of the structure in its service environment. For this reason, the material characterization testing for tensile, low cycle fatigue and fatigue crack growth responses were conducted on Ti64 forged alloy subjected to a specified heat treatment. Strain based mechanical hysteresis concepts have been used to establish cyclic stress strain behavior and fatigue life characteristics of the present material at ambient and elevated temperatures. The stress intensity factor concept of fracture mechanics has been used to establish the fatigue crack growth rate data at ambient and elevated temperatures. These selected test temperatures are corresponding to the conditions of normal operation in different stages of compressors for some aircraft engines. The analysis of the experimental results are based mainly on the phenomenological approach and predicted methods. So that a quantitative evaluation can be used to assess the effect of test temperatures on the cyclic deformation behavior, tensile mean stress relaxation resulted from the positive mean strains, and its effect of fatigue damage. In addition, the measured notch fatigue lives and lives predicted from smooth specimen fatigue data are illustrated based on Neuber's rule.

4:55 PM
Low Temperature Superplasticity of Ultrafine Grained 5083 Al Alloy: *Kyung-Tae Park*[1]; Deok-Young Hwang[1]; Ki-Seung Lee[2]; Dong Hyuk Shin[2]; [1]Hanbat National University, Div. of Adv. Matls. Sci. & Eng., San 16-1, Dukmyung-Dong, Yuseong-Gu, Taejon 305-719 S. Korea; [2]Hanyang University, Dept. of Metall. & Matls. Sci., 1271 Sa 1-Dong, Ansan, Kyunggi-Do 425-791 S. Korea

A ultrafine grained structure was introduced in a 5083 Al alloy by imposing an effective strain of 8 through equal channel angular pressing (ECAP). Tensile tests were performed on the as-ECAPed samples in the strain rate range of 10-5x10-2 s-1 at temperatures of 498-548 K. In order to examine low temperature superplastic behavior of the alloy. The maximum elongation of 315 % was obtained at 548 K and 5x10-4 s-1. In addition, the analysis of the mechanical data indicated that low temperature superplasticity of the present ultrafine grained alloy was attributed to grain boundary sliding that is rate-controlled by grain boundary diffusion with low activation energy associated with non-equilibrium grain boundaries. Necking instability during low temperature superplastic deformation of the alloy was also discussed by applying the Hart's necking instability criterion.

General Abstracts: Ferrous Materials: Carbon and Low-Alloy Steels

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA

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February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Robert Hackenberg, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

2:00 PM
The Effect of Surfactant and Environmental Parameters on Mild Steel Corrosion Inhibition: *Michael L. Free*[1]; [1]University of Utah, Metlgcl. Eng., 135 S. 1460 E. Rm. 412, Salt Lake City, UT 84112 USA

The effects of parameters such as solution environment, hydrocarbon chain length, critical micelle concentration, surfactant concentration, and functional group on mild steel corrosion were evaluated by electrochemical testing. The results from the electrochemical testing were utilized together with theoretical considerations to develop a mathematical model for the prediction of mild steel corrosion inhibition using a variety of surfactants.

2:25 PM
Effects of Microstructure and Hardness on Machinability of 0.6% Carbon Steels for Machine Structural Use: *Yasuhiro Omori*[1]; Toshiyuki Hoshino[1]; Akihiro Matsuzaki[1]; Keniti Amano[1]; [1]Kawasaki Steel Corporation, Techl. Rsrch. Labs., Kawasakidori 1-Chome, Mizushima, Kurashiki, Okayama 712-8511 Japan

In order to improve the machinability of a 0.6%C steel in terms of the fatigue strength without using free cutting additives such as Pb, effects of both microstructure and hardness on machinability were investigated. Though the tool life measured by turning test was approximately controlled by hardness of steel, the tool life of bainitic steel was longer than that of the pearlite-ferrite steel, even though the hardness was the same. The tool life, on the other hand, had a good correlation with cutting force independently of microstructure of steel. Since coarse planar carbides which had random orientation in bainite played a big role as origins of stress concentration, shear angle increased and thus the cutting force decreased. The decrease in the cutting force also decreased tool temperature during cutting, which improved the tool life by preventing Co in the tool from diffusing into chips.

2:50 PM
Decomposition of Retained Austenite during the Coiling Process of Hot Rolled TRIP Steels: *Hyun Jo Jun*[1]; Sung Ho Park[2]; Chan Gyung Park[1]; [1]Pohang University of Science & Technology, Matls. Sci. & Eng., Hyojadong Namgu san 31, Pohang, Kyungbuk 790-784 Korea; [2]Technical Research Laboratory, POSCO, Pohang, Kyungbuk Korea

A good combination of strength and formability of TRIP steels is mainly attributed to the retained austenite transforming into martensite during the plastic deformation. However, the retained austenite in hot rolled sheet steels can be decomposed into other phases during the coiling process, which can produce serious inhomogeneity of the TRIP sheet steels. In order to evaluate this decomposing behavior of the retained austenite, the XRD and TEM in-situ heating observations have been made in the hot-rolled C-Mn-Si TRIP steels. It was found that the retained austenite was decomposed into various phases depending on the coiling temperatures ranging from 350 to 500C: carbide-free bainitic ferrite, ferrite associated with iron carbide and pearlite. The difference of these decomposition behaviors will be discussed in terms of the variation of carbon concentration and thermal stability of the retained austenite at high temperatures.

3:15 PM
Fracture Toughness Analysis in Transition Temperature Region of Mn-Mo-Ni Low Alloy Steels: *Sangho Kim*[1]; Sunghak Lee[1]; Bong Sang Lee[2]; Yong Jun Oh[2]; [1]POSTECH, Dept. of Matl. Sci. & Eng., San 31, Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 Korea; [2]Korea Atomic Energy Research Institute, Nucl. Matls. Tech. Dvlp. Team, 150 Dukjin-dong, Yuseong-ku, Taejon 305-353 Korea

An investigation was conducted into the effect of the microstructures on fracture toughness in the ductile-brittle transition temperature region of various Mn-Mo-Ni high strength low alloy steels. Microstructures were analyzed using OM, SEM, and TEM. In order to evaluate the fracture toughness in the transition range, reference temperatures were determined according to ASTM E1921-97 standard test method. Fracture toughness in the transition region was interpreted by metallurgical factors such as yield strength, carbide size distribution, local fracture stress. It is shown that the critical nearest-neighbor distance between coarse carbides was an important microstructural factor affecting elastic-plastic fracture toughness, since it satisfied a linear relationship with the critical distance between a crack tip to a cleavage initiation site. These findings suggested that reducing the total number of carbides and M3C carbide fraction was useful to improve fracture toughness in the transition region.

3:40 PM
Change in the Precipitation Behavior of MnS with Ti Addition in Mn/Si/Ti Deoxidized Steels: *Han Soo Kim*[1]; Hae-Geon Lee[1]; [1]Pohang University of Science and Technology, Matls. Sci. & Metlgcl. Eng., San 31, Hyoja-dong, Pohang, Kyungbuk 790-784 Korea

Precipitation behavior of MnS on oxide inclusions was studied during solidification of Mn/Si/Ti deoxidized steels. The morphology of MnS precipitates accompanying oxides was varied with Ti addition, which might be attributed to the change in composition of oxide inclusions. Thermodynamic calculations were carried out to predict the change in the liquidus temperature of oxide inclusions with varying composition using computational thermodynamic method. Morphological change of MnS precipitates accompanying oxide inclusions could be interpreted using variation of liquidus temperature of oxide inclusions. Formation of Mn-depleted zone around oxide inclusions was also investigated. The change in the formation of Mn-depletion zone with Ti addition in the vicinity of oxide inclusions accompanying MnS could be explained with the help of understanding MnS precipitation behavior.

High Performance Metallic Materials for Cost Sensitive Applications: Lightweight Metals

Sponsored by: Structural Materials Division, Structural Materials Committee, Titanium Committee
Program Organizers: Edward Y. Chen, TiTech International, Inc., Pomona, CA 91768 USA; Rod Boyer, Boeing Commercial Airplane Group, Seattle, WA 98124-2207 USA; F. H. (Sam) Froes, University of Idaho, Institute of Materials and Advanced Processes, Moscow, ID 83844-3026 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Monday PM — Room: 213
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Eric Taleff, The University of Texas at Austin, Austin, TX 78705 USA; Theodore Reinhart, Boeing Military Airplanes, Seattle, WA 98124 USA

2:00 PM Invited
Weld Rework of Alloy D357 Castings: *Theodore L. Reinhart*[1]; Fred J. Feiertag[2]; [1]Boeing Military Airplanes, PO Box 3707, MS 4E-03,

Seattle, WA 98124 USA; [2]The Boeing Company, PO Box 3707, MC 5L-14, Seattle, WA 98124 USA

Weld rework of aluminum alloy castings is an integral part of the production process despite restrictions to noncritical/nondesignated/ low stress regions of aerospace castings. This restriction, however, is a greater issue when larger, higher value parts are considered. Scrapping such a part for a relatively minor defect, one which could otherwise be reworked by welding, carries a painful economic penalty. In fact, the penalty may be sufficiently serious as to preclude consideration in some applications. This presentation covers the results achieved using improved welding procedures. These new procedures yielded results demonstrably better than those achieved using welding practices currently used by the casting industry.

2:30 PM Cancelled
Synthesis of Aluminum Alloys using Semi-Solid Thermal Transformations: *Michael E. Kassner*

2:30 PM
ALE-Finite Element Simulation of U-Shape Aluminum Profile Extrusion: *Zezhong Chen*[1]; Zhongxu Bao[1]; Hesheng Liu[1]; [1]Nanchang University, Sch. of Mechl. & Elect. Eng., Nanchang, 330029 China

Because the coefficient of extrusion (Ratio of billet and profile cross section area) is too large (more than 30), it is almost impossible to simulate aluminum profile extrusion process using Lagrange finite element method. It is hard to get satisfactory result since grid distortion level is exceptional and grid remeshing can not be conduct. On the other hand, Eulerian finite element method is widely used in fluid flow simulation since it is a good way for description of fluid flow. Therefore, Arbitrary Lagrange-Eulerian finite element method is adopted in the paper to simulate the "U" shape profile extrusion process. Lagrange method is used for mould and Eulerian method is used for metal flow. Satisfactory result, such as deformation process, stress and strain distribution, etc., are obtained.

2:50 PM
Spray Forming of Hypereutectic Al-25Si Alloy and Processing Condition for Hot Extrusion: *Woo-Jin Park*[1]; Tae Kown Ha[2]; Joseph Kim[2]; Sangho Ahn[1]; Young Won Chang[2]; [1]Research Institute of Industrial Science and Technology, Matls. & Proc. Rsrch. Ctr., Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-600 S. Korea; [2]Pohang University of Science and Technology, Ctr. for Adv. Aeros. Matls., Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 S. Korea

The fabrication of hypereutectic Al-25Si alloy via spray forming process, which are expected to be applied to the cylinder liner part of the engine block of an automobile due to excellent wear resistance, low density and low thermal expansion coefficient, has been reported in this article. The characterization of the microstructural and the mechanical properties were also carried out. The microstructure of the hypereutectic Al-25Si alloy appeared to consist of Al matrix and equiaxed Si particles of average diameter of 5~7 μm. To obtain the condition for hot extrusion, a necessary process for application as the cylinder liner, a series of load relaxation and compression tests have been conducted at temperatures ranging from RT to 500°C. The strain rate sensitivity parameter (m) of this alloy has been found to be very low (≤ 0.1) below 300°C and reached maximum value of about 0.2 at 500°C. The dynamic material model has been applied to construct the processing map of this alloy. The experimental extrusion has been successfully conducted at the temperatures of 300°C and above with the ratio of area reduction of 28 and 40.

3:10 PM Cancelled
Forming Properties and Mathematical Model of Semi-Solid AlSi9Cu3 Aluminum Alloy for Thixoforming: *Xie Shuisheng*

3:10 PM Invited
Materials for Antiterrorism and Other Critical National Needs: *Toni Grobstein Marechaux*[1]; [1]National Research Council, National Materials Advisory Board, 2101 Constitution Ave. NW, Washington, DC 20418 USA

High-performance and cost-effective materials are needed to address many of the nation's high-visibility needs. Many technologies to battle terrorism, for example, can only be implemented if they are cost-effective. Such advanced equipment as night-vision goggles currently cost more than most local law-enforcement organizations can afford, and containers for checked baggage that are hardened against explosions are currently too heavy and too costly for widespread use. Other critical needs such as crime prevention, elder care, counterfeit deterrence, public health, education, and environmental protection can all utilize a variety of advanced materials–but only if they perform a highly specific function (or more likely, perform multiple functions) and are reliably and reasonably priced.

3:40 PM Break

3:50 PM
Hot-Formable, Age-Hardenable, Low-Cost Aluminum Alloys: *Eric M. Taleff*[1]; Jun Qiao[1]; [1]The University of Texas at Austin, Mechl. Eng., Matls. Sci. & Eng. Program, 160 E. Dean Keeton St., Austin, TX 78705 USA

A basis has been developed for engineering a new group of aluminum alloys which exhibit high tensile ductility at hot temperatures and good age-hardening at lower temperatures. High tensile ductilities, 150 to 300%, are achieved at high temperatures and low strain rates ($\varepsilon . /D \leq 10^{13}\ m^{-2}$) with an alloying addition of Mg to Al, which induces solute drag creep. Solute drag creep provides a high strain-rate sensitivity of m = 0.3, which reduces the rate of neck formation and allows high tensile elongations to be achieved. Ternary alloying additions of Zn at concentrations up to 5 wt. pct. remain in solution during hot forming and do not significantly interfere with the increased ductilities from solute-drag creep. The ternary Zn additions provide significant age hardening at temperatures well below those associated with hot forming and yield a significant paint-bake response. Avoidance of the need for ultra-fine grain sizes is expected to keep the cost of these alloys low.

4:10 PM
Light Magnesium Constructions for Transportation Applications: *Adi Ben-Artzy*[1]; Illan Makover[1]; Gavri Cohn[1]; Avigdor Shtechman[1]; Arie Bussiba[2]; [1]Rotem Industries, Ltd., Metal Forming Grp., POB 9046, Beer-Sheva 84190 Isreal; [2]N.R.C.N, POB 9001, Beer-Sheva 84190 Isreal

In recent years the use of high-pressure die-cast magnesium components for automotive applications, has markedly increased. This casting technology became widely used due to high productivity, short cycle time of tools and longer tool life. In addition, Thixomolding and other new developments in casting processes allow thin wall and complex shapes parts to be produced in mass production. In cases where high strength to density ratio and long fatigue life are required (usually in automotive moving parts) wrought magnesium alloys become necessary. Wrought alloys also have suitable elongation, which is reflected by good energy absorption capacity-a property which safety engineers in the car industry are looking for. In order to manufacture transportation vehicles like a handicapped scooter, other small vehicles or light constructions, one must use hybrid combination of wrought and cast alloys, that would provide the appropriate combination of weight and strength. Extruded profiles (tubes and bars) and forged rigid parts can be joined together by welding to create such structures. The main difficulty is still to select the appropriate welding technology that would be suitable for different hybrid combinations and would provide the essential mechanical properties of the hybrid joints. Several hybrid combinations were tested, using different welding technologies. Extruded AZ31B and AZ80 wrought magnesium alloys where joined to forged AZ31B joints. High-pressure die-cast AM50 tubes were welded to AZ31B forged parts using various welding technologies. The joints were characterized by mechanical testing, by optical and scanning electron microscopy as well as by an acoustic emission method. It was found that reliable and proper mechanical response combined with good corrosion resistance, characterize some of the hybrid magnesium joints. The optimum combination of alloys, welding technology and process parameters, would establish the final needed properties of the hybrid light magnesium construction. The use of hybrid, light-magnesium structures opens a wide horizon for the use of magnesium in transportation applications.

4:30 PM
New Magnesium Alloys for Automobile Applications: *D. Eliezer*[1]; [1]Ben Gurion University of the Negev, Dept. of Matls. Eng., Beer Sheva 84105 Israel

Magnesium is the lightest of all the commonly used metals and is thus very attractive for transportation applications. It also has other desirable features including reasonable ductility, better damping characteristics than aluminium and excellent castability. Because of increasing pressures to improve automotive gas mileage in most parts of the world, in combination with the required property combinations of the automobile industry, use of magnesium in cars and light trucks is increasing significantly. Magnesium alloy components are usually produced by various casting processes. The most applicable methods are high-pressure die-casting and gravity casting, particularly sand and permanent mold casting. Other relevant production technologies are: Squeeze Casting, Thixocasting and Thixomolding. This paper will evaluate the necessity of magnesium alloys development for expanding the current magnesium applications in the automotive industry. The paper will also address the correlation between the alloy develop-

ment and the production technologies of the actual components. Some achievement in alloys development will be described.

4:50 PM
Fabrication of Cu-Based 3-D Parts by Direct Laser Sintering: *Zhu Hai Hong*[1]; [1]National University of Singapore, Dept. of Mechl. Eng., 10 Kent Ridge Crescent 119260 Singapore

The direct laser sintering is a layered manufacturing process which can produce any desired three-dimensional parts with simple or complex shape. Recent research in this area has focused on the direct laser sintering of metal powder. Cu-based metal systems are the active research area in direct laser sintering because of its good thermal performance and high electrical conductivity. This paper reports on research work in direct laser sintering of a multiphase Cu-based powder in details. A composite metal powder consisting of Cu, Ag and P was successfully sintered in a self-developed Rapid Prototyping machine in ambient atmosphere at room temperature. 68.9% theoretical density and average surface roughness Ra 14-16mm were achieved with negligible distortion and shrinkage. Some 3-D parts have been fabricated successfully. Furthermore, the influence of the amount of liquid phase on mechanical properties, such as tensile strength and impact toughness were also investigated in this paper.

