Microanalysis Society

Topical Conference

Program and Abstracts

Carnegie Mellon University, Pittsburgh, PA

June 19-21, 2012
Microanalysis Society

Topical Conference

Organizing Committee:
Andrew Deal, GE Global Research
Steven Claves, Bechtel Marine Propulsion Corporation
Desmond Moser, The University of Western Ontario
Yoosuf Picard, Carnegie Mellon University

Ex Officio Committee Member:
Joe Michael, Sandia National Laboratories

Local Arrangements:
Marygrace Antikowski, Tom Nuhfer, Adam Wise, Jason Wolf,
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Additional Logistical Support:
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Jeff Davis, NIST-Bethesda
John Fournelle, UW-Madison
John McCarthy, UW-Madison
Welcome to EBSD 2012

The organizers would like to welcome you to the third topical conference on Electron Backscatter Diffraction (EBSD), sponsored by the Microanalysis Society (MAS) and hosted by Carnegie Mellon University. The first two meetings were held at the University of Wisconsin in 2008 and 2010, and both were very successful.

MAS was originally founded over 40 years ago “to advance and disseminate knowledge concerning the principles, instrumentation and applications of microanalysis.” MAS strives to support the spread of knowledge to the next generation of scientists and technologists in the nano- and microanalytical fields.

This year's EBSD topical conference will follow in the footsteps of the prior meetings, with a leadoff day of tutorials presented by experts in the field. During this initial day, manufacturers of EBSD equipment will also be demonstrating the technique on four SEMs, live in the J. Earl and Mary Roberts (JEMR) Microstructural Characterization Suite. These SEMs are made available by the Materials Science and Engineering department at Carnegie Mellon University. The following two days of EBSD 2012 comprises the technical symposia, with excellent invited speakers, contributed talks, and posters. We are extremely pleased with the response to this meeting. We particularly welcome the many students – close to 40, nearly one fourth of the registrants – that are attending. We encourage them to interact with the over 100 EBSD professionals and experts that are also in attendance.

EBSD 2012 was made possible by the generous financial support of our commercial sponsors, and we, on behalf of MAS, are extremely grateful. Likewise, we would like to thank the National Science Foundation for providing substantial scholarships to nearly two dozen students. We also appreciate all of the volunteers who have helped make EBSD 2012 a reality. Finally, we offer our thanks to Carnegie Mellon University for the use of their excellent facilities.

Welcome to EBSD 2012. We hope you enjoy the conference!

Andrew Deal    Steven Claves    Desmond Moser    Yoosuf Picard

Pittsburgh, PA    June 19, 2012
EBSD Listserver

One of the recommendations from the first conference, EBSD 2008, was the creation of a listserver dedicated to EBSD questions. One has been created (ebsd@lists.wisc.edu, hosted at the University of Wisconsin-Madison) and if you are interested, you can join.

Log in to the web interface at https://lists.wisc.edu/read/?forum=ebsd and select “all forums” in left menu, then scroll down to “ebsd” in the list, then click ‘subscribe’ in the right column and enter your email address and name.

Within a day or two, you should get an email indicating that you have been subscribed.

As is the case with such lists, weeks can go by with nary a word, and then there is a burst of discussion . . . .

It is archived, so past postings are searchable.

If you have questions, contact the list manager, John Fournelle at johnf@geology.wisc.edu

This conference and others like it are organized by MAS. We are an organization composed of individuals in universities, government and private industry labs who utilize a range of analysis instruments in the course of our study of a variety of materials, ranging from meteorites to metals to ceramics to volcanic ash (and sometimes even food).

We provide opportunities for students and young (and some not-so-young) professionals to network, learn about new techniques, ask questions about hard-to-study materials, and find post-doc opportunities or even jobs.

Membership in the Microanalysis Society is inexpensive ($40), and members receive several benefits, including reduced rate subscriptions to the Microscopy and Microanalysis journals. Students can join for only $10 a year and receive FREE the Microscopy and Microanalysis journal (otherwise it is $20/year). Everyone receives the monthly Microscopy Today magazine. There are reduced rates for attending the annual Microscopy and Microanalysis conventions, where all the major instrument manufacturers display.