Hume-Rothery Award Symposium: CALPHAD and Alloy Thermodynamics: Thermodynamic Databases and Assessments

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Robert D. Shull, NIST, Magnetic Materials, Boyds, MD 20841-9015 USA

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February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Gerhard Inden, Max Planck Institut für Eisenforschung, GmBH, Max Planck Strasse 1, Postfach 140 444, Düsseldorf 40237 Germany; Frederick H. Hayes, University of Manchester/UMIST, Matls. Sci. Ctr., Grosvernor St., Manchester M1 7HS UK

2:00 PM Invited
Thermodynamic Tools for Alloy Design: *Joanne L. Murray*[1]; [1]Alcoa Technical Center, TPAL, 100 Technical Dr., Alcoa Center, PA 15069 USA

CALPHAD-type thermodynamic calculations of phase diagrams are now one of several computational tools for microstructure simulation. The eventual goal is to span the length scales from the atomic level to the macroscopic level and relate microstructure to properties and performance. Work on precipitation reactions during aging of 2xxx series aluminum alloys will be described.

2:30 PM Invited
Construction of a Thermodynamic Database for Ni-Base Superalloys: A Case Study: *Ursula R. Kattner*[1]; [1]NIST, Metall. Div., 100 Bureau Dr., MS 8555, Gaithersburg, MD 20899-8555 USA

Ni-base alloys are the most widely used superalloys in applications involving high temperatures and severe environmental conditions and are also among the most complex. To model the various processing steps improvements in thermodynamic and diffusion databases are required. The first thermodynamic database for superalloys was developed by Larry Kaufman and co-workers in the 70's. Since then, computational technology has constantly improved resulting in advanced model descriptions and better reproducibility of the experimental observations. A database for Ni-base superalloys with emphasis on single crystal alloys has been assembled for 10 components: Ni, Al, Co, Cr, Hf, Mo, Re, Ta, Ti, W. Since the number of subsystems for such a database is enormous, it is desirable to utilize assessments from literature. These assessments must be reviewed for consistency and, if necessary, partially remodeled. Experience from assembling this database with discussion of more systematic estimation procedures for the unknown quantities will be presented.

3:00 PM Break

3:30 PM Invited
The State of the Art of Assessment Techniques, Part I: *Suzana Gomes Fries*[1]; *Bo Sundman*[2]; Hans Leo Lukas[3]; [1]ACCESS e.V., RWTH-Aachen, Intzestrasse 5, Aachen D-52072 Germany; [2]KTH, MSE, Stockholm SE-10044 Sweden; [3]MPI fuer Metallforschung, NMAM, Universitaet Stuttgart, Heisenberg Str 5, Stuttgart D-70569 Germany

The CALPHAD technique has reached maturity. It started from the vision to combine data from thermodynamics, phase diagram and atomistic properties like magnetism into a unified model. It is now a powerful method in a wide field of applications. The successful use of CALPHAD comes from the development of multicomponent databases which describe many different kinds of experimental data in a consistent way. The construction of these databases is still a very demanding task, requiring expertise and experience. There are quite a lot of subjective factors in the decisions when judging and selecting between redundant experimental data which are the most trustful. Furthermore the growing range of applications of these databases increase the feedback and several corrections and modifications are required. The development of new models and the rapid advancement of atomistic calculations make the assessments techniques very dynamic and challenging. The assessment experiences collected since many by the three authors, which have differnt backgrounds, will be reported.

4:00 PM Invited
The State of the Art of Assessment Techniques, Part II: *Bo Sundman*[1]; Suzana Gomes Fries[2]; Hans Leo Lukas[3]; [1]KTH, MSE, Stockholm SE 100 44 Sweden; [2]Access, Inc., Inzelstr 5, Aachen Germany; [3]MPI, PML, Heisenbergstr 5, Stuttgart Germany

The Calphad technique is now a powerful method in a wide field of applications where calculated Gibbs energies and derivatives thereof are used to calculate properties and simulate transformations of multicomponent phases. Chemical potentials and the thermodynamic factor, the second derivative of the Gibbs energy, is used in diffusion simulations. The driving forces of metastable phases are used to simulate the evolution of microstructures In solidification simulations the segregation and fractions of different solid phases as well heat capacities and entropies can be obtained during slow as well as rapid solidification. Whenever the thermodynamic description of a phase is required, the Calphad method can be applied. The construction of these databases is a demanding task, requiring expertise and experience and there is still quite a lot of subjective factors involved when judging and selecting between redundant experimental data which are the most trustful. The growing range of applications increase the feedback and several corrections and modifications are required. The development of new models and the rapid advancement of atomistic calculations makes the assessments techniques very dynamic and challenging. Some of the assessment experiences the three authors has collected, since many years, will be reported in this presentation.

4:30 PM Invited
An Associate Model and Thermodynamic Database for Dilute Solutions of Oxygen and Metals in Molten Iron: *In-Ho Jung*[1]; Sergei Degterov[1]; Arthur Pelton[1]; [1]Ecole Polytechnique, Matls. Eng., PO Box 6079, Sta. "Downtown", Montreal, Quebec H3C 3A7 Canada

In the classical interaction parameter formalism of Wagner, the solute atoms are assumed to mix randomly. However, for solutions of oxygen and highly reactive metals M = Ca, Mg, Ba, Al,.... in molten iron, there is a strong tendency for the formation of associated solute "molecules" MO. When this is taken into account, the model is greatly simplified. Usually only one temperature-independent parameter is required to reproduce all experimental deoxidation equilibria and oxygen activities at all measured compositions and temperatures. The deoxidation curves for M = Ca, Mg are elucidated for the first time. A database has been prepared with evaluated parameters for M = Al,B,Ba,Ca,Ce,Cr,Hf,La,Mg,Mn,Nb,Nd,Ta,Ti,Th,Si,V,Zr.

International Symposium on Science and Technology of Interfaces in Honor of Dr. Bhakta Rath: Interface Effects

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Physical Metallurgy Committee, Superconducting Materials Committee, Jt. Mechanical Behavior of Materials, Titanium Committee

Program Organizers: Sreeramamurthy Ankem, University of Maryland, Department of Material & Nuclear Engineering, College Park, MD 20742-2115 USA; I. Ovidko, Russian Academy of Sciences, Institute of Problems of Mechanical Engineering, Laboratory for Theory of Defects in Materials, St. Petersburg 199178 Russia; Chandra Pande, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5000 USA; S. Ranganathan, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India

Monday PM — Room: 617
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: John Cahn, NIST, Matls. Sci. & Eng. Lab., 223/A153, Gaithersburg, MD 20899-8555 USA; S. Ranganathan, Centre for Advanced Study, Dept. of Metall., Bangalore 560012 India

2:00 PM Invited
Interpretation of Surface Relief Effects in Terms of Interphase Boundary Structure: *Hubert I. Aaronson*[1]; Barrington C. Muddle[2]; Jian Feng Nie[2]; [1]Carnegie Mellon University, Matls. Sci. & Eng., Pittsburgh, PA 15213-3890 USA; [2]Monash University, Sch. of Phys. & Matls. Eng., Monash University, Victoria 3800 Australia

During a recent symposium on the mechanisms of the massive transformation (MT), the central focus was on the structure of micron-size planar massive:matrix boundaries formed by irrational orientation relationships and habit planes at which misfit-compensating defects could not be detected even with HRTEM. Whereas most participants considered these boundaries to be incoherent, some described them as partly coherent with interfacial structures yet to be resolved. Although surface relief effects (SRE) of the invariant plane strain or tent types are considered to be absent during the MT, such effects can be discerned in published micrographs of Fe-Ni and Ag-Cd alloys. On the view of Christian (1962) that such SREs result from passage of partially coherent boundaries, the presence or absence of such effects should be a useful test for boundary structure. However, this test must be applied with caution. Partly coherent boundaries should not yield such SREs when: (i) there is no change in stacking sequence across the boundaries; (ii) successive "shears" mutually accommodate; (iii) terraces of growth ledges are nearly parallel to the specimen surface; (iv) the terrace plane is of CSL type (Nie and Muddle, 2000); and (v) all interfaces of product phase crystals have their equilibrium interfacial structure (W. Z. Zhang, 1999).

2:25 PM Invited
Thermodynamics of Interfaces in Mechanically Alloyed Metals: *Harshad Kumar Dharamshi Hansraj Bhadeshia*[1]; [1]University of Cambridge, Matls. Sci. & Metall., Cambridge CB3 7EQ UK

Normal thermodynamic theory for solutions begins with the mixing of component atoms. Many solutions are, however, prepared by mixing together lumps of the components, each of which might contain millions of identical atoms. In previous work, Badmos and Bhadeshia examined the way in which a solution evolves from these large clusters of components, from a purely thermodynamic point of view. They predicted that solution formation by the mechanical alloying of solid components cannot occur unless there is a gain in coherency as the particles become small. The nature of the barrier to mechanical alloying was discovered. There is also the possibility of a metastable state prior to the achievement of full solution, when the component atoms prefer like-neighbours. The evolution of the interface as mechanical alloying proceeds is investigated in this paper, together with implications on the formation of colloidal suspensions.

2:50 PM Invited
Diffusion at Zero-Flux Multicomponent Interfaces: *Martin E. Glicksman*[1]; Afina O. Lupulescu[1]; [1]Rensselaer Polytechnic Institute, MS&E Dept., CII-9111, Troy, NY 12180-3590 USA

Multicomponent diffusion gives rise to zero flux planes, ZFP's, where the flux of a component vanishes. The conditions for developing ZFP's in multicomponent alloys are now well understood through the works of Morral and co-workers. In this presentation we analyze dynamics of multicomponent diffusion near stationary ZFP's. In contrast to normal "global" component mixing that occurs in binary diffusion couples, the average equilibrium composition is achieved with a stationary ZFP by release of a pair of coupled diffusion waves. Curiously, each wave independently establishes the average concentration near the Matano interface. A stationary ZFP always prevents net transport of the component across the diffusion zone, which has both practically interesting implications for the design and lifetime of multicomponent films, coatings, and claddings that resist loss of a component into the substrate. Flux behavior is discussed for combinations of Cr-Al-Ni ternary alloys chosen near the composition 10at.%-Cr, 10at.%-Al, balance Ni.

3:15 PM Invited
Relationships among Interface Diffusion, Energies and Solute Segregations in Materials: *Devendra Gupta*[1]; [1]IBM, T. J. Watson Rsrch. Ctr., PO Box 218, Yorktown Heights, NY 10598 USA

During the last several decades, a strong connection among the important properties of interfaces, notably, the energy, solute segregation and diffusion has been firmly established. We will discuss the methodology and the thermodynamical analysis of the diffusion measurements to obtain these parameters and illustrate them by results obtained in a variety of materials. Investigations carried out in pure polycrystalline metals have yielded grain boundary energies comparable to those directly measured. Furthermore, we will discuss the role of solute segregation at grain boundaries in alloys in altering diffusion. From the perturbations caused, solute segregation parameters,the enthalpy and the entropy of binding, have been extracted and levels of solute concentrations estimated. Finally, it will be shown that similar analyses when applied to complex materials' interfaces, e.g. the Ni_3Al intermetallic compound, the PbSn eutectic alloy and the Ag-piezoelectric ceramic system, also result in acceptable values of energies and segregation factors.

3:40 PM
Effect of Irradiation-Induced Interfaces on Thin Film Stability: *Paolo M. Ossi*[1]; [1]INFM-Politecnico di Milano, Dip. di Ingegneria Nucleare, via Ponzio, 34/3, Milano, MI I-20133 Italy

Dramatic amorphisability differences are observed in chemically similar compounds, irradiated under identical conditions. The atomistic Segregation-Charge Transfer (SCT) model for the structural stability of binary systems, bombarded under conditions such that dense collision cascades form is presented. At each cascade-matrix interface an off-equilibrium compositional profile develops, following interface enrichment in one compound constituent. Concurrently, a local electronic density profile develops, which can be non-equilibrium over the relevant cascade quenching timescale. Relaxation to (meta)stable equilibrium is schematised by charge transfer (CT) reactions, each involving two dissimilar atoms of the initial compound (IC), that generate an effective compound (EC) dimer. Every EC is compared to the corresponding IC, considering the CT associated energy cost, the enthalpy change at EC formation, and the local volume change, related to strain effects. For a meaningful set of compounds, both metallic and non-metallic, threshold parameter values separate amorphised materials from materials remaining crystalline upon irradiation.

4:00 PM Invited
The Role of Liquid-Solid Interface during Melting and Solidification during Surface Alloying and Dissimilar Metal Welding by Laser: *Kamanio Chattopadhyay*[1]; Gandham Phanikumar[1]; Pradip Dutta[2]; [1]Indian Institute of Science, Metall., Bangalore, Karnataka 560012 India; [2]Indian Institute of Science, Mechl. Eng., Bangalore, Karnataka 560012 India

Solidification is a first order transformation, where the transformation and the resulting microstructural changes take place by the movement of the transformation interface. The thermal and solutal conditions prevailing at the solid liquid interface and the thermodynamic constraints that exist at the interface control this movement and determine the evolution of the solidification microstructure. In the case of laser surface alloying and dissimilar metal welding, the situation is further complicated by the rapid heat transfer and fluid flow. The microstructure is often inhomogeneous and contains different concentration domains which are temporally varying. Thus the interface often experiences a changing condition which affects its motion and hence the microstructure. In this presentation, we shall give examples from the work done in our laboratory to elucidate these complexities. Attempts will be made to relate the origin of the complexities in the microstructure to the changing interface conditions.

4:25 PM
Interphase Interfaces and the Mechanisms of the α to γ_m Massive Transformation in Ti-Al Alloys: Ping Wang[2]; Veer Dhandapani[3];

Mukul Kumar[4]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Brown University, Div. of Eng., Providence, RI 29212 USA; [3]Motorola, Adv. Prod. R&D Lab., Austin, TX 78721 USA; [4]Lawrence Livermore National Laboratory, L-356, Livermore, CA 94550 USA

The massive-matrix interphase interfaces associated with the α to γ_m massive transformation in Ti-(46-48)Al alloys were studied. Special experiments were performed to arrest the transformation at an early stage. Nucleation and growth kinetics were determined and the former compared with calculations for various nuclei shapes using classical nucleation theory. Orientation relations between the γ_m and parent α (retained α_2) phases were determined using EBSD in an SEM and by electron diffraction, and the interphase interfaces characterized by two-beam bright-field/weak-beam dark-field TEM and HRTEM. The results reveal that the γ_m nucleates at grain boundaries with a low-index orientation relation and coherent interface with one parent grain, but grows into the adjacent grain with a high-index/irrational orientation relation. The growth interfaces between the two phases are generally free of misfit dislocations and consist of curved parts as well as planar facets whose macroscopic habit varies from high-index/irrational to low-index orientation. On an atomic scale the growth interfaces are often found to be faceted along low-index planes with steps, but are incoherent with respect to the parent grain into which growth occurs. The implications of these results on the nucleation and growth mechanisms associated with the α to γ_m massive transformation will be discussed. The authors are grateful for support of this research by the National Science Foundation under grants DMR-9224473 and 9731349, Dr. Bruce MacDonald, Program Monitor.

4:45 PM
In-Situ Transmission Electron Microscopy of Combustion Synthesized TiC-NiTi Composites: *E. R. Strutt*[1]; T. Radetic[2]; M. A. Meyers[1]; [1]University of California-San Diego, Dept. of Mechl. & Aeros. Eng., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA; [2]National Center for Electron Microcopy, Lawrence Berkeley National Lab., Berkeley, CA 94720 USA

In-situ transmission electron microscopy was performed on combustion synthesized TiC-NiTi composites in order to observe how stiff ceramic inclusions affect the martensitic transformations in NiTi. The mechanisms of nucleation and growth of martensitic B19' have been elucidated. The effect of thermal cycling on the mechanism of transformation has been studied. The similarities and differences of the transformation mechanism between reinforced and unreinforced Ti-rich NiTi alloys are discussed in terms of a possible optimum dislocation density for the formation of the R-phase in TiC- NiTi composites. Research supported by the US Army Research Office under contracts DAAH04-95-1-0236 and DAAH04-96-1-0376, and by the Director, Office of Science, Office of Basic Energy Sciences, of the US Department of Energy under Contract No. DE-AC03-76SF00098. The transmission electron microscopy work was performed at the National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, University of California, Berkeley, CA 94720.

Lead-Free Solders and Materials Issues in Microelectronic Packaging: Interfacial Reactions and Intermetallics

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Materials Committee, Electronic Packaging and Interconnection Materials Committee
Program Organizers: Srini Chada, Motorola, Department APTC, Fort Lauderdale, FL 33322 USA; Darrel R. Frear, Motorola, Tempe, AZ 85284 USA; Sung-Ho Jin, Lucent Technologies, Bell Laboratories, Murray Hill, NJ 07974 USA; Sung Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Michael J. Pfeifer, Motorola, Northbrook, IL 60062 USA; Martin Weiser, Honeywell Electronics Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Monday PM Room: 612
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Srini Chada, Motorola, APTC, 8000 W. Sunrise Blvd., Plantation, FL 33322 USA; Sung K. Kang, IBM, T. J. Watson Rsrch. Ctr., PO Box 218, Yorktown Heights, NY 10598 USA

2:00 PM Opening Remarks

2:05 PM Invited
Mechanical and Electrical Properties of Lead(Pb)-Free Solder Joints: *Sung K. Kang*[1]; M. J. Yim[2]; W. K. Choi[1]; D. Y. Shih[1]; [1]IBM, T. J. Watson Rsrch. Ctr., PO Box 218, Yorktown Heights, NY 10598 USA; [2]Korea Advanced Institute Science & Technology, Dept. of Matls. Sci. & Eng., Taejon Korea

The mechanical and electrical properties of several Pb-free solder joints were investigated in terms of their interfacial reactions, namely, the thickness and morphology of the intermetallic layers. In order to measure both the shear strength of a solder joint and its electrical resistance, a model joint was made by joining two "L-shaped" copper coupons. Three Pb-free solders investigated are Sn-3.5%Ag, Sn-3.8%Ag-0.7%Cu, and Sn-3.5%Ag-3%Bi (all in weight %), combined with two surface finishes, Cu and Au/Ni(P). By varying the reflow time and aging time, the thickness of the intermetallic layers grown at the joint interface was changed from a few micron to more than 10 micron. The morphology of the intermetallic compounds was significantly influenced by the solder composition as well as the choice of a surface finish layer. The correlations between the mechanical and electrical properties of Pb-free solder joints and their interfacial reactions are made. The microstructure of the solder joints has also been investigated to understand the characteristic behavior of the Pb-free solder joints.

2:30 PM
Nucleation Kinetics of Cu_6Sn_5 by Reaction of Molten Tin with a Copper Substrate: *Robert A. Gagliano*[1]; Morris E. Fine[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The nucleation kinetics of η-phase (Cu_6Sn_5) intermetallic compound is being investigated by hot dipping copper coupons in molten tin for several very short times, beginning with one second, at temperatures varying from 240ºC to 300ºC. In the SEM, the Cu_6Sn_5 phase appears as small, rounded bumps of varying size jutting out from the surface of the copper. There are generally more bumps present at lower reaction temperatures than at higher reaction temperatures. The role of coarsening is being examined by comparing the one sec. reaction time results with results for 2 and 3 sec. reaction time. Quantitative measurements of the experimentally determined nucleation rate/unit area will be presented and compared with theoretical predictions over the temperature range investigated. Activation energies, kinetic parameters, and nucleation models will also be discussed.

2:50 PM
The Effect of Cu on Interfacial Reaction in Lead-Free Systems: *Ka Yau Lee*[1]; Ming Li[1]; [1]Institute of Materials Research & Engineering, 3 Research Link, 117602 Singapore

The interactions of two lead-free solders: Sn-3.5Ag and Sn-3.0Ag-0.5Cu with Ni/Au metallization after liquid- and solid-state reaction have been investigated. Both systems were comparable in ball shear strength and performed better than SnPbAg system after solid-state annealing. Unlike SnPbAg system after solid-state reaction, re-precipitation of Au as $Au_{0.5}Ni_{0.5}Sn_4$ at the interface was not observed in both lead-free systems. Comparison was also made on interfacial morphologies of Cu-Sn-Ni intermetallic compound formed in SnAgCu system and Ni_3Sn_4 found in SnAg system. Thick layer of acicular Cu-Sn-Ni with large aspect ratio formed after liquid-state reaction. In contrast, the intermetallic compound became denser and more faceted with solid-state annealing time. The compound also grew slower than Ni_3Sn_4, with an activation energy of ~200 kJ/mol. The preferential formation of this Cu_6Sn_5-based compound was explained with the help of ternary phase diagram.

3:10 PM
A Study of the Effects of Solder Volume on the Interfacial Reactions in Solder Joints using Differential Scanning Calorimetry Technique: *Won Kyoung Choi*[1]; Sung K. Kang[1]; Da-Yuan Shih[1]; [1]IBM, T. J. Watson Rsrch. Ctr., PO Box 218, Yorktown Heights, NY 10598 USA

The interfacial reactions during soldering have been extensively investigated in the literature by employing various experimental techniques such as conventional cross-sectional metallography, scanning electron microscopy (SEM), electron microprobe analysis, X-ray diffraction and others. However, these techniques are time consuming and require special sample preparation. In this study, the differential scanning calorimetry (DSC) technique was employed to understand the interfacial reactions. In DSC, the simulation of a soldering process and the analysis of the interfacial reactions can be performed at the same time. The measurements of peak temperatures and heat involved provide useful information about the interfacial reactions such as the amount of intermetallic compounds (IMC) formed and that of Cu

dissolved. Sn-plated Cu balls were prepared as samples for the DSC study. Cu balls with a different Sn thickness were used to investigate the solder volume effect. In addition, a Ni diffusion barrier layer (electroless or electrolytic) was introduced between Cu and Sn to examine its role on the dissolution kinetics of Cu into a molten Sn. The conventional metallography combined with SEM was also used to verify the DSC results.

3:30 PM
Isothermal Solidification of Cu/Sn Diffusion Couples to Form Thin Interconnects: *Judy Sooyean Kang*[1]; Robert A. Gagliano[1]; Gautam Ghosh[1]; Morris E. Fine[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The ever increasing demand for higher density of input/output (I/O) connections in electronic devices require smaller and smaller interconnects. Utilizing a lead-free approach for chip to chip carrier interconnects of a size scale that allows a very high level of integration is a viable approach to this problem. Recently, we have applied the concept of isothermal solidification to form a joint as thin as 30 µm between two Cu plates. Using pure Sn and a relatively low processing temperature, 275 to 325°C, we have been able to form intermetallic based joints containing only Cu_3Sn. The reaction between liquid Sn and Cu leads to formation of Cu_6Sn_5 and Cu_3Sn intermetallics which continue to grow until all Sn is consumed to form a solid bond. Depending on the extent of further reaction desired, the end product may be either Cu_6Sn_5+Cu_3Sn or only Cu_3Sn. The former requires less Cu. We will discuss the dynamics and morphology of Cu_6Sn_5 and Cu_3Sn intermetallic growth which are essential for controlling the isothermal solidification process.

3:50 PM Break

4:05 PM Invited
Textured Growth of Cu/Sn Intermetallic Compounds: *Kithva Prakash Hariram*[1]; Thirumany Sritharan[1]; [1]Nanyang Technological University, Sch. of Matls. Eng., Nanyang Ave. 639798 Singapore

The growth of Cu-Sn intermetallic compounds (IMC) at the interface of Cu and molten Sn-Pb solder alloys is studied over a range of temperature. X-ray studies show a very strong (10.2) and (10.1) peaks of η-phase (Cu_6Sn_5) when the Sn content was high, at all temperatures. In the low Sn solder (27Sn-73Pb), the η-phase peaks were absent at the two high temperatures but the (2 12 0) peak of ε-phase (Cu_3Sn) was prominent. (10.1) and (002) pole figures were constructed, using the conventional texture goniometer, for η-phase and ε-phase, respectively. The growth directions were identified to be <101> and <102> for η-phase and <102> and <031> for ε-phase, normal to the Cu surface. The growth directions do not change with the morphology and the layer thickness. The morphology of the η-phase layer varies gradually from a cellular film with rugged interface to a dense film with scalloped interface as the Pb content, temperature and reaction time increase. The ε-phase was dense and planar.

4:30 PM
Interfacial Reactions of In-Containing Solder Alloys on the Au Deposited Substrates Ni and Cu: *Chang Youl Lee*[1]; *Seung Boo Jung*[1]; *Chang Jae Shur*[1]; [1]Sung Kyun Kwan University, Metlgcl. & Matls. Eng., 300 Chunchun-Dong, Jangan-Gu, Suwon, Kyonggi-Do 440-746 Korea

The reliability of the solder joint is affected by type and extent of the interfacial reaction between solder and substrates. Therefore, understanding of intermetallic phase produced by soldering in electronic packaging is essential. Among many solder alloys, In-alloy solders are used as low-melting temperature solder. In the present work, the interfacial reactions of In solder alloy on the Au deposited substrates (Cu and Ni) are investigated. Also the wettability of liquid In solder on the various substrates was determined from meniscope method. Experimental results showed that the intermetallic compounds are observed Cu11In9, Ni10In27 and AuIn2 for different substrate respectively. Additionally, the growth rate of these intermetallic compounds were increased with the reaction temperature and time. We found the growth of this intermetallic compound in aging followed the parabolic law, which indicates that the growth is diffusion-controlled.

4:50 PM
Solder/UBM Reaction Characteristics in Optical Packages: Jong-Hwan Park[1]; Jong-Hyun Lee[1]; *Yong-Seog Kim*[1]; [1]Hong-Ik University, Matls. Sci. & Eng., 72-1 Sangsu Dong, Seoul 121-791 S. Korea

Au/Pt/Ti UBM has been used frequently for Au-Sn solder pad in optical packages. Under a typical reflow condition, the diffusion barrier layer, Pt, was found to dissolve into the solder completely, exposing the adhesion layer of poor wettability to the solder. The dissolution is due to high reflow soldering temperature as well as formation of complete solid solution with Au. In this study, several metals and intermetallics were employed as the diffusion barrier layer and their dissolution and reaction kinetics at the solder/UBM interface were investigated. Preliminary investigations indicated that Ni, Cu, Cu6Sn5, and Ni3Sn4 layers dissolve into the solder readily and form intermetallics at the interface. Other diffusion barrier materials investigated include refractory metals such as W, Mo, and Nb. The dissolution and reaction kinetics of the metal layers with the solder were examined and fracture energy of the solder interconnection was measured by a ball shear tester.

5:10 PM
Failure Mechanism of Lead-Free Solder Joints for Flip Chip Packages: Fan Zhang[1]; *Ming Li*[1]; Balakrisnan Bavani[1]; William T. Chen[2]; *K. Y. Lee*[1]; [1]Institute of Materials Research and Engineering, Opto- & Elect. Sys. Cluster, 3 Research Link 117602 Singapore; [2]ASE (US), Inc., 3590 Peterson Way, Santa Clara, CA 95054 USA

The failure mechanisms of SnAgCu solders on Al/Ni(V)/Cu under bump metallization (UBM) were investigated after multiple reflows and high temperature storage using ball shear test, fracture surface analysis, and cross-sectional microstructure examination. The results were compared with those of eutectic SnPb solder. It was found that in the SnAgCu system, the failure mode changed with the number of reflow from ductile failure inside the solder ball to brittle failure near the interface between intermetallic layer and UBM. The change in the failure mode was found relating to the degradation of UBM structure due to the severe interfacial reaction between solder and UBM layer. After high temperature storage, the solder joints failed inside solder ball in a ductile manner in both SnAgCu and SnPb systems. The reduction of shear strength was observed with the increase of the aging time, which was explained by the microstructure evolution of the solder material.

5:30 PM
The Phenomena of Electroless Ni-P Stripping and Dissolving in the Eutectic Sn-Bi Solder: *Chien Sheng Huang*[1]; Bi Lian Young[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., 101, Sect. 2 Kuang Fu Rd., Hsinchu 300 Taiwan

Interfacial reactions between electroless Ni-P (EN) and eutectic Sn-Bi solder was characterized. Joints of 42Sn-58Bi/Au/Ni-5.5wt%P/ Cu/ Al2O3 and 42Sn-58Bi/Au/Ni-12.1wt%P/Cu/Al2O3 were annealed at 145ºC and 185ºC for 30~180 minutes. EN deposited with a high pH value exhibited lower P content and larger nodules. After annealing, there are two interfacial intermetallics (Ni3Sn4 and Ni3P) formed between solder and EN. In the 42Sn-58Bi/Au/Ni-5.5wt%P/Cu joint, Ni3Sn4 was formed and stripped into the solder after annealed for long periods of time. The EN was first stripped from the initial interface and then dissolved into the Sn-Bi solder during annealing at 185ºC. In the 42Sn-58Bi/Au/ Ni-12.1wt%P/Cu joint, the growth of IMC was limited even annealed at either elevated temperature of 185ºC or long periods of 180 minutes. No EN stripping was found. It is believed that the tendency of EN stripping was related to the nodule size or surface roughness of EN. From the morphology of the interface between solder and EN, the mechanism of EN stripping and dissolving could be probed.

Magnesium Technology 2002: Magnesium Recycling and Environmental Issues

Sponsored by: Light Metals Division, Magnesium Committee, International Magnesium Association

Program Organizers: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA; Gerald S. Cole, Ford Motor Company, Ford Research Laboratories, Dearborn, MI 48121 USA; Rod Esdale; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA; Ramaswami Neelameggham, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Eric A. Nyberg, Pacific Northwest National Laboratory, Materials Processing Group, Richland, WA 99352 USA; Mihriban O. Pekguleryuz, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H9R 1G5 Canada; Bob R. Powell, General Motor Corporation, NAO Research and Development Center, Warren, MI 48090-9055 USA; Allen Schultz, Hatch, Mississauga, Ontario L5K 2R7 Canada

Monday PM — Room: 606
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: John N. Hryn, Argonne National Laboratory, 9700 S. Cass Ave., Bldg. 362, Argonne, IL 60439-4815 USA; Eric A. Nyberg, Pacific Northwest National Laboratory, Matls. Procg. Grp., 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

2:00 PM

Solubility of Fluorine in Molten Magnesium: *Kari Aarstad*[1]; Martin Syvertsen[2]; Thorvald Abel Engh[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., Alfred Getz vei 2b, Trondheim 7491 Norway; [2]SINTEF Materials Technology, Proc. Metall. & Cer., Alfred Getz vei 2, Trondheim Norway

As an alternative to the high global warming potential gas SF_6, oxidation and burning of liquid magnesium could be prevented by dissolving fluorine directly into the molten metal. The solubility of fluorine in molten magnesium was hence measured at temperatures between 700°C and 950°C. In these experiments, magnesium was equilibrated under argon in a MgF_2 crucible. Twice the furnace is evacuated to 1 mbar and then 99.99% argon is fed through. Samples of the equilibrated melt were taken using alumina tubes and a syringe. The fluorine solubility was determined employing the "Sintalyzer method", an electrochemical method developed at Sintef, Norway. As expected, the fluorine content in liquid magnesium is described by an exponential function of inverse temperature. Fluorine varies between 10 and 100 ppm by weight in the measured temperature interval.

2:30 PM

Progress towards Climate Friendly Magnesium Production and Casting: *Scott Charles Bartos*[1]; [1]US Environmental Protection Agency, Global Programs Div., 1200 Pennsylvania Ave. N.W., (6205J), Washington, DC 20460 USA

The US Environmental Protection Agency (EPA) is working cooperatively with the magnesium industry to reduce emissions of sulfur hexafluoride (SF6), a persistent and very potent greenhouse gas. EPA launched its domestic magnesium industry partnership in 1999 and is now proud to support the International Magnesium Association in its effort to identify and implement more environmentally friendly protective cover gas alternatives. This paper will discuss the impressive progress being made by EPA's US partners to protect the climate as well as other exciting initiatives that are taking place around the world.

3:00 PM

Recycling of Different Types of MG-Scrap: *Helmut Antrekowitsch*[1]; Gerhard Hanko[1]; Hubert Sommerhofer[1]; [1]University of Leoben, Non-Ferrous-Metall., Franz-Josef-Straße 18, Leoben, Styria 8700 Austria

The importance of magnesium recycling has become more evident in the last ten years. The more materials are recycled, the lower is the energy consumption and the fewer waste products will end up in landfills, in the water and in the air. Nowadays only high grade scrap such as gates, runners and drippings from die casting operations are used for the recycling process. For the coated and contaminated magnesium materials no economical and ecological processes exist. The department of nonferrous metallurgy at the university of Leoben, Austria, investigates different processes of melting and refining of these types of scrap. Concerning the reactivity of magnesium with other elements different techniques have to be tested. The European guideline on old automobiles, which gratifies that recycling processes considered in construction and design of new cars, relieve the winning of secondary magnesium.

3:30 PM Break

3:50 PM

The Successful Briquetting of Magnesium Chips and Turnings for Secondary Melting and Recycling: *David J. Roth*[1]; *Gerd Von Aschwege*[2]; [1]Altek, LLC, 314 Exton Commons, Exton, PA 19341 USA; [2]RUF, Gmbh & Co. KG, Tussenhausener StraBe 6, Zaisertshofen D-86874 Germany

The briquetting of magnesium chips and grindings has been successfully accomplished in Europe with the RUF two ram closed die system. Densities of 1.4-1.5 g/cm3 are achievable with this process allowing for safer handling by stabilizing and reducing the volatility of the fine magnesium particles. Government regulations and legislation on waste disposal have highlighted the importance of finding new, low-cost methods of recycling waste materials. Briquetting the fine material dramatically reduces the volume, making the shipping of the material safer, easier and more cost effective. This paper will review case studies on the recycling of the briquetted magnesium chips. It will discuss the equipment and the uses and remelt possibilities of the briquetted magnesium. It will also discuss the findings from an ongoing study partially funded by the European community to handle the magnesium chips produced by the automotive industry.

4:20 PM

Fluxless Refining of Clean Diecast Scrap for Noranda's AJ52 High Temperature Mg-Al-Sr Alloy: *Peter Forakis*[1]; Eric Richard[1]; Donald Argo[2]; [1]Noranda, Inc. Technology Centre, Metal Proc., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada; [2]Noranda, Inc. Technology Centre, Matls. Eng., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada

The AJ52 high temperature Mg-Al-Sr alloy developed by Noranda has shown its potential for use in automotive powertrain components requiring elevated temperature creep resistance and other high temperature mechanical properties through extensive property evaluations and casting trials. A factor that must be addressed in using the alloy, as for other alloys, is the recycling of trim and scrap components (type 1 scrap). Many of the alloying elements (most notably Sr, Ca and to a lesser extent the rare earths) used in the design of alloys for improved high temperature properties tend to react with the common fluxes used for recycling and refining the typical AZ and AM alloys. This results in the loss of needed alloying ingredients, excessive dross generation and can affect the overall quality of the refined metal. This study investigates, through laboratory and pilot plant molten metal trials, the loss of these alloying ingredients when in contact with fluxes and specifically the potential of fluxless refining techniques for bringing AJ52 type 1 scrap to a high level of metal cleanliness with minimal Sr losses.