Go to www.microbeamanalysis.org for more information
Program Schedule

Mon., June 18th
3:00pm – 6:00pm  | ON-SITE CHECK-IN & DEMONSTRATION SIGN-UP  | RANGOS BALLROOM
Receive conference badge, booklets, banquet ticket, etc.
Sign-up for open live demonstration times held the following day

Tues., June 19th
7:30am – 5:30pm  | TUTORIAL & LIVE DEMONSTRATIONS  | Rangos Ballroom
Continued on-site check-in and live demonstration sign-up
9:00am – 12:00pm  | Open live demonstrations (requires prior sign-up)  | JEMR Characterization Suite, Roberts Engineering Hall
11:00am  | Sample Preparation (Brian Hess)  | Rangos Ballroom
11:30am  | Sample Preparation (Ron Witt)  | Rangos Ballroom
12:00pm  | ------LUNCH------  | GM/Fosters Dining Rooms
1:00pm – 3:00pm  | Open live demonstrations (requires prior sign-up)  | JEMR Characterization Suite
1:00pm  | Data Acquisition (John Sutliff)  | Rangos Ballroom
2:00pm  | Data Processing (Tony Rollett)  | Rangos Ballroom
3:00pm  | ------BREAK------  |
3:15pm  | Materials Science Case Studies (Joe Michael)  | Rangos Ballroom
AND (parallel session)
3:15pm  | Geoscience Live Demonstration  | JEMR Characterization Suite
4:15pm  | ------BREAK------  |
4:30pm  | Geoscience Case Studies (Des Moser)  | Singleton Room, Roberts Hall
AND (parallel session)
5:30pm  | Social hour  | Singleton Room & Atrium, Roberts Hall
5:30pm – 8:00pm  | Open live demonstrations (requires prior sign-up)  | JEMR Characterization Suite
## EBSD 2012

**An MAS Topical Conference on Electron Backscatter Diffraction**

### TECHNICAL SYMPOSIA, DAY 1

**Wed., June 20th**

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
<th>Location</th>
<th>Presenter</th>
</tr>
</thead>
<tbody>
<tr>
<td>8:00am – 12:00pm</td>
<td>Continued on-site check-in</td>
<td>All</td>
<td></td>
</tr>
<tr>
<td>8:00am – 8:30am</td>
<td>Congregate &amp; coffee</td>
<td>All</td>
<td></td>
</tr>
<tr>
<td>8:30am – 8:40am</td>
<td>Welcome &amp; announcements</td>
<td>All</td>
<td>Andrew Deal</td>
</tr>
</tbody>
</table>

#### Session 1

<table>
<thead>
<tr>
<th>Time</th>
<th>Title</th>
<th>Speaker</th>
</tr>
</thead>
<tbody>
<tr>
<td>9:20am</td>
<td>EBSD Applications to Predict Nuclear Fuel Performance [A-41]</td>
<td>Isabella van Rooyen</td>
</tr>
<tr>
<td>9:40am</td>
<td>Deformation Characterization of Zircaloy-4 Sheet Deformed by Plane Strain Compression [A-33]</td>
<td>Alexey Rempel</td>
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<tr>
<td>10:00am</td>
<td>------BREAK------</td>
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#### Session 2

<table>
<thead>
<tr>
<th>Time</th>
<th>Title</th>
<th>Speaker</th>
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<tbody>
<tr>
<td>10:30am</td>
<td>EBSD Analysis in the Geosciences: High Temperature Mylonites [A-26]</td>
<td>Elena Miranda (Invited)</td>
</tr>
<tr>
<td>11:10am</td>
<td>Seismic Anisotropy In Subduction Zones From EBSD Measurements of Antigorite Crystal-Preferred Orientations (CPO) [A-3]</td>
<td>Sarah Brownlee</td>
</tr>
<tr>
<td>11:50am</td>
<td>------LUNCH------</td>
<td>General Motors/Fosters Dining Rooms</td>
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#### Session 3

<table>
<thead>
<tr>
<th>Time</th>
<th>Title</th>
<th>Speaker</th>
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</thead>
<tbody>
<tr>
<td>1:00pm</td>
<td>Measuring Lattice Strain with High Resolution EBSD [A-17]</td>
<td>David Fullwood (Invited)</td>
</tr>
<tr>
<td>1:40pm</td>
<td>Some Advances in and Applications of Cross-Correlation Based Analysis of EBSD Patterns [A-42]</td>
<td>Angus Wilkinson</td>
</tr>
<tr>
<td>2:00pm</td>
<td>An Investigation of Grain Orientation and Strain Fields in BCC Tantalum [A-4]</td>
<td>Jay D. Carroll</td>
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<tr>
<td>2:20pm</td>
<td>------BREAK------</td>
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#### Session 4