4:50 PM

Application of Magoxide Method for Cleanliness Evaluation of Magnesium Alloys: *Boris Bronfin*[1]; Nataly Polyak[1]; Eli Aghion[1]; Carl Carlton Fuerst[2]; David Barris[3]; [1]Dead Sea Magnesium, Magnesium Rsrch. Div., PO Box 1195, Beer-Sheva 84111 Israel; [2]General Motors, Global R&D Ops., PO Box 9055, Warren, MI 48090-9055 USA; [3]General Motors, Non Ferrous Metals Worldwide Purch., PO Box 9015, Warren, MI 48090-9015 USA

A new method named "MagOxide" aiming at evaluation of the MgO and Al-Mn-Fe intermetallics in magnesium alloys was developed by DSM's Research Division. The newly developed method is based on wet chemistry procedure and is compatible with the Fast Neutron Activation Analysis (FNAA) technique. The method was successfully implemented on a variety of primary and recycled Mg alloys. The present paper aims at presenting the systematic correlation of results obtained by FNAA and "MagOxide" methods when primary and recycled Mg alloys of AZ and AM series were analyzed.

5:20 PM

Use of Fluoroketones in Cover Gases for Molten Magnesium: Dean S. Milbrath[1]; John G. Owens[1]; [1]3M Specialty Materials Laboratory, St. Paul, MN, USA

Abstract unavailable

Processing and Properties of Lightweight Cellular Metals and Structures
The MPMD Third Global Symposium
Fabrication Methods and Principles - Session II

Sponsored by: Materials Processing & Manufacturing Division, Jt. Computational Materials Science & Engineering, Powder Materials Committee, Jt. Processing Modeling Analysis & Control Committee, Surface Engineering Committee, Shaping and Forming Committee, Solidification Committee

Program Organizers: Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; T. Dennis Claar, Fraunhofer USA, Newark, DE 19716 USA; T. H. Sanders, Georgia Institute of Technology, Department of Materials Science and Engineering, Atlanta, GA 30332 USA

Monday PM Room: 205
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: David C. Dunand, Northwest University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA; Iver Anderson, Iowa State University, Ames Lab., 122 Metals Development Bldg., Ames, IA 50011-3020 USA

2:00 PM
Stabilization Mechanisms of Metal Foams by Ceramic Particles in Liquid-State Processing: *Y. Q. Sun*[1]; [1]University of Illinois, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA

The stabilization mechanisms of metal foams by ceramic particles in their liquid-state processing are determined by experiments. The focus is on the relative foam stabilizing effects of liquid viscosity and the wetting properties of the solid particles in the liquid. The stabilization of liquid metal foams by partially wetting and non-dissolving ceramic particles is simulated experimentally with liquid solutions with controllable surface tensions and continuously variable wetting contact angles. The results show that liquid foams with micrometer pore sizes can be stabilized with partially wetting particles alone and that there is an intermediate wetting angular range around 80° in which the liquid foam has the optimum stability. The key steps in the liquid-state processing of metal foams stabilized by solid particles are also identified by the experimental simulations.

2:25 PM
Rheological Changes of Cellular Structure in Foaming Metal: *Zhenlun Song*[1]; Steven Nutt[1]; [1]University of Southern California, Composites Ctr., 3651 Watt Way, VHE-602, Los Angeles, CA 90089-0241 USA

Controlling melt viscosity is a key process parameter in the production of metal foams by melt processing. In this work, rheological changes of molten aluminum during foaming were investigated. In this process, the aluminum charge is placed in a crucible and heated to the melting point. Mechanical agitation is introduced, and when suitable viscosity is achieved, a granular foaming agent is injected. Foam expansion ensues rapidly, and the melt viscosity is critical to achieve stable foaming and avoid slumping. Three types of cell shapes in the foam are considered: (1) near the sidewall of the crucible, where cells experience shear deformation, (2) near the top of the foaming melt, where cells experience compression deformation, and (3) near the bottom of the foaming melt, where cells are well-developed. A qualitative model of the process is proposed explain the observations, and matched to experimental data.

2:50 PM
The Preparation of Highly Porous Structures from Filamentary Nickel Powders: *Alexandre Yu. Zaitsev*[1]; David S. Wilkinson[1]; George C. Weatherly[1]; Thomas F. Stephenson[2]; [1]McMaster University, Matl. Sci. & Eng., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada; [2]Inco Technical Services Limited, 2060 Flavelle Boulevard, Mississauga, Ontario L5K 1Z9 Canada

Porous nickel structures are in high demand for battery, catalyst and filter materials applications. Traditionally such structures are made by sintering of fine filamentary nickel powders. However, the strength of such structures is rather low, when compared for example with Ni foams of similar density. Our work is focused on increasing the strength through improved processing. In particular, colloidal processing techniques can be used to improve the powder particle distribution and, hence, the strength of the final sintered structure. By dispersing Ni powder in water prior to introducing a binder, better separation of particles and break-up of conglomerates is achieved. The addition of dispersant agents further improves the particle distribution. This method also improves control of the slurry viscosity and the green density of the nickel porous body. The structure of battery plaque prepared according to the new technique is much more uniform than the structure of the conventional plaque.

3:15 PM Break

3:35 PM
Novel Lightweight Open Cell Metal Foam Process and Resulting Properties: *Amit K. Ghosh*[1]; Xingcun Tong[1]; [1]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

A new low cost process has been developed for fabricating both stochastic and periodic open cell metal foam structures by direct deposition of solidifying liquid wires, droplets and spray, and simultaneously welding them in-situ. The process is capable of producing a wide range of pore spaces from microns to millimeters and amenable to computer-aided manufacturing technique. Open cell structures with closed outer surfaces and solid face sheets have been produced. The process is also capable of producing graded porosity, and various internal geometries when deposition is carried out over removable geometric inserts. Samples with a variety of internal geometries were produced from several aluminum alloys to create multiscale porosity levels, and tested under compression for elastic modulus and strength. The results show that plateau strength and modulus can be influenced by the choice of internal geometry, which are not uniquely predicted by ligament bending-based models. Simple models demonstrate that the orientation of internal columns and walls are directly connected with the measured deformation behavior. (This research was supported by the US Office of Naval Research, under grant: DOD-G-N00014-97-1-0510, Program Manager: Dr. S. G. Fishman).

4:00 PM
Processing of IN-718 Lattice Block Castings: *Mohan Hebsur*[1]; [1]NASA Glenn Research Center, OAI, Brookpark Rd., Cleveland, OH 44135 USA

Recently a low cost casting method known as lattice block casting has been developed by JAM Corp, Boston MA for engineering materials such as Aluminum and Stainless steels that has shown to provide very high stiffness and strength with only a fraction of density of the parent alloy. NASA-Glenn has initiated a research to investigate lattice block castings of high temperature Ni-base superalloys such as IN-718 for lightweight nozzle applications. Initially there were numerous problems in making and assembling the wax patterns of our design. It was decided to try a new wax, less fragile and a lot stronger than anything tried earlier. Not only did the molds filled easily with the new formula, also the parts came out of the molds without breaking and were easy to assemble. Totally ten Inconel-718 lattice block castings (each measuring 6 inch wide x 12 inch long x 0.5 inch thick) have been successfully produced by MCT Inc., Manchester NH using their patented counter gravity casting techniques. Details of the processing and resulting microstructures will be discussed. Post casting processing and evaluation of system specific mechanical property of these specimens are in progress. This work is in support of the Ultra Efficient Engine Technology (UEET) lightweight nozzle program.

4:25 PM
Processing of Controlled Porosity Titanium-Based Materials: *T. Dennis Claar*[1]; Ulf Waag[2]; Hartmut Goehler[2]; Donald M. Kupp[1]; [1]Fraunhofer USA, Ctr. for Mfg. & Adv. Matls., 501 Wyoming Rd., Newark, DE 19716 USA; [2]Fraunhofer IFAM, Powder Metall. & Composite Matls.-Dresden, Winterbergstr. 28, Dresden 01277 Germany

Alloys and intermetallic compounds based on titanium are used extensively in aerospace and biomedical applications, based on their high strength, light weight, and excellent corrosion resistance. To extend the range of applications, designers and engineers are now seeking titanium-based materials with controlled porosity. Ultra-lightweight, porous titanium alloys and intermetallics are being fabricated based on technologies being developed at the Fraunhofer Institute and Fraunhofer USA Center for Manufacturing and Advanced Materials, located in Dresden, Germany and Newark, DE, respectively. Powder metallurgy processing routes are being utilized to fabricate hollow metal spheres and open-celled foams in titanium, Ti-6Al-4V alloy, and TiAl and TiAl3 intermetallics. The processing, microstructures, and selected properties of these controlled-porosity materials will be presented, along with a discussion of potential applications in the aerospace, biotechnology, and other industries.

Processing and Properties of Lightweight Cellular Metals and Structures
The MPMD Third Global Symposium

Poster Session: Monday 5:30 PM – 8:00 PM

Second International Symposium on Ultrafine Grained Materials: Processing and Structure - II

Sponsored by: Materials Processing & Manufacturing Division, Shaping and Forming Committee
Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; Terence G. Langdon, University of Southern California, Department of Mechanical Engineering, Los Angeles, CA 90089-1453 USA; Terry C. Lowe, Technanogy, Newport Beach, CA 92627 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; Michael Jeremi Saran, Case Western Reserve University, Cleveland, OH 44106 USA; S. Lee Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Monday PM — Room: 210
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: K. Ted Hartwig, Texas A&M University, Mechl. Eng., 319 Engineering Physics Bldg., Spence St., College Station, TX USA; Ke Lu, Institute of Metal Research, Shenyang Natl. Lab. for Matls. Sci., Chinese Academy of Sciences, Shenyang 110016 China

2:00 PM Keynote
Recent Developments of SPD Processing of Fabrication of Bulk Nanostructured Materials: *Ruslan Z. Valiev*[1]; [1]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Matls., 12 K. Marks Str., Ufa, Bashkortostan 450000 Russia

During the last decade severe plastic deformation (SPD) techniques have been successfully used for the fabrication of nanostructures in various bulk metallic materials. This paper presents several results from recent investigations of SPD materials focussing on two main objectives: modelling and experimental works on SPD techniques, namely equal-channel angular (ECA) pressing, high pressure torsion (HPT) and their modifications aiming to produce homogeneous nanostructures in bulk large-size billets and to process hard-to-deform and low-ductile materials, e.g. Ti, W and their alloys; determination of critical SPD (strain amount, temperature, applied pressure etc.) and microstructure (types of grain boundaries, defect structures) parameters resulting in enhancement of properties in as-processed materials. Examples of obtaining very high strength and ductility, enhanced superplasticity and toughness in SPD materials are considered and their origin is discussed.

2:25 PM Invited
Equal Channel Angular Pressing of Steels (BCC), Al Alloys (FCC) and Pure Titanium (HCP): *Dong Hyuk Shin*[1]; Si-Young Chang[1]; Yong-Seog Kim[2]; Kyung-Tae Park[3]; [1]Hanyang University, Dept. of Metal. & Matls. Sci., Ansan, Kyunggi-Do 425-791 Korea; [2]Hong-Ik University, Dept. of Metal. & Matls. Sci., Seoul 121-791 Korea; [3]Taejon National University of Technology, Dept. of Adv. Matls. Sci. & Eng., Taejon 300-717 Korea

Several aspects of severe plastic deformation (SPD), such as grain refinement, microstructural modification, superplasticity and mechanical properties, etc, of steels, aluminum alloys and pure titanium, were examined. The grain refinement process of steels and aluminum alloys during SPD appeared to be due to slip band formation, but that of titanium seemed to be entirely different. In addition, SPD was found to be very effective in modifying microstructure. In the low carbon steels, for an example, a spheroidization of rod-like pearlitic cementite was enhanced and the spheroidized cementite particles were distributed uniformly in the ferrite matrix by SPD followed by static annealing treatment. The morphological change in the low carbon steels during SPD was discussed based on dislocation-cementite interaction. The mechanical properties of the severely deformed FCC and BCC metals were measured and discussed with slip systems operating in each class of metals. Finally, the grain refinement mechanism during severe plastic deformation was analyzed on the basis of observations regarding the formation of deformation bands associated with slip systems and their interactions.

2:45 PM
Nanostructures and their Evolution in Copper Processed by Repetitive Corrugation and Straightening (RCS): *Yuntian T. Zhu*[1]; Jianyu Huang[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87544 USA

A new Severe Plastic Deformation (SPD) process, Repetitive Corrugation and Straightening (RCS), has been developed to create bulk, nanostructured copper. In this presentation, the microstructures of copper processed by RCS will be discussed. HRTEM on RCS-processed Cu shows the existence of non-equilibrium grain/subgrain boundaries, a microstructural feature in SPD-produced nanomaterials that has been controversial. TEM observation revealed some unique microstructural features not observed in other deformation modes. The microstructural evolution during the RCS mode is compared with those during rolling and fatigue deformation modes.

3:00 PM Invited
Equal Channel Angular Processing of Magnesium Alloys: *Sean R. Agnew*[1]; G. M. Stoica[2]; L. Chen[2]; T. M. Lillo[3]; J. Macheret[3]; Peter K. Liaw[2]; [1]Oak Ridge National Laboratory, Oak Ridge, TN USA; [2]University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996 USA; [3]Idaho National Engineering and Environmental Laboratory, Idaho Falls, ID USA

Equal channel angular (ECA) processing offers the potential to introduce very large strains into a workpiece without changing its cross-section. Hence, it is an attractive technique for developing homogeneous fine-grained metals with good forming characteristics. Magnesium alloys are ideal candidates to benefit from the ECA process, because they exhibit poor low temperature forming characteristics due to a hexagonal close packed crystal structure. Magnesium has a low melting point, which enables its alloys to be processed isothermally at temperatures of 325°C and below. An assessment of the technique has been made with three commercial magnesium alloys ZK60, WE43 and AZ31 and one experimental alloy Mg-4wt%Li. The ductilities of the former two alloys, which contain Zr, were substantially improved. The latter two alloys, which do not contain Zr, showed little change in their properties for the processing conditions explored. Relative to the as-received material, the ductility of ZK60 was improved by 2 to 3 times over the entire temperature range investigated (24-450°C); with up to 350% elongations. Explanations for the property enhancements are discussed in terms of microstructure observations and deformation mechanisms.

3:20 PM
Syntheses of Nd2Ti2O7/Al2O3 Nanocomposites by Spark-Plasma-Sintering and High-Energy Ball-Milling: *Guodong Zhan*[1]; Joshua D. Kuntz[1]; Julin Wan[1]; Javier Garay[1]; Amiya K. Mukherjee[1]; [1]University of California, Dept. of Chem. Eng. & Matl. Sci., One Shields Ave., Davis, CA 95616 USA

Al2O3/3mol%Nd2Ti2O7 nanocomposites with different crystal structure of starting alumina nanopowders have been successfully consolidated to > 98% of theoretical density by SPS at relatively low temperatures. High-energy ball milling can decrease the agglomeration of nanoscaled powders and lead to the gamma-Al2O3 to alpha-Al2O3 phase transformation during milling and then enhance the densification at lower temperatures. The 3mol%Nd2Ti2O7/Al2O3 nanocomposite through high-energy ball-milling of gamma-Al2O3 nanopowder could be consolidated by SPS at a temperature as low as 1050°C and the resultant microstructure consists of ultrafine equiaxed grains with an average grain size of ~200 nm. However, the sintering temperature for 3mol%Nd2Ti2O7/Al2O3 nanocomposite without high-energy ball-milling of alpha-Al2O3 nanopowder was 50°C higher than that for composite by high-energy ball-milling and grain size was up to 280 nm. These results suggest that the combination of spark-plasma-sintering and high-energy ball milling could result in ultrafine matrix composites. This investigation was supported by a grant #G-DAAD 19-00-1-0185 from US Army Research Office.

3:35 PM
Evolution of Microstructure and Mechanical Behavior of Titanium during Warm Multiple Forging: *G. A. Salishchev*[1]; S. V. Zherebtsov[1]; R. M. Galeyev[1]; [1]Russian Academy of Sciences, Inst. for Metals Superplasticity Problems, Khalturina str. 39, Ufa 450001 Russia

Along with ECAP the multiple forging (MF) can be used in producing bulk ultrafine-grained materials. MF of titanium is carried out at tem-

peratures of warm deformation (0.34-0.37Tm) in a die set of special design. The formation of ultrafine-grained structure in titanium during MF at 400°C (0.35Tm) and strain rate of about 10-3 s-1 was studied. The flow stress curve was plotted up to cumulative strains around 6. The curve shows maximum in stress at strains around 2 followed by a minor strain softening. The transformation in structure occurring at low to moderate strains is connected with formation of low angle grain boundaries inside of initial grains. As strain rises their misorientation increases up to high angle that results in formation of uniform structure with the grain size of about 200 nm. The mechanisms of ultrafine-grained structure formation as well as the relationship between microstructures, textures and mechanical properties are discussed.

3:50 PM Break

4:00 PM Keynote
Influence of Processing Route on Microstructure and Grain Boundary Development during Equal-Channel Angular Pressing of Pure Aluminum: *Terry R. McNelley*[1]; Douglas L. Swisher[1]; Zenji Horita[2]; Terry G. Langdon[3]; [1]Naval Postgraduate School, Dept. of Mech. Eng., 700 Dyer Rd., Monterey, CA 93943-5146 USA; [2]Kyushu University, Dept. of Matls. Sci. & Eng., Fac. of Eng., Fukuoka 812-8581 Japan; [3]University of Southern California, Dept. of Matls. Sci., Los Angeles, CA 90089-1453 USA

High-purity (99.99%) aluminum was subjected to equal-channel angular pressing (ECAP) and then analyzed by orientation imaging microscopy (OIM) and backscatter electron (BSE) imaging methods. The analysis of microtexture and microstructure by OIM will be reviewed. The ECAP was conducted at room temperature with a die having a 90° angle between the die channels. Repetitive pressing operations were accomplished by either route A, BC or C. Billets were examined after one pass and after four or twelve passes by each of the ECAP routes. The microstructure after one pressing operation consisted of inhomogeneous, elongated grains that aligned with the shearing direction. The grain boundary disorientation distribution obtained by OIM, exhibited a peak at 2°-5°, indicating a deformation microstructure. After four pressings by each route, OIM and BSE data indicated the microstructures had become homogenized and exhibited a similar (sub)grain size (~1.3 mm). Elongation and alignment of the (sub)grains with the shearing direction of the last pressing operation was observed in all cases. The corresponding OIM disorientation distributions showed a significant decrease in the population of low (2°-5°) angle boundaries with a corresponding increase in the high (>15°) angle boundary fraction. The texture data from each route were similar in having only one or two dominant orientations, while the exact texture varied from route to route. Following twelve pressing operations, all routes produced a material with apparent further grain refinement (~1 mm) and an increase in high angle boundary population, while retaining a significant fraction (>10%) of low (2°-5°) angle boundaries. The preferred orientations in the material became more diffuse with repetitive pressings but all routes exhibited a <111> near the shear plane of the final pressing. After twelve ECAP passes, the microstructure comprised a mixture of deformation and recrystallization features regardless of the process route.