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<thead>
<tr>
<th>Time</th>
<th>Title</th>
<th>Speaker</th>
</tr>
</thead>
<tbody>
<tr>
<td>2:50pm</td>
<td>Industrial Application of Orientation Imaging Microscopy [A-54]</td>
<td>Hasso Weiland (Invited)</td>
</tr>
<tr>
<td>3:30pm</td>
<td>Round Robin of EBSD Grain Size Measurements (ASTM E2627) [A-49]</td>
<td>Andrew Deal</td>
</tr>
<tr>
<td>3:50pm</td>
<td>Analyzing Void Nucleation &amp; Growth on the Spall Plane in Shock Loaded Aluminum with EBSD [A-34]</td>
<td>Nathaniel Sanchez</td>
</tr>
</tbody>
</table>

#### Poster Session

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>4:10pm – 4:20pm</td>
<td>Preview in RANGOS BALLROOM</td>
<td>RANGOS BALLROOM</td>
</tr>
<tr>
<td>4:20pm – 5:30pm</td>
<td>Poster session in CONNAN ROOM</td>
<td>CONNAN ROOM</td>
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</tbody>
</table>

#### BANQUET

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
<th>Location</th>
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<tbody>
<tr>
<td>6:00pm – 7:00pm</td>
<td>Drinks at Tartans Pavillion (1ST Floor of Resnik House)</td>
<td>Tartans Pavillion</td>
</tr>
<tr>
<td>7:00pm – 9:00pm</td>
<td>Dinner at Tartans Pavillion</td>
<td>Tartans Pavillion</td>
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</table>
EBSD 2012
An MAS Topical Conference on Electron Backscatter Diffraction

Thurs., June 21st
8:00am – 8:30am TECHNICAL SYMPOSIA, DAY 2 RANGOS BALLROOM
Congregate & coffee All

Session 5
8:30am Development and Application of a Novel Characterization System to Quantify Grain Structures in 3D [A-40] Michael Uchic (Invited)
9:50am In-situ Analysis of the Tensile Deformation Mechanism in Rolled AZ31 and Extruded MN11 Magnesium Alloys [A-8] Zhe Chen
10:10am -----BREAK-----

Session 6
11:00am EBSD Of Lunar Zircon and Baddeleyite Grains in Meteorite NWA 2200 – Witnesses to the Late Heavy Bombardment? [A-13] James Darling
11:40am Correlating Planar Microstructures on 3D Grain Exteriors and 2D Polished Interiors of Zircon from the Vredefort Dome, South Africa [A-16] Timmons Erickson
12:00pm Characterization of 19th Century and Modern Daguerreotypes Using EBSD [A-7] Lisa Chan
12:20pm -----LUNCH-----General Motors/Fosters Dining Rooms

Session 7
1:20pm Microstructure Optimization of Laser-Engineered Net-Shape (LENS) Formed Ti [A-52] David Field (Invited)
2:00pm Fast Orientation Projection using OpenGL [A-28] Gert Nolze
2:40pm Characterizing Artifacts in Local Misorientation Measurements at Grain Boundaries [A-43] Stuart Wright
3:00pm Closing remarks and adjourn Andrew Deal
### Poster Session
**Wednesday, 4:20-5:30 pm; Connan Room**