4:25 PM Invited
Mechanisms of Formation of Submicron Grain Structures during Severe Deformation Processing: *Philip Prangnell*[1]; Jacob Bowen[2]; Ali Gholinia[3]; [1]UMIST, Manchester Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK; [2] Risø National Labs, Matls. Dept., Roslkilde DK-4000 Denmark; [3]NIMR, Rotterdamseweg 137, PO Box 5008, Manchester 2600GA Netherlands

Severe deformation processing is an emerging method for the production of submicron grain structures in conventional alloys. By severely deforming metallic alloys to very high plastic strains materials can be produced with grain sizes as fine as 30 nm, containing mainly high angle grain boundaries (HAGBs). In this paper the structure of severely deformed alloys and the mechanisms by which grain refinement takes place are reviewed. Grain refinement occurs through grains subdividing by new HAGBs being formed discontinuously, on finer and finer length scales, until a limit is reached where the grain size merges with the subgrain size, which is the smallest available length scale within which dislocations can be emitted and adsorbed. The formation of new HAGBs is a natural consequence of crystal plasticity, that is initiated by grains splitting into deformation bands, and is promoted by heterogeneity and instability in plastic flow.

4:45 PM Invited
Surface Mechanical Attrition Induced Nanostructured Surface Layer on Metallic Materials: *Ke Lu*[1]; *Gang Liu*[1]; Jian Lu[2]; [1]Shenyang National Laboratory for Material Sciences, Inst. of Metal Rsrch., Chinese Acad. of Scis., Shenyang 110016 China; [2]LASMIS, Univ. of Tech. of Troyes, Troyes 10000 France

In the past decade, nanocrystalline (nc) materials have drawn much attention due to their novel properties and special microstructures. Meanwhile more and more experimental results indicated that nc materials possess some superior properties and performances compared with those of the conventional coarse-grained counterparts, synthesis of bulk nc materials for technological applications, nowadays, is still facing serious difficulties. In this work, we will present the synthesis of a nc surface layer on metallic materials (including pure metals and alloys) by means of the surface mechanical attrition (SMA) technique. Preliminary experimental results will be shown on microstructure characterization of the mechanically-induced nc surface layer and properties (mechanical, wear and friction, corrosion) for the treated materials. The micromechanism and processing techniques for realizing the surface nanocrystallization process will be discussed. Further development and prospects will be addressed with respect to the performance and technological applications of the engineering materials with a nc surface layer.

5:05 PM
The Effect of Strain Per Pass on the Microstructure Developed in Aluminum Processed by Equal Channel Angular Extrusion: *Pei-Ling Sun*[1]; P. W. Kao[1]; C. P. Chang[1]; [1]National Sun Yat-Sen University, Inst. of Matls. Sci. & Eng., Kaohsiung 804 Taiwan

The die angle used in equal channel angular extrusion (ECAE) determines both the shear plane orientation and the strain per pass. It is interesting to separate the effect of the shear plane orientation and the strain per pass on microstructural development. By rotating the billet 180° about the extrusion axis between passes (route C), the shear plane is kept unchanged during straining. Then the effect of strain per pass can be studied by using different die angles. In the present study, commercial pure aluminum was deformed by ECAE route C with two different die angles (90° and 120°). The deformed microstructure was characterized by TEM and the boundary misorientations were determined from TEM Kikuchi diffraction.

5:20 PM
Microstructure Evolution in Nanocrystal Formation during Ball Milling: *Zhiguang Liu*[1]; Jun Yin[2]; Yan Xu[1]; Koichi Tsuchiya[1]; Minoru Umemoto[1]; [1]Toyohashi University of Technology, Dept. of Production Sys. Eng., 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Aichi 441-8580 Japan; [2]Institute of Metal Research, 72 Wenhua Rd., Shenhe, Shenyang, Liaoning 110016 China

Nanocrystal formation by ball milling in pure Fe and eutectoid carbon steels has been studied. It was found that nanocrystalline ferrite (NF) first occurred as band structure at certain outer surface areas of powders at the early stage of ball milling due to deformation localization, while the interior remains the starting structure with much weaker deformation (coarse-grained region, CGR). The boundaries between NF and CGR were observed to be either quite clear or gradually changed. At least three types of microstructures were observed: coarse grains with either high density of dislocations or dislocation cells inside, layered nanocrystalline ferrite with a thickness ranging from 100 to less than 10 nm, and equiaxed nanocrystalline ferrite with a grain size of 5-10 nm. It has been suggested that a transition from dislocation cell wall created by work hardening during ball milling to grain boundary may be responsible to the observed nanocrystalline ferrite formation.

5:35 PM
Ultrafine Grain Formation during Equal Channel Extrusion in an Al-Mg-Sc Alloy: *Oleg Sitdikov*[1]; *Rustam Kaibyshev*[1]; Semeon Olenyov[1]; Gunter Gottstein[2]; [1]Institute for Metals Superplasticity Problems RAS, Khalturina 39, Ufa 450001 Russia; [2]Institute of Metal Science and Physics of Metals, RWTH, Kopernikusstrasse, 14, Aachen D-52056 Germany

Microstructure evolution in an Al-6%Mg-0.3%Sc alloy during intense plastic straining has been studied at T=250°C. Equal channel angular extrusion (ECAE) was carried out to total strains above 12. TEM observations showed that plastic deformation to e=1 results in the formation of areas of equiaxed subgrains which alternate with bands of dense dislocation walls. Following strain leads to transformation of dense dislocation walls into low-angle boundaries and an increase in misorientation of deformation induced boundaries. Low-angle boundaries gradually transform into high-angle boundaries with strain. Ultra-fine grains were revealed after e=2 within areas of equiaxed subgrains. Volume fraction of recrystallized grains tends to increase with strain. It can be concluded that continuous dynamic recrystallization (CDRX) occurs. The main feature of CDRX in the Al-Mg-Sc alloy is low rate of dislocation rearrangements and low mobility of deforma-

tion induced boundaries. The role of Al3Sc dispersoid particles in CDRX is discussed.

Second International Symposium on Ultrafine Grained Materials

Poster Session: Monday – Wednesday

Deformation Induced Ferrite Transformation and Grain Refinement in Steels: *Han Dong*[1]; [1]Central Iron and Steel Research Institute, Div. of Structl. Matls., Beijing 100081 China

Austenite to ferrite transformation in steels is one of the typical diffusion transformation which is encountered in a variety of structural steels. For commercial structural steels, the diving force for austenite to ferrite transformation, free energy, is dependent on chemical composition and temperature. For deformed austenite, the accumulative deformation energy could contribute to transformation driving force which results in the promotion to austenite to ferrite transformation if deformation energy could not be released through recrystallization. This kind of transformation is named as deformation induced ferrite transformation (DIFT). Ferrite grain through DIFT is much finer compared with conventional transformed ferrite grain. The accumulative energy in austenite mainly depends upon chemical composition, deforming temperature, strain, strain rate, sample dimensions, and loading manner, etc. Uniformity dispersion of ultra-fine ferrite grains induced by DIFT can be controlled through variables to change accumulative energy. By adopting the theory of DIFT, mean grain size of 4m in diameter can be obtained in plain low carbon steel, and corresponding mean grain size of 1m in diameter can be achieved in microalloyed steel. It is proved that DIFT is a very effective method for grain refinement in steels.

Strength of Submicrocrystalline Severely Deformed Commercial Aluminum Alloys: *Mikhail Vyacheslavovich Markushev*[1]; Maxim Yurievich Murashkin[1]; [1]Institute for Metals Superlasticity Problems of the Russian Academy of Sciences, Sector 27, Khalturin St. 39, Ufa, Bashkortostan 450001 Russia

Understanding the mechanical behavior of submicrocrystalline (SMC) materials processed by severe plastic deformation (SPD) is of great importance. The data on ambient temperature tensile properties of a range of commercial aluminum alloys will be reviewed. The influence of alloy composition, deformation technique (complex and equal-channel angular extrusion) and treatment mode will be discussed. The properties of SMC deformed and annealed conditions will be compared with standard values for conventionally strengthened products. It is shown that SPD could be effective to improve static strength of non-heat treatable and low-alloyed heat treatable alloys. For better realization of the effect the alloy should be processed by complex treatment, involving angular extrusion, conventional work hardening and heat treatment. The combination of strength and ductility achievable is comparable or even better to that found with many high-strength precipitation-hardened alloys. For most heat treatable alloys the use of SPD is ineffective.

On the Properties of Severe Plastic Deformations under Twist: *Yan Beygelzimer*[1]; Viktor Varyukhin[1]; Dmitriy Orlov[2]; [1]Donetsk Physical & Technical Institute NASU, R. Lyuksembourg 72, Donetsk 83114 Ukraine; [2]Donetsk State Technical University, Donetsk Ukraine

One of the most effective methods for obtaining bulk ultrafine grained materials is severe plastic deformation that is typically done using equal channel angular pressing. In this work we describe and analyze a new method for obtaining severe plastic deformations proposed by the first author. The method is based on the direct extrusion and was termed “twist extrusion”. The main idea of the method is to extrude a prism bulk through a matrix with a twist channel. The cross-section of the channel that is orthogonal to the pressing axis, is constant along this axis. In the general case, it is possible to have channels of quite arbitrary cross-section. Several variations of twist extrusion corresponding to known direct extrusion schemes are also possible; for example, twist extrusion in a container with a thin lubrication layer, hydrostatic or hydromechanical twist extrusion. We argue that in some cases twist extrusion has certain advantages. In particular, it allows to obtain not only bulks, but also ready-to-use products in the form of a prism with an arbitrary cross-section.

Friction Stir Processing to Achieve Fine Grains in Hexagonal Metals: *David J. Alexander*[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-6, MS G770, Los Alamos, NM 87545 USA

Friction stir processing is being applied to several hexagonal metals, to determine whether this method can be used to achieve fine-grained material. The intended application is for Be alloys to be used for fabrication of targets for the National Ignition Facility. Because of the difficulties in working with Be due to environmental and safety concerns, Mg and Ti have been used as surrogate materials for preliminary investigations. Information about initial results of friction stir processing of these hexagonal metals will be presented.

Ultrafine-Grained Tungsten Produced by SPD Techniques: *Igor V. Alexandrov*[1]; Georgy I. Raab[1]; Vil U. Kazyhanov[1]; Larissa O. Shestakova[1]; Ruslan Z. Valiev[1]; Robert J. Dowding[2]; [1]Institute of Physics of Advanced Materials, Ufa State Aviation Tech. Univ., 12 K. Marks Str., Ufa, Bashkortostan 450000 Russia; [2]US Army Research Laboratory, AMSRL-WM-MD, Aberdeen Proving Ground, MD 21005-5066 USA

The paper deals with the recent development of producing ultrafine grains in tungsten, using two SPD techniques, i.e. high pressure torsion (HPT) and equal channel angular pressing (ECAP). Tungsten is a typical low ductile and hard-to-deform metal and its SPD processing is a new and complex task. The application of HPT enables us to develop the ultrafine-grained (UFG) microstructure with a grain size of about 100 nm in the disk shape of a diameter about 10 mm. As a result, the microhardness has been increased almost twice. An investigation of the dependence of the accumulated strain, pressure and temperature on the features of the processed UFG microstructures and the final grain size has been performed. The regimes of equal channel angular pressing resulting in the formation of the UFG microstructures with the average grain size of about 1 mm in bulk rod shape ingots have been developed. It has been shown that further grain size refinement can be achieved by a variation of the strain rate and the temperature of ECAP as well as of the applied back pressure. The influence of the grain size on the strength and ductility of the commercial pure W samples has been established. A strong dependence of the brittle-to-dictile transition on the microstructure features has been revealed.

Effects of Hot Deformation on Austenite Transformation in HSLA Steel: *Yang H. Bae*[1]; Soon H. Hong[1]; Dong-Han Seo[2]; Jong-Kyo Choi[2]; Woong Y. Choo[2]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., 373-1 Kusung-dong, Yusung-gu, Taejeon 305-701 Korea; [2]POSCO, Tech. Rsrch. Labs., PO Box 36, Pohang 790-785 Korea

This study investigates the effects of hot working and isothermal heat treatment on austenite/ferrite phase transformation of Fe-0.2C-0.4Si-1.1Mn low alloy steel. The hot working process is known as the severe plastic deformation being employed for the purpose of refining the ferrite grain size of high strength low alloy(HSLA) steels. The alloy steel used in this study was manufactured by vacuum induction melting and followed by sizing process for the steel plate with a thickness of 15mm. The alloy steel specimens were austenized at the temperature of 950°C and then cooled with 40°C/s to several selected hot working temperatures ranged from 600°C to 800°C. It is shown from the experimental results that the volume fraction of the ferrite increased with increasing the isothermal heat treatment time and also with increasing the amount of hot working. It is also noted that the ferrite grain size decreased from 30μm to 5μm through the hot working process at the temperature of 775°C. By comparing the normalized TTT diagrams for both deformed and not-deformed specimens, it is concluded that the austenite/ferrite phase transformation kinetics mainly depend on the γ/γ grain boundary area.

Changing Titan Structure under Twist Extrusion: Yan Beygelzimer[1]; *Viktor Varyukhin*[1]; Dmitriy Orlov[2]; Boris Efros[1]; [1]Donetsk Physical & Technical Institute NASU, R. Lyuksembourg 72, Donetsk 83114 Ukraine; [2]Donetsk State Technical University, Donetsk Ukraine

We present some experimental results describing titan evolution during severe plastic deformation by twist extrusion (TE). In particular, we show that increasing the number of iterations of TE leads to the decrease of the grain size d in both orthogonal and parallel cross-sections of the bulk. For example, if in its original state titan had d~25mkm, then after three TE iterations, we had d<=1mkm. Furthermore, the bulk is deformed sufficiently uniformly across the entire cross-section. The micro-hardness H_mu of titan grows monotonically with the increase of the number of iterations. For example, if in undeformed state titan had H_mu~1720MPa on average, then already after the first iteration the average increased to 1990MPa. At the same time, the intensity of hardening gradually decreases and, after a large number of iterations, practically approaches zero. The structure and mechanical properties of titan after TE are compared against the analogous characteristics after equal-channel pressing.

Phase Transformations in Ultrafine Grained Fe and Fe-Mn Alloys: *Boris Efros*[1]; [1]Donetsk Physical & Technical Institute NASU, High Pres. Phys. & Adv. Tech., 72 R. Luxembourg St., Donetsk 83114 Ukraine

Influence pressure on structure-phase state of ultrafine-grained (UFG) iron and Fe-Mn alloys has been studied. Alloys with wide concentration of manganese (from » 0 to 55 wt. %), having in initial state BCC, HCP and FCC (stable and metastable state) phases were as the objects under investigation. The complex study of iron and Fe-Mn alloys structures, deformed by severe plastic deformation (SPD) under pressure, including the UFG structures, by methods of X-ray diffraction (XRD), transmission electron microscopy (TEM), Mössbauer spectroscopy (MOSS) and optical microscopy (OM) has been carried out. There is the range of pressure P » 0.1MPa - » 60 GPa and logarithmic deformation degree e = 0 - » 10 of studied alloys in experiments. In work is shown, that the influence the UFG structure has an effect in increase of forward-alpha-epsilon and reduction of reverse-alpha-epsilon on » 4 GPa in iron. Also is established, that the increase of pressure till 19-20 GPa in the UFG structure of Fe-Mn alloys in hcp of phase area lowers of reverse-gamma-epsilon so, that allows to stabilize 100% of a phase of high pressure under normal conditions and long endurance after removal of pressure. The research of temperature stability of a phase of high pressure has shown that the heating to temperature is higher 790 K initiates reverse transformation. For reception stable (under normal conditions) the FCC phase need to be sustained in an interval of temperatures 880-930 K during 5 minutes. Thus the obtained results of complex investigation (XRD, TEM, MOSS, OM) of studied metals and alloys in the UFG state have revealed the strong dependence of phase solid transformations on parameters of SPD and/or pressure and initial phase, structure state and concentration of Mn in alloys.

Determination of Dynamic Ferrite Transformation Kinetics during Deformation in Austenite: *Seung Chan Hong*[1]; Sung Hwan Lim[1]; Kyung Jong Lee[1]; Kyung Sub Lee[1]; [1]Hanyang University, Matls. Sci. & Eng., Haengdang-dong, Seoungdong-ku, 17, Seoul 133-791 Korea

The dynamic ferrite transformation during heavy deformation has been studied to obtain the ultrafine-grained structure in steel. In this study, the flow stress was measured at various temperatures with up to 1.2 of true strain and constant strain rate (10/s) by Gleeble 1500 in order to determine dynamic ferrite transformation kinetics quantitatively. The formation of dynamic ferrite decreased the flow stress during hot deformation in austenite. The flow stress was partly attributed by the flow stress of austenite and that of ferrite. Therefore, the fraction of dynamic ferrite was determined as follows; Xc = (óm-óa)/(óf-óa) Where, Xc is the calculated dynamic ferrite fraction, óm is the measured flow stress, óa is the extrapolated flow stress of austenite and óf is the extrapolated flow stress of ferrite. It was also confirmed that the critical strain of dynamic ferrite at high cooling rate to deformation temperature was shifted to lower strain compared to that at low cooling rate with the aid of increasing undercooling.

Nanostructure Formation and Carbides Dissolution in Rail Steel Deformed by High Pressure Torsion: Yu. V. Ivanisenko[1]; R. Z. Valiev[2]; W. Lojkowsky[3]; A. Grob[4]; H.-J. Fecht[4]; [1]IMSP RAS, Khalturin 39, Ufa 450001 Russia; [2]IPAM Ufa State Aviation Technical University, Ufa 450000 Russia; [3]High Pressure Research Centre PAS, Sokolowska 29, Warsaw 01-142 Poland; [4]Ulm University, Div. of Matls., Albert Einstein Allee 47, 89081 Ulm, Germany

The microstructure and the phase composition evolution of commercial UIC 860 steel during the high pressure torsion (HPT) were investigated by using transmission electron microscopy, thermomagnetic analysis and X-ray diffraction. Upon strain degree increasing, grain refinement up to 20 nm takes place and total cementite dissolution occurs. In the nanocrystalline state the steel is characterised by a maximum level of hardness (9.7 GPa), which is 2.5 times higher than that of the initial state. It was revealed, that the deformation is first concentrated in the soft phase-ferrite, where the cell structure forms. In the end a very high density of the defects, such as grain and cell boundaries, dislocations and vacancies achieves. The partial cementite dissolution starts already at the early stages of the HPT deformation and develops gradually after beginning of nanostructure formation in ferrite phase.

Formation of Nanocrystalline Structure in a Ni-20%Cr Alloy: *Rustam Kaibyshev*[1]; Nadia Dudova[1]; Vener Valitov[1]; [1]Institute for Metals Superplasticity Problems RAS, Khalturina 39, Ufa 450001 Russia

Microstructure evolution resulting in nanocrystalline structure was examined during plastic deformation of a Ni-20%Cr alloy at a temperature of 500°C (0.46Tm) both in compression and torsion. It was shown that a low energy dislocation structure evolves at initial stage of plastic deformation. Nanoscale grains are formed at a strain of about 1.2. Their size was 80 nm and 50 nm in samples subjected by compression and torsion, respectively. Initial boundaries and areas of intersections of deformation bands play a role of nucleation sites. Following deformation leads to increase in volume fraction of nanoscale grains, and fully grained structure was revealed after e=5.5. Nanocrystalline structure evolved is characterized by high value of microhardness (4.6GPa) and increased internal elastic strain. An effect of deformation scheme on formation of such a structure is discussed.

Formation of Ultrafine Grained Structure in an Al-Li Alloy during Intense Plastic Straining at Elevated Temperature: *Rustam Kaibyshev*[1]; *Fanil Musin*[1]; Ksenia Saytaeva[1]; Yoshinobu Motohashi[2]; [1]Institute for Metals Superplasticity Problems of RAS, Khalturina 39, Ufa 450001 Russia; [2]Ibaraki University, Rsrch. Ctr. of Superplasticity, Nakanarusawa-cho, 4-12-1, Hitachi, Ibaraki 316-8511 Japan

Microstructural evolution of 1421 aluminum alloy (Al-4.1%Mg-2.0%Li-0.16%Sc-0.07%Zr in wt. pct) has been studied at T=400°C and at a strain rate of about 10-2 s-1. The 1421 alloy was subjected to equal-channel angular extrusion (ECAE) up to true strain of 12. It was shown that continuous dynamic recrystallization occurs in the 1421 alloy and results in the formation of ultrafine grained structure. After e=1, the arrays of low-angle boundaries are evolved. Gradual transformation of low-angle boundaries into high-angle boundaries takes place with strain. First recrystallization grains with a size of about 1µm were revealed after e=2. Following deformation leads to increasing volume fraction of recrystallized grains which attends about 80 pct at e=12. In the same time, the deformation induced microstructure is non-uniform. At e=8, the bands of recrystallized grains alternate with bands of elongated subgrains. Mechanisms of ultrafine grain formation are discussed.

Structure and Properties of Commercial Aluminum Alloy 1560 (Al-Mg-Mn) after Severe Plastic Deformation and Annealing: Maxim Yurievich Murashkin[1]; *Mikhail Vyacheslavovich Markushev*[1]; [1]Institute for Metals Superplasticity Problems of the Russian Academy of Sciences, Sector 27, Khalturin St. 39, Ufa, Bashkortostan 450001 Russia

Specific features of microstructure and crystallographic texture of the 1560 (Al 6.0Mg 0.6Mn) alloy after severe plastic deformation (SPD) via equi-channel angular extrusion and annealing are considered. The phenomenology and nature of processes occurring at transformation of the submicrocrystalline deformation structure to the grain one are discussed. Mechanical behavior of the severely deformed and annealed alloy at tension and bending at room temperature is investigated. The influence of a structural state on a serrated flow, strength, hardness, ductility toughness and crack resistance (crack formation and crack growth resistances) is shown. The reasons for improved strength and low crack resistance of the alloy after SPD are analyzed. Based on the evaluation of microcrack formation intensity, the analysis of deformation relief and fracture, the origin of plastic flow and failure of the alloy having submicrocrystalline deformation, submicrocrystalline grain and microcrystalline structure is discussed.

Structure Evolution of Ultrafine Grain Copper and Nickel at Plastic Deformation: *Eduard Victorovich Kozlov*[1]; Anatolii Nikolaevich Zhdanov[1]; Lina Nikolaevna Ignatenko[1]; Nina Alexsandrovna Koneva[1]; [1]Tomsk State University of Architecture and Building, Phys. Dept., Solyanaya Sq. 2, Tomsk 634003 Russia

The structure of nanocrystalline materials determines mechanisms of plastic deformation. With the grain size of hundreds of nanometers, the main deformation mechanisms are intergranular dislocation glide and grain boundary sliding. Under conditions of self-coordination of intergranular glide and grain boundary sliding, dislocations are not retained inside grains, but get pulled in the boundaries. In the work the structure evolution of ultrafine grained copper, nickel and alloys on their base at active plastic deformation and creep is investigated. The behavior of grain structure, grain boundaries and distributions of grain sizes on deformation degree is studied. The changes of scalar dislocation density, amplitude of internal stresses are measured. The estimation of shear value on grain boundaries and the contribution of this shear in total deformation are carried out. The behavior of second phase particles is watched. It is paid attention to contributions in plastic deformation of grains of different sizes.

The Use of SPD for the Fabrication of Nanostructured Materials for Ball-Milled Powders: *Georgy I. Raab*[1]; Nickolai A. Krasilnikov[1]; Robert Klemm[2]; Ellen Thiele[2]; Vladimir S. Zhernakov[1];

[1]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Metals, 12 K. Marks Str., Ufa, Bashkortostan 450000 Russia; [2]Technical University Dresden, Inst. for Physl. Metal., Zellescher Weg 17, Dresden D-01097 Germany

Bulk nanostructured samples with high density were obtained from nanocrystalline Cu, Fe and Ni ball-milled powders using high pressure and severe plastic deformation (SPD). The process of the fabrication of the samples consists of a preliminary compacting and a following SPD, including such methods as high pressure torsion (HTP), equal channel angular pressing (ECAP) and multiforging under high pressure. The advantage of the suggested approach consists in the possibility of the fabrication of compacts with a density >98% and a nanocrystalline structure due to the decrease in the temperature of SPD consolidation under high pressure. Thus, the grain size of HTP Cu and Fe with a diameter of 20 mm and thickness of 2 mm was 100 mm. The Ni samples with a cross-section of 5x5 mm2 and a length of up to 50 mm having a grain size less than 100 nm were obtained by ECAP and multiforging. The samples obtained are characterized by a high strength, a homogeneous microstructure in the whole volume and high thermal stability. Meanwhile, some increase in die durability at low homologous temperature and elevated pressure remains the technical problem of the consolidation of nanostructured samples from powders. The technological aspects of the fabrication of high density samples from nanostructured powders by the applied methods, microstructural data and the mechanical behavior of the processed materials are discussed.

Formation of Nanocrystalline Structure in Two-Phase Titanium Alloys by Combining Thermohydrogen Processing and Warm Severe Plastic Deformation: *G. A. Salishchev*[1]; M. A. Murzinova[1]; S. V. Zherebtsov[1]; R. M. Galeyev[1]; O. R. Valiakhmetov[1]; [1]Russian Academy of Sciences, Inst. for Metals Superplasticity Problems, Khalturina str. 39, Ufa 450001 Russia

Warm severe plastic deformation realized via multiple forging can be used for formation of fine-grained microstructure with of a grain size of several hundred nanometers or less in titanium alloys. The less grain size the better superplastic properties resulted from lower temperature deformation. It has been established that the least grain size not only depends on the deformation temperature, but also the phase volume fraction, phase particle size and interparticle distance. The application of thermohydrogen treatment improves hot workability of titanium alloys at lower temperatures and permits to combine the deformation with metastable phases decomposition. Additional grain refinement is observed after hydrogen removal during vacuum annealing due to polymorphic transformation accompanied by recrystallization. The microstructure with a grain size of 25 nm was obtained in two-phase titanium alloy Ti-6.3Al-3.5Mo-1.7Zr wt.%. This alloy is superplastic at 550°C and e. =2¥10?4 s-1: m=0.52, relative elongation achieves 550%.

Nanostructured Metastable Alloys Processed by Severe Plastic Deformation: *Vladimir Vladimirovich Stolyarov*[1]; Ruslan Zufarovich Valiev[1]; [1]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Matls., K. Marx, 12, Ufa 450000 Russia

Severe plastic deformation (SPD) during processing two-or multiphase alloys has an ability in both strongly refine microstructure till up nanometer range and change essentially a phase composition forming highly metastable states. As a result, there is a potential to achieve new and extraordinary properties in these alloys. This paper demonstrates examples of attaining such properties in several Ti, Al and Fe-Pr(Nd)-based alloys using SPD processing by high pressure torsion (HPT) and equal channel angular pressing (ECAP). It is shown that for microstructures of the SPD materials it is typical to have a presence of not only small grain sizes, but also a formation of supersaturated solid solutions and amorphyzation of intermetallic phases alloys. During further heating aging effects and reordering take place and processed alloys demonstrate unique mechanical (strength and superplasticity) and magnetic properties.

Tensile and Fatigue Properties of Al-Mg-Sc-Zr Alloy Fine-Grained by Equal-Channel Angular Pressing: *Alexei Vinogradov*[1]; Vladimir I. Kopylov[2]; Atsuo Washikita[3]; Kazuo Kitagawa[3]; [1]Osaka City University, Dept. of Intelligent Matls. Eng., Osaka 558-8585 Japan; [2]Physico-Technical Institute of the National Academy of Science, Minsk 220730 Belarus; [3]Kanazawa University, Dept. of Mech. Sys. Eng., Kanazawa 920-8667 Japan

A significant material hardening is observed after equal-channel angular pressing (ECAP) so that an apparent yield stress approaches an ultimate tensile strength leaving for the material a very limited number of ways to accommodate external strains during loading after fabrication. Although it has been emphasized that the ductility of ECAP materials is rather high, special conditions such as those at superplasticity are often needed to demonstrate that high ductility. At room temperature most ECAP metals are less ductile than their coarse-grain counterparts. Although the higher yield stress may result in the improved fatigue limit, the low-cyclic fatigue performance degrades owing to reduced ductility. Using the Al-Mg-Sc-Zr alloy, it is shown that ECAP is capable of fabricating materials with enhanced both low- and high-cyclic fatigue properties. The ECAP Al-Mg-Sc-Zr demonstrates notable strain hardening during tensile testing and significant improvement of fatigue life in comparison with other commercial middle-strength Al-alloys.

Microstructure of a Low Carbon Steel after Equal-Channel Angular Pressing: Jingtao Wang[1]; Xicheng Zhao[1]; Yuan Wang[1]; Lizhong Wang[1]; Terence G. Langdon[2]; [1]Xi'an University of Architecture & Technology, Sch. of Metlgcl. Eng., Yanta Rd., Xi'an 710055 China; [2]University of Southern California, Dept. of Aeros. & Mechl. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Equal-Channel Angular pressing (ECAP) was successfully carried out on a low carbon steel at room temperature up to an equivalent true strain of ~11 via route C. Strongly banded structure was observed on the cross section after ECAP. The grain size was refined from 25 micrometers in the as-received hot rolled plate to a band width of ~0.2 micrometer after equal channel angular pressing to an equivalent true strain of ~11. The length of the grain fragment in the strongly banded structure also decreases with the increase of ECAP strain, and reaches a value of less then ~0.6 micrometer when the ECAP passage reaches or above. This paper reports investigations on microstructure characterization of the processed low carbon steel.

Synthesis, Microstructure and Characterization of Nanoscale Binary Aluminum Alloys: Jixiong Han[1]; Marty J. Pluth[1]; Jai A. Sekhar[1]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA

A study was made of the synthesis, microstructure and transformation behavior of nanoscale Al-Cu and Al-Zn alloy particles. Rapid plasma ablation followed by rapid quenching was used to synthesize nanoparticles from Al-4.4 wt.% Cu and Al-15 wt.% Zn alloy ingots and the microstructure and transformation behavior of the particles were studied by transmission electron microscopy. The particles were found to be in the supersaturated state in both types of alloys, but displayed a variation in the individual particle composition when compared with the precursor bulk alloys. After the plasma ablation and quenching process, the particles were exposed to air and were found to contain a 3-5 nm thick adherent aluminum oxide scale which prevented any further oxidation. Several of the particles were faceted and bound by (111) planes. The nanoparticles of the alloys were heat treated in order to examine the precipitation sequence in the Al-Cu alloys and spinodal decomposition in Al-Zn alloys. Differences in the transformation behavior of these alloy particles when compared with bulk alloys of the same composition were observed. In the Al-Cu alloys, precipitates which were considerably enriched in copper were noted at the beginning of the transformation. These subsequently transformed to precipitates with lower copper content, as the heat treatment progressed. In the Al-Zn alloy, a spinodal structure was noted in the nanoparticles, as well as precipitates of pure zinc with a f.c.c. structure upon aging. In both the Al-Cu and Al-Zn alloy particles, the precipitates were observed to form along the aluminum oxide-particle interface. Details of the precipitation sequence, nature and structure of second phase precipitates and interphase interfaces and formation mechanisms will be reported. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

Shear Banding in Materials: Metals

Sponsored by: ASM International: Materials Science Critical Technology Sector

Program Organizers: Walter W. Milligan, Michigan Technological University, Metallurgical Engineering M, Houghton, MI 49931-1295 USA; Todd C. Hufnagel, Johns Hopkins University, Department of Materials Science and Engineering, Baltimore, MD 21218-2689 USA; Marc Andre Meyers, IMM, University of California, San Diego, CA 92093 USA; Hans Jorgen Roven, Norwegian University of Science and Technology, NTNU, Department of Metallurgy, Trondheim N-7034 Norway

Monday PM Room: 303
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Todd Hufnagel, Johns Hopkins University, Dept. of Matls. Sci. & Eng., Baltimore, MD 21218 USA; Marc Meyers, University of California-San Diego, Appl. Mech. & Eng. Scis., La Jolla, CA 92093 USA

2:00 PM
Shear Banding in Different Classes of Materials: *Walter W. Milligan*[1]; [1]Michigan Technological University, Matls. Sci. & Eng., Houghton, MI 49931 USA

An overview of the phenomenon of shear banding in various types of materials, as well as the goals of the workshop, will be presented.

2:20 PM
Shear Banding as the Dominant Deformation Mechanism in Nanophase and Ultrafine-Grained Iron: *E. Ma*[1]; D. Jia[1]; Q. Wei[1]; K. T. Ramesh[1]; [1]Johns Hopkins University, Dept. of Matls. Sci. & Eng., Baltimore, MD 21218 USA

Iron samples with a range of grain sizes have been processed by mechanical attrition followed by consolidation. The consolidation was a two-step process. In the first step, the powder was compacted under a pressure of 1.4 GPa at room temperature for about 10 hours using a WC die. In the second step, the compact was transferred into a larger WC die for hot consolidation. Temperatures in the range of 480-680°C and a pressure of 850 MPa were applied to obtain different grain sizes, ranging from sub-micron to ca. 100 nanometer. Samples for mechanical testing were cut from the fully dense compacts using electrical discharge machining (EDM). Compression tests under a quasi-static strain rate were performed with an MTS machine. High strain rate testing was conducted using a miniature Kolsky bar uint (or Desktop Kolsky Bar (DKB)). Strain rates up to ~5 x 10+4 s-1 were obtained with DKB. Shear banding was established as the predominant plastic deformation mechanism under both quasistatic and dynamic conditions when the grain size is below 300 nm. The development of multiple shear bands has been monitored as a function of plastic strain. Transmission electron microscopy (TEM) was used to study the microstructure inside and outside the shear bands. Nano-indentation measurements were carried out to investigate the nano-scale mechanical behavior inside and outside the shear bands.

2:40 PM
Shear Localization and Failure in Pre-Shocked Metals: *Geoffrey H. Campbell*[1]; Mukul Kumar[1]; James S. Stölken[1]; F. Xabier Garaizar[2]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., PO Box 808, MS L-356, Livermore, CA 94550 USA; [2]Lawrence Livermore National Laboratory, Computations Direct., PO Box 808 L-561, Livermore, CA 94550 USA

Strain localization is an important phenomena for understanding metal dynamics in general and many types of failure observed in practice. There exists much information on both localization and failure of ductile metals that start with a low defect density; however, less is known about highly defective metals. Shocks induce deformation microstructures with high dislocation densities (>10^12 /cm2) that are comprised, for the most part, of statistically stored dislocations. This highly defective state allows recovery mechanisms to begin at an earlier point in the deformation behavior, reducing the work hardening rate and increasing the propensity for shear localization. We have induced shocks using laser drives in pure Ta, Ta-2.5% W alloy, and pure Cu under a variety of conditions. We have measured their mechanical properties and characterized their microstructures in the pristine, shock processed and mechanically strained conditions with a variety of techniques, including EBSD mapping and TEM. We have also performed plane stress fracture mechanics tests with the double edge notched tension specimen. The mechanical response will be discussed in terms of the observed shear localization behaviors.

3:00 PM
The Shear Band in 7050 Aluminum Alloy: *Shih-Chin Chang*[1]; Yaw-Shing Wang[1]; Yuan-Yuan Lin[1]; [1]National Tsing-Hua University, Dept. of Matls. Sci. & Eng., Hsinchu 30043 Taiwan

In plane strain compressed 7050 aluminum alloy specimens with either T or L direction constrained, macroscopic shear bands were visible on the constrained plane surface of the specimen. The shear bands formed along planes of maximum shear stress which make an angle 45 degree to the S and the non-constrained plane. The maximum shear strain observed in a shear band is 2. It was noticed that the strength of specimens constrained in L direction were higher than that of their counterpart specimens constrained in T direction. For under aged specimen, the shear bands was sharply localized. Specimen will shear rupture along shear bands with a sudden drop of the stress. Similar but wider shear bands were observed in peak and over aged specimens. The width of over aged specimens was the largest. No rupture were observed in over aged specimens even when the true compression strain went up to 0.6 when the test ended. For under aged specimen with T direction constrained, the true strain of rupture is 0.32. For cold rolled specimen, the formation of shear bands occurred at a smaller strain and the true strain of rupture is 0.27. When constrained in L direction, the rupture strain, 0.29 for underaged and 0.125 for underaged and cold rolled specimens was smaller than those in T direction. In contrast to the underaged case, for over aged specimens the stain of shear band formation was larger when constrained in L direction than in T direction. A model based on the dislocation-precipitate interaction was proposed to explain these observations.

3:20 PM Break

3:40 PM
The Effect of Shear Localization on Ductility and Fracture: *Fereshteh Ebrahimi*[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, Gainesville, FL 32611 USA

There is a broad range of behaviors that lead to plastic strain localization. Usually, local softening due to microstructural weaknesses (e.g., particle or void shearing, short range disordering), hotness (e.g. high strain rate, cryogenic deformation), inhomogenity of microstructure/composition (e.g. grain size or hydrogen distribution) or crystal rotation (e.g., non-Schmid effects in single crystals) creates the conditions for shear banding. In this presentation examples of shear localization in single crystals (NiAl and a superalloy) and nanocrystals (nickel and copper) are presented. While a low resistance to ductile fracture can be explained by tendencies to shear localization, it is not clear why softening should encourage cleavage fracture. It is shown that indeed cleavage facets develop within the shear bands but on planes that experience the highest tensile stress components. An explanation based on strain incompatibility that leads to the development of high hydrostatic tensile stress components is suggested.

4:00 PM
Shear Band Phenomena in Extreme Deformation Processes: From Ballistic Penetration to Friction-Stir Welding: *Lawrence E. Murr*[1]; Sridhar Pappu[1]; Christine Kennedy[1]; Mario Guerra[1]; [1]University of Texas at El Paso, Metall. & Matls. Eng., 500 W. University Ave., El Paso, TX 79968-0520 USA

The interplay between strain and strain rate as well as strain saturation in the context of strain hardening can produce intermittent regions of shear instability or overlapping, broad regimes of adiabatic shear bands which facilitate superplastic, solid-state flow as a consequence of intrinsic, dynamic recrystallization (DRX). During the penetration of ductile targets such as copper by tungsten-heavy alloy (WHA) rods, the target material flows during cratering to form the entrance rim and jetting. Penetration occurs by overlapping shear bands composed of mostly equiaxed DRX grains. Similar features are observed for the penetration of tungsten single crystal rods into RHA steel targets. For 7039 aluminum targets strain-hardened to the maximum, intermittent shear bands combined with a very thin DRX regime at the channel wall facilities WHA rod penetration. Correspondingly, the friction-stir welding (FSW) of the same or dissimilar metals and alloys such as brass to copper or 6061 Al to itself produces intercalations or mixtures of narrow and very wide shear bands which flow and produce residual vortex and other complex flow patterns within the weld zone. Solid material between the shear bands can flow as large blocks. Detailed light and transmission electron microscopy studies confirm that in all of these flow phenomena involving various shear bands DRX provides the fundamental solid state flow mechanism.

Research supported by ARO-DAAG55-97-1-0238 and GSA Grant PF-90-018.

4:20 PM
The Effect of Shear Banding on Ductility and Bendability of Automotive Aluminum Alloys: *David S. Wilkinson*[1]; J. David Embury[1]; David J. Lloyd[2]; [1]McMaster University, Matls. Sci. & Eng., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada; [2]Alcan International, Kingston R&D Ctr., Kingston, ON K7L 5L9 Canada

Aluminum alloys such as AA5754 and AA6111 have been developed as sheet materials for automotive applications. One of the technical issues controlling the utility of these materials is their bendability, for example in hemming operations. Through extensive work on these alloys we have found that the interaction between shear bands and Fe-aluminide constituent particles plays a critical role in the failure process that controls both tensile ductility (as measured by the reduction in area) and bendability. As the level of Fe (and thus the constituent particle density) increases the failure mode can sometimes change quite dramatically. In this presentation we will present results in which the effect of Fe content has been studied at a range of temperatures in both tension and bending. We will also present data for tensile tests performed under superimposed pressure.

4:40 PM
TEM Study on Microstructural Evolution of Rolled Al-1%Mn Single Crystals of {001}<110> Orientation: Zhengjie Li[1]; [1]Tsinghua University, Dept. of Matls. Sci. & Eng., Bldg. 14# 208, Beijing 100084 China

Aluminum single crystals of {001}<110> orientation have been rolled to 18, 30, 50, 70, 80 and 90% reductions under controlled homogeneous rolling conditions. The evolution of the deformation structure during rolling was investigated using the Transmission Electron Microscopy (TEM) and the local orientations were measured by a simple semi-automatic TEM method. The regions investigated are characterized by two types of bands with sharply alternate orientations and strong TD crystal rotation was observed. Long and Straight dislocation boundaries parallel to the slip planes of {111} (i.e. crystallographic boundaries) are observed at low to medium strains whereas shortened and more or less equiaxed deformation structure are developed at higher strains. A theoretical analysis based on the idea of crystal slip geometry and Frank formula was proposed to explain the microscopic subdivision and local crystallography of crystals with the present orientation.

Surface Engineering: Science & Technology II: Surface Modification Technologies

Sponsored by: Materials Processing & Manufacturing Division, Surface Engineering Committee
Program Organizers: Ashok Kumar, University of South Florida, Department of Mechanical Engineering, Tampa, FL 33620 USA; Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Gary L. Doll, The Timken Company, Canton, OH 44706 USA; D. S. Misra, Indian Institute of Technology-Bombay, Department of Physics, Powai, Mumbai 400076 India; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Kryoshi Yatsui, Nagoka University of Technology, Nagaoka, Niigata 840-2188 Japan

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Session Chairs: Ashok Kumar, University of South Florida, Dept. of Mechl. Eng. & Ctr. for Microelect. Rsrch., Tampa, FL 33620 USA; Yang-tse Cheng, General Motors, Matls. & Proc. Labs., Warren, MI 48090 USA

2:00 PM Opening Remarks

2:05 PM Invited
Laser-Based Micro- and Nanoprocessing: *Y. F. Lu*[1]; [1]National University of Singapore, Elec. & Comp. Eng., 10 Kent Ridge Crescent 119260 Singapore

Laser-induced micro and nanoprocessing haven investigated for potential applications in microelectronics, data storage and photonics. The following toptics will be addressed in detail: laser surface cleaning, laser deflash and plasma-assisted laser microprocessing, laser texturing, laser bumping and related technologies, laser deposition of hard coatings and thin films, real-time Monitoring of laser surface processing, laser-induced controllable periodic structures, laser etching and nanolithography by tip-enhanced laser irradiation and optical resonance in microparticle.

2:30 PM
Surface Modification of Austenitic Stainless Steels: *Howard Ocken*[1]; Roger Asay[2]; [1]EPRI, 3412 Hillview Ave., Palo Alto, CA 94304-1395 USA; [2]Centec XXI, 8870 Muraoka Dr., Gilroy, CA 95020 USA

Austenitic stainless steels are widely used as structural alloys in nuclear power plants. The passive films that form when in contact with high-temperature primary coolant incorporate activated corrosion products, which are responsible for the exposure of plant personnel to ionizing radiation. The industry's need to minimize such exposures has led to the development of various surface modification techniques. EPRI has supported development of a surface modification technique, the Stabilized Chromium Process (SCrP), that is more effective than electropolishing. This paper will address the following topics: [1] early experiments on small coupons, [2] developing approaches for applying SCrP to complex shapes, [3] evaluating the response of components installed in commercial nuclear reactors, and [4] evaluating SCrP for possible non-nuclear applications.

2:45 PM
Deposition of Metal Coatings Utilizing Ablation Plasma Ion Implantation (APII): *Ronald M. Gilgenbach*[1]; Bo Qi[1]; Yue Ying Lau[1]; Mark D. Johnston[1]; Michael C. Jones[1]; Gary L. Doll[2]; Alexander G. Lazarides[2]; [1]University of Michigan, Nucl. Eng. & Rad. Scis. Dept., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA; [2]The Timken Company, Timken Rsrch., Adv. Matls. R&D, 1835 Deuber Ave. S.W., Canton, OH 44706 USA

We present results on a new technique for deposition and implantation of metal coatings by means of ions generated by KrF laser ablation of metals and accelerated by pulsed bias to the substrate. Initial APII experiments have implanted iron ions into silicon substrates at bias voltages up to negative 10 kV. Materials have been analyzed by Transmission Electron Microscopy (TEM) and X-ray Photoelectron Spectroscopy (XPS). Results prove that ion implantation has occurred, consistent with a maximum effective ion energy of about 8 keV. The lower effective energy is due to voltage droop and limited penetration of the overlying Fe film. XPS performed during an argon ion etch shows a depth profile confirming Fe implantation and deposition on the Si substrate. Future research will concentrate on ion implantation and deposition of hard coatings over softer metals. This research was funded by the National Science Foundation.

3:00 PM
Surface Modification of P/M TiC Reinforced Iron Matrix Composites: *Omer N. Dogan*[1]; David E. Alman[1]; Jeffrey A. Hawk[1]; [1]US Department of Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA

Surface modification of wear resistant Fe-M_xC_y-TiC composites using a surface melting technique was explored. Powder metallurgy (P/M) was utilized to fabricate the composite alloys. Iron powder was mixed with elemental titanium, chromium, and graphite powders, and then hot-pressed to full density. During the hot pressing cycle, an SHS-type reaction occurred, transforming the titanium and chromium powders to TiC and $(Fe,Cr)_xC_y$ in a steel (Fe-Cr-C) matrix. The influence of alloy composition and SHS process parameters on the microstructure and abrasive wear resistance of the TiC reinforced composites were discussed. It is demonstrated that during the surface modification, the TiC phase does not melt but the steel matrix and $(Fe,Cr)_xC_y$ phase melt. During solidification of the austenite and M_7C_3 eutectic, a finer distribution of M_7C_3 particles is obtained. The in-situ formation of TiC and $(Fe, Cr)_xC_y$ precipitates via SHS reaction processing and surface melting produce a composite with wear resistance superior to conventional wear resistant materials.

3:15 PM
Synthesis of Nickel Ferrite Nanosize Powder by Pulsed Wire Discharge: K. Ishizaka[1]; Y. Kinemuchi[1]; T. Suzuki[1]; H. Suematsu[1]; W. Jiang[1]; *K. Yatsui*[1]; [1]Nagaoka University of Technology, Extreme Energy-Density Rsrch. Inst., 1603-1 Kamitomiokamachi, Nagaoka, Niigata 940-2188 Japan

Various metal, simple oxide and simple nitride nanosize powders have been synthesized by a novel pulsed wire discharge (PWD) method. However, synthesis of double oxides has not been attempted because of difficulties in discharging two different metal wires simultaneously. In order to investigate possibilities of synthesizing double oxides by PWD, synthesis of nickel ferrite ($NiFe_2O_4$) nanosize powder was carried out. Nickel and iron wires were discharged in a chamber filled with oxygen

gas. The synthesized powders floating in the gas were collected by pumping the gas through a molecular sieve. X-ray diffraction results indicated that small amount of a nickel oxide phase was remained in the powders. However, the main phase was $NiFe_2O_4$ and essentially single phase $NiFe_2O_4$ nanosize powders were successfully synthesized. From the results, we demonstrated capabilities of synthesizing complex oxide nanosize powders by PWD.

3:30 PM Invited
Laser Surface Modification of Electronic Properties in Wide Band Gap Materials: *I. A. Salama*[1]; N. R. Quick[2]; A. Kar[1]; [1]University of Central Florida, LAMMP, CREOL, Mech., Matls. & Aeros. Eng. Dept., Orlando, FL 32816 USA; [2]Applicote Associates, 894 Silverado Ct., Lake Mary, FL 32746 USA

A direct-write laser conversion technique is used to induce variation in both electrical and microstructural properties on the surface of wide band gap materials such as silicon carbide (SiC), diamond and gallium nitride (GaN). The effects of various processing parameters, e.g., laser-matter interaction time, laser beam power, number of repeated irradiation and ambient gas in the irradiation chamber are examined. The microstructural variations induced by laser beam irradiation are assessed for different process parameters. Scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS), X-ray diffraction (XRD), x-ray photoelectron spectroscopy (XPS) are used to study the laser-irradiated surfaces and to understand the improvement in electrical properties observed in different materials. Different applications of the laser conversion technology will be discussed.

3:55 PM Break

4:10 PM Invited
Modifying the Surface of Nanoparticles by Coating: *Dieter Vollath*[1]; Dorotheé Vinga Szabó[1]; Sabine Schlabach[1]; Bin Xu[1]; [1]Forschungszentrum Karlsruhe, IMF III, PO Box 3640, Karlsruhe D-76021 Germany

Using the microwave plasma process, it is possible to synthesize ceramic nanoparticles with narrow size distribution. In many instances, the application of this kind of materials requires a modification of the surface. The reasons may be the reduction of the particle interaction using a coating as distance holder or an alteration of the chemical properties of the surface. It is a special feature of the microwave plasma process that the particles leave the reaction zone with electric charges of equal sign. Therefore, the particles are repelling each other. This makes it possible to coat the particles individually in a second reaction step. Depending on the application, this coating may consist of a polymer or a second ceramic. This coating–in the simplest case–influences the dispersion behavior in water. Other typical examples of application are superparamagnetic composites, where the coating acts as a distance holder in-between the particles reducing interaction.

4:35 PM
The Spectral Emittance and Long-Term Thermal Stability of Coatings for Thermophotovoltaic (TPV) Radiator Applications: *Brian Vern Cockeram*[1]; Jim L. Hollenbeck[1]; [1]Bechtel-Bettis, ZAP 08D/MT, PO Box 79, W. Mifflin, PA 15122 USA

Vacuum plasma spray coatings (ZrO2 + 18%TiO2 + 10%Y2O3, ZrC, Fe2TiO5, ZrTiO4, ZrO2 + 8%Y2O3 + 2%HfO2, and Al2O3 + TiO2) have been developed to improve the surface emissivity of materials under consideration for TPV radiator applications. These coatings have been shown to be thermally stable and have produced a desired increase in the surface emissivity of refractory metals and nickel-base materials. The spectral emissivity of these coatings is measured before and after long-term vacuum anneals to determine the power density that would be provided to a TPV cell. The thermal stability of the coatings is further evaluated by characterization of the coatings after long-term vacuum annealing. A kinetic model of the volatility of oxide phases is used to describe the excellent thermal stability of the coatings that possess high post-anneal emittance values (ZrO2 + 18%TiO2 + 10%Y2O3, ZrC, and Al2O3 + TiO2.

4:50 PM
Surface Treatment of Aluminum Alloys with Potasium Ferrate Solutions: *Myung K. Han*[1]; S. K. Kim[1]; [1]Korea Institute of Industrial Technology, Ctr. for R&D Assistance for SMEs, 472 Kajwa-dong, Seo-ku, Inchon 404-254 Korea

A new and environment-friendly surface treatment method for aluminum alloys has been studied, which appears to be applicable as an alternative to the chromate conversion coating. The major drawback for the chromate conversion coating process is the toxicity of the hexavalent chromium, which is classified as a "known" carcinogen. Surface treatment with potassium ferrate solutions appears to be an ideal alternative to this process in that (1)the quality of the coated surface is superior, (2)ferrate compounds can easily be reduced to ferrous compound the thus become environmentally benign substance, and (3)the process consists of a simple dip-and-dry procedure. In this paper we present (1)pilot scale production of potassium ferrate powder, (2)analysis of the purity of the powder, (3)salt fog tests of the aluminum alloys treated with potassium ferrate solutions, and (4)analysis of the ferrate-coated surface.

5:05 PM
Nanostructuring Diamond Polytypes using Direct Ion Beam Deposition: *Quan Li*[1]; X. F. Duan[2]; N. G. Shang[1]; I. Bello[1]; Y. Lifshitz[1]; S. T. Lee[1]; [1]City University of Hong Kong, Dept. of Appl. Phys., Tat Chee Ave., Kowloon Tong, Hong Kong China; [2]Beijing Laboratory of Electron Microscopy, Beijing China

Direct ion beam deposition using an ion source consisting of mixtures of hydrocarbon/argon/hydrogen ions is capable of producing different diamond polytypes during diamond-like-carbon (DLC) film growth. A large range of experimental conditions is investigated including ion energy, substrate temperature and gas concentration ratios. Transmission electron microscopy (TEM) is used as the major analytical tool to study the film/substrate interface and film microstructures. Bombardment of argon ions together with hydrogen ions can modify silicon substrate surface by creating hillock patterns, exposing different silicon surfaces and increasing the substrate surface roughness. These modified silicon surfaces are proved to serve as epitaxial nucleation sites for diamond crystallites. Polytypes of diamond, including cubic, rhombohedral and hexagonal structures are observed in two forms concerning the original nucleation sites, i.e., directly grown on silicon or embedded in the amorphous DLC films. While the former exhibits certain epitaxial relationship with the silicon substrate, the later does not show any preferential orientation.

Third International Sulfide Smelting Symposium - "Sulfide Smelting '02": Smelter Gas Handling

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee, Copper, Nickel, Cobalt Committee, Lead and Zinc Committee, Non-Ferrous Metals Committee
Program Organizers: Robert L. Stephens, TeckCominco Metals, Ltd., Trail, British Columbia V1R 4L8 Canada; Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112 USA

Monday PM	Room: 607
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Norbert L. Piret, Piret & Stolberg Partners, Im Licht 12, Duisburg 47279 Germany; Pekka Taskinen, Outokumpu Research Oy, PO Box 60, Pori FIN-28101 Finland

2:00 PM
Effective Design of Converter Hoods: *Paykan Safe*[1]; John Deakin[2]; Sam Matson[1]; [1]Gas Cleaning Technologies, 4950 N. O'Connor Rd., Ste. 250, Irving, TX 75062 USA; [2]H. G. Engineering, Ltd., 400 Carlingview Dr., Toronto, ON M9W 5X9 Canada

The primary source of fugitive gas emissions in most copper smelters are the Peirce Smith converter operations. The modern trend is towards installation of secondary gas collection systems which are intended to collect fugitive gases from charging, standy, and skimming operations. These have been installed with varying degrees of success. Often, the major source of emissions from the converter area is leakage of process gas from the primary hoods when the converter is blowing. This gas is typically collected by the secondary system and discharged to atmosphere or scrubbed with an alkaline agent. Those smelters considering installation of secondary hoods or scrubbing of gases from secondary systems should first address the issue of gas leakage from the primary hoods. Improving the primary hood gas collection efficiency will often be more cost effective than installing secondary hooding. In the case of secondary gas scrubbing, it will significantly reduce the reagent consumption and operating cost of the scrubbing system. This paper examines the parameters that determine whether or not a primary hood will effectively collect process gases without leakage and demonstrates, using Computational Fluid Dynamics (CFD), the effects of variations in these parameters on the overall performance of the hood. It also looks at the application of CFD techniques in the design of secondary hoods and presents a summary of measured emissions from converter buildings.

2:25 PM
Boliden's Specialized Technology for Smelter Gas Handling and Effluent Treatment: *Bjorn Lindquist*[1]; [1]Boliden Contech AB, PO Box 745, Gymnasievagen 14, SE-931 27 Skelleftea Sweden

In this paper, Boliden's technologies in the field of gas cleaning and effluent treatment will be described. Special attention will be given to the use of high pressure venturis, the Editube wet electrostatic precipitator, mercury removal processes, and two-stage effluent treatment.

2:50 PM
Optimization of Metallurgical Processes using High Rate Biotechnology: Henk Dijkman[1]; *Johannes Boonstra*[1]; Rick Lawrence[2]; Cees J.N. Buisman[1]; [1]Paques B.V., PO Box 52, 8560 AB, Balk The Netherlands; [2]BioteQ Environmental Technologies, Inc., Ste. 1150, 355 Burrard St., Vancouver, B.C. Canada

Existing metallurgical operations can be optimized by implementing engineered bioreactor systems in the process configuration. For instance, metals can safely and economically be recovered from process and waste streams using biogenic sulfide. The paper focuses upon possible applications of high rate biotechnology in metallurgy. Metal recovery from liquid streams (electrolyte bleed streams, leach water, waste streams), integrated removal of (fugitive) SO2, treatment of acid blowdown and other options will be described. Practical examples will be given. A large scale application of biotechnology at Pasminco's Budel Zink refinery will be described. Here acid plant blowdown is treated together with electrolyte magnesium bleed. Concentrated zinc sulfide (10 tpd) is produced biologically, eliminating the production of gypsum and closing the refinery sulfur cycle. A smaller scale application of biotechnology at Kovohute Primbram in the Czech Republic is described. In this case H2S is produced on-site for metal removal from a waste water stream.

3:15 PM
A Novel Cyclic Process for Producing Elemental Sulfur from Sulfur Dioxide without Generating Secondary Pollutants: *Hong Yong Sohn*[1]; Byung-Su Kim[1]; [1]University of Utah, Dept. of Metallurgical Eng., Rm. 412, 135 S. 1460 E., Salt Lake City, UT 84112 USA

A thermodynamic and experiemental investigation has been performed to develop a new process for converting sulfur dioxide to elemental sulfur by a cyclic process. The most promising new process is a cyclic reaction scheme involving calcium sulfide and calcium sulfate. In this process, the reaction between sulfur dioxide and calcium sulfide produces elemental sulfur vapour and solid calcium sulfate. The latter is reduced by a suitable reductant, such as hydrogen, to regenerate calcium sulfide. Experimental results show that up to 60% of the calcium sulfide powder was converted to calcium sulfate in 10 minutes at 1153 K under a sulfur dioxide partial pressure of 28.5 kPa. More than 95% of the nickel-catalyzed calcium sulfate powder was converted to calcium sulfide in 20 minutes at 1123 K under a hydrogen partial pressure of 86.1 kPa, water vapor being the only gaseous product. Sulfur dioxide-containing streams from certain new sulfide smelting plants contain much higher partial pressures of sulfur dioxide. The rate of the first reaction is expected to be accordingly higher than in the test conditions reported in this paper.

3:40 PM Break

3:55 PM
Recent Improvements in the Acid Plant at Naoshima Smelter: *Shin-ya Yagishita*[1]; *Fumitaka Sakurai*[1]; [1]Mitsubishi Materials Corporation, Naoshima Smelter & Refinery, 4049-1 Naoshima-cho, Kagawa-Gun, Kagawa 761-3110 Japan

The Mitsubishi process in Naoshima has kept steady operation since May 1991. Yet the sulfuric acid plants have been using old equipment for more than 30 years operation. In 1999, the acid plants were modernized by renewing some of main equipment like, drying tower, gas heat exchanger and introducing new technology. At the same time, as an electric energy saving measure, the electric power consumption in sulfuric acid plant was vastly improved by decreasing treated gas volume based on installation of low temperature catalyst in 1st pass of converter, and optimization of main gas blower runner. Furthermore, last year, the oxygen plant was replaced and enlarged, and anode production increased from 240 to 280 KMT/Y. And the sulfuric acid production also increased from 550 to 660 KMT/Y. This paper describes these recent improvement in the acid plant at Naoshima smelter.

4:20 PM
Recovery of Sulphur in Off-Gases from Roasting of Molybdenum Sulphide: *Jens Laursen*[1]; [1]Haldor Topsoe A/S, Nymollevej 55, 2800 Lyngby Denmark

In 1999, Molymex S.A. de C.V., Mexico (a company wholly owned by Molybdenos y Metales S.A. [Molymet], Chile) decided to install the Haldor Topsoe WSA process for cleaning of the 4-5% S02 off-gas from their molybdenum sulphide roaster in Cumpas, Mexico, (WSA-the Wet gas Sulphuric Acid process for converting S02 in off-gases to sulphuric acid). The paper will describe the process layout and the initial experiences from the start up scheduled for end 2001. The installation of the WSA plant was decided, on the basis of successful operation since 1993 of a WSA plant at Molymet's molybdenum sulphide roaster in Santiago, Chile. The paper will describe the operating experience of the Santiago plant and furthermore summarise the application of the WSA process for other roasting plants and smelters within the non-ferrous metals industry (Copper, Lead, and Molybdenum).

4:45 PM
Modern Off-Gas Treatment Secures the Future of Sulfide Smelting: *Karl-Heinz Daum*[1]; *Hans-Jochen Koenig*[1]; Peter Luedtke[1]; [1]Lurgi Metallurgie GmbH, Ludwig-Erhard-Str. 21, D-61440 Oberursel Germany

In order to meet environmental regulations, sulfur capture in smelters has to be further optimized. At the same time, efficiency, reliability, ad economics of smelter operation have to be improved. Thus, off-gas handling systems should give saleable byproducts, not interfere with smelter performance, treat all gases simultaneously, and comply with environmental regulations. The latest developments in sulfur acid plant design will be presented.

5:10 PM Moved to Monday AM
Budel Zink Sets a New Standard for NO_x Reduction in a Sulphuric Acid Plant: *J. van Driel,* B. Giesen, A. Berryman, S. Sampat, S. Enevoldsen, H. Jensen-Holm

Water Vapor Effects on Oxidation of High-Temperature Materials: Alloys I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee
Program Organizers: Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA; Karren L. More, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6064 USA; Elizabeth J. Opila, NASA Glenn Research Center, Cleveland, OH 44135 USA

Monday PM	Room: 305
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: M. J. McNallan, University of Illinois-Chicago, Matls. Sci., Chicago, IL 60607-7023 USA; P. F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA

2:00 PM
Breakaway Oxidation of Ferritic Stainless Steels in Water Vapor Atmospheres: *A. Galerie*[1]; S. Henry[1]; Y. Wouters[1]; L. Antoni[2]; [1]Institute National Polytechnique de Grenoble, Laboratoire de Thermodynamique et de Physicochimie Métallurgiques, UMR CNRS/INPG/UJF 5614, BP 75, Saint Martin d'Heres 38402 France; [2]Usinor Recherche et Development, Centre de Recherches d'Ugine, Avenue Paul Girod, Ugine 73000 France

The well-known breakaway oxidation of ferritic stainless steels occurring in water vapor-containing atmospheres appears after an incubation period where a chromia-rich scale grows slowly. It is characterized by nucleation and rapid growth of iron oxide nodules. The mechanisms responsible for this remarkable effect of water vapor are reviewed and discussed under the light of a series of experimental results on 12 to 18%Cr-containing stainless steels. Mechanical features, as microcracking of the chromia scale, are shown to be of importance, but cannot alone explain what is observed. Scale volatilization may occur, but it was calculated to be negligible in oxygen-free water vapor atmospheres. Observations of iron nodule nucleation associated to thermodynamic calculations showed that stress levels are not high enough to induce iron oxide stability at the stainless steel-chromia interface, but that interface microporosity would promote the right driving force. Kinetic considerations, based on the concept of surface acidity promoting H_2O molecule dissociation, help to understand the rapid growth of the iron oxide nodules.

2:30 PM
The Oxidation Behaviour of Ferritic-Martensitic 9% Cr Steels in H2O Containing Environments: *M. Schütze*[1]; [1]Dechema E.V.,

Karl-Winnacker-Inst., Theodor-Heuss-Allee 25, Frankfurt am Main D-60486 Germany

Three commercial 9% Cr steels (P91, E911, Nf616) and 6 laboratory versions of Nf616 with Si contents from 0.04 to 0.78 were investigated at 650°C in dry synthetic air and air enriched with 4 and 10% water vapour, respectively. The aim of these investigations was to measure the time to breakaway for the different steels in these atmospheres and to elucidate the mechanisms responsible for the different behaviour. It was observed that the steels can be classified by 3 groups of behaviour. The first group exhibits breakaway behaviour almost from the beginning of oxidation and oxidizes with high rates. Steels of the second group show protective behaviour at the beginning but after several 100 to 1000 hrs. breakaway oxidation starts. In the third group no breakaway behaviour was observed for at least 10,000 hrs. As a general tendency it was found that increasing the water vapour content in the environment decreases the time to breakaway and shifts steels of groups II and III into group I or II, respectively. Silicon has a beneficial effect and increases the time to breakaway or possibly even suppresses breakaway completely. Interestingly steels which form a very thin protective oxide scale show a mass decrease during oxidation in atmospheres of high water vapour content. As revealed by UV absorption spectroscopy CrH2O4 evaporates from the surface to an extent which makes the mass decrease only detectable for slowly growing scales. The results from microstructural investigations are used for explanation of the behaviour of the different steels.

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Void and Crack Formation in Oxide Scales on Ferritic 9-12%Cr Steels during Steam Oxidation: *W. J. Quadakkers*[1]; J. Ehlers[1]; J. Zurek[1]; L. Singheiser[1]; [1]Forschungszentrum Jülich, Inst. for Matls. & Proc. in Energy Sys., Jülich 52425 Germany

In future, high efficiency fossil fuel fired power plants steam parameters will be increased in the range of 600-650°C and 300 bar. To meet these stringent stress conditions, new ferritic-martensitic 9-11%Cr steels have been developed to replace commonly used steels such as 1CrMoV and 12CrMoV. One of the life time limiting factors of the new high strength steels at the high service temperatures is the surface scale formation occurring during service in steam environments. In this context, not only the growth rates but also the spalling resistance of the scales is an important issue. The spalling characteristics of the magnetite based surface oxides appear to be strongly affected by the formation and growth of in-scale voids. In the present study, the mechanisms of void formation and growth was studied for a number of ferritic steels during oxidation in a simulated steam environment (Ar-50% H2O) at temperatures between 550 and 650°C with exposure times ranging from a few to 5000 hours. The results indicate that void formation is not related to one single mechanisms. In the early stages of oxidation gaps and voids can be formed by formation of a volatile specie, likely Fe-hydroxide. The voids formed by this mechanism heal during prolonged exposure. After longer exposure times formation of tiny voids starts in the inner scale as a result of vacancy condensation. The void nucleation and morphology is related to spinel stringers resulting from oxidation of Cr-rich carbides. In the outer part of the oxide scale formation of large voids starts only after prolonged exposure times. They result from impeded transport of Fe cations through the inner scale. As scale cracking and spalling characteristics during thermal cycling are strongly affected by the location and morphology of voids, knowledge of the void formation mechanisms is of great importance in the estimation of actual materials long term behaviour.

3:30 PM

The Corrosivity of H_2O/O_2 Environments towards Chromia-Forming Austenitic and Ferritic/Martensitic Steels at 500-800°C: *Karin Segerdahl*[1]; Henrik Asteman[1]; Jan-Erik Svensson[1]; Lars-Gunnar Johansson[1]; [1]Chalmers University of Technology, High Temp. Corrosion Ctr., Kemivägen 10, Göteborg 412 75 Sweden

The effect of water vapor on the oxidation of chromia-forming steels with 11-25% Cr between 500 and 800°C is reported. The samples are investigated by thermogravimetry, GI-XRD, SEM/EDX, and SAM. The oxidation Fe/Cr and Fe/Cr/Ni steels in H_2O/O_2 environment is strongly affected by gas velocity because chromium from the oxide is vaporized in the form of $CrO_2(OH)_2$. The oxidation behavior at a certain temperature, gas composition and gas velocity is to a large extent determined by the ability of the metallic substrate to supply the oxide with chromium to compensate for the losses by vaporization. The corrosion resistance of chromia-forming steels is enhanced by high Cr concentration, fast diffusion in the steel bulk (ferrite/martensite rather than austenite), and a high density of steel grain boundaries (small grain size). The corrosivity of the environment increases with the concentration of water vapor and oxygen, with the gas velocity and with temperature.

4:00 PM

Effect of Chromium and Nickel Contents on High Temperature Oxidation of Stainless Steels in Mixed Air and Water Vapor: *R. Peraldi*[1]; J. A. Schenning[1]; B. A. Pint[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., Corrosion Sci. & Tech., PO Box 2008, Oak Ridge, TN 37831-6156 USA

The corrosion resistance of chromia-forming stainless steels depends on their potential to form and maintain a protective Cr-rich oxide scale. Accelerated corrosion attack has been observed for some commercial stainless steels exposed to exhaust gas at 600°-800°C. The rapid growth of an Fe-rich oxide scale significantly reduces the performance of thin-walled components in power generation applications. In order to better understand alloy composition effects, the oxidation behavior of several model alloys with Ni and Cr contents ranging between 0-30wt.% and 10-20wt.%, respectively, have been studied in air + 10vol.% water vapor under various isothermal and cyclic conditions. Performance improvements were observed by increasing either the Ni or Cr contents of the steel. However, corrosion-resistant compositions must maintain good creep properties and meet cost targets. Other factors to be investigated are the effects of alloy grain size, phase composition, surface preparation, and minor alloy additions. The composition and microstructure effects observed in this study also will provide data for life-prediction models and may suggest a mechanistic explanation for the effect of water vapor. Research sponsored by the Microturbines Program, Office of Power Technologies, US Department of Energy (DOE), under contract DE-AC05-00OR22725 with UT-Battelle, LLC, and supported, in part, by an appointment to the Oak Ridge National Laboratory Postdoctoral Research Associates Program administered jointly by the Oak Ridge Institute For Science and Education and Oak Ridge National Laboratory.

4:30 PM Cancelled

High Temperature Oxidation of Ferritic Cr-Steels for Solid Oxide Fuel Cell Application: *P. B. Friehling*

5:00 PM

Thermochemical Analysis of Oxidation and Corrosion Processes in High Temperature Fuel Cells: *P. Singh*[1]; Gary Yang[1]; [1]Pacific Northwest National Laboratory, Richland, WA 99352 USA

This paper examines various oxidation and corrosion processes operating in high temperature fuel cells. Mechanisms for the scale growth in the multi component fuel gas environment containing H_2, H_2O, CO, CO_2, CH_x etc. are proposed and thermochemistry of oxidation processes in the redox gas atmosphere (H_2-H_2O), oxide stability, hydrated oxide formation etc. are analyzed. Role of substrate and oxide stability on the long-term performance of fuel cells are presented and discussed in detail.