<table>
<thead>
<tr>
<th>A-1</th>
<th>Ousama Abdelhadi</th>
<th>“Optimization of Preparation Procedure for Successful Electronic Backscatter Diffraction (EBSD) of Multi-Layer Specimen”</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-5</td>
<td>Nicolau Castro</td>
<td>“EBSD as a Tool to Analyse Strain on the Edge Cutting of Steel Sheets”</td>
</tr>
<tr>
<td>A-6</td>
<td>Rocco Cerchiara</td>
<td>“Preparation of Materials for EBSD using an Adjustable Broad Beam Ion Source”</td>
</tr>
<tr>
<td>A-9</td>
<td>Kailynn Cho</td>
<td>“EBSD analysis of Pt-20Ir Wire as Lead Conductor in Implantable Medical Device”</td>
</tr>
<tr>
<td>A-11</td>
<td>Ellen Crapster-Pregont</td>
<td>“Potential Applications of EBSD to Extraterrestrial Samples”</td>
</tr>
<tr>
<td>A-12</td>
<td>Robert Stroud</td>
<td>“TEM Alternative to SEM-EBSD Based Orientation Imaging”</td>
</tr>
<tr>
<td>A-14</td>
<td>Nafiseh Ebrahimi</td>
<td>“Grain and Grain Boundary Corrosion Resistance of Ni-Cr-Mo Alloys”</td>
</tr>
<tr>
<td>A-18</td>
<td>Ali Gholinia</td>
<td>“High resolution analysis with the aid of the latest SEM and EBSD systems”</td>
</tr>
<tr>
<td>A-19</td>
<td>Daniel Goran</td>
<td>“Dealing with the limitations of “live” orientation contrast imaging and EBSD analysis on materials deformed by in-situ compression and tensile testing”</td>
</tr>
<tr>
<td>A-20</td>
<td>Jenny Goulden</td>
<td>“Characterization of Hydrides in a Zirconium Alloy by EBSD”</td>
</tr>
<tr>
<td>A-21</td>
<td>Wolfgang Gruenewald</td>
<td>“Preparation of Perfect Sample Surfaces for EBSD Using Ion milling”</td>
</tr>
<tr>
<td>A-22</td>
<td>Hongmei Li</td>
<td>“Analysis of Deformation Behavior of Commercially Pure Titanium and Ti-5Al-2.5Sn (wt.%) using In-situ Scanning Electron Microscopy and Electron Backscattered Diffraction”</td>
</tr>
<tr>
<td>A-23</td>
<td>Yi-Chia Lu</td>
<td>“Factors Controlling the Crystal Morphology and Orientation of Carbonate Minerals Precipitated in the Pipe of hot spring, Taiwan”</td>
</tr>
<tr>
<td>A-24</td>
<td>Michael McMurtrey</td>
<td>“Determination of the stress and strain states at dislocation channel-grain boundary intersections in irradiated stainless steels”</td>
</tr>
<tr>
<td>A-30</td>
<td>Chad Parish</td>
<td>“Using EBSD to aid understanding of irradiation creep in CVD-3C SiC”</td>
</tr>
<tr>
<td>A-31</td>
<td>Gert Nolze</td>
<td>“The BSE signal as an additional tool for phase assignment in EBSD”</td>
</tr>
</tbody>
</table>
### A-32 Pitcheswara Kamineni

"Effect of Hot Compression Conditions on Microstructure and Texture of TX32 and TXA321 Magnesium Alloys"

### A-36 Thomas Schwager

"New approach to characterizing multi-phase materials by advanced EBSD/EDS integration"

### A-37 James Seal

"In-Situ Characterization of slip transfer across α/β interfaces in equiaxed Ti-5Al-2.5Sn (wt. %) using EBSD and Microcantilever Beams"

### A-38 Clayton Stein

"An Analysis of Fatigue Crack Initiation Using 2d Orientation Mapping and Full-Field Simulation of Elastic Stress Response"

### A-39 Kameshwaran Swaminathan

"Dependence of creep fracture on microtexture of polycrystalline Ni-based cast superalloys"

### A-44 Jieqiong Wu

"EBSD study of dynamic recrystallization mechanisms during hot deformation in Nimonic 80A"

### A-46 Ajith Chakkedath

"In situ study of the tensile deformation and fracture mode in peak-aged Mg-11Y-5Gd-2Zn-0.5Zr alloy"

### A-47 Lisa Zellmer

"Investigation of the microstructure in the vicinity of a fatigue crack in in-depth direction in a ferritic-martensitic steel"

### A-48 Bite Zhou

"Sn crystal orientation and microstructure evolution during thermal cycling in a high-stress package design"

### A-50 Gene Lucadamo

"Investigating the Relationship between Oxide Thickness and Crystallographic Orientation in a Zirconium Alloy using Electron Backscatter Diffraction (EBSD)"
Main Meeting Area in: 29 University Center
JEMR Facility in: 22 Roberts Engineering Hall
Wed. Dinner Banquet in: 54 Resnik House (Tartans Pavilion)
Main Parking Garage: P6 Resnik House (Tartans Pavilion)
University Center 2nd Floor

- Rangos Hall
  - Sign-In, Main Meeting Room, and Vendor Exhibit Hall
- Foster and General Motors Dining Rooms
  - Lunch Eating Areas

University Center 1st Floor

- Connan Room
  - Poster Session
- Kirr Commons and Merson Courtyard
  - Alternative Lunch Eating Areas
Exiting Rangos Hall

Rangos Hall to JEMR Facility (1 of 6)

Walking to 1st Floor Exit
Rangos Hall to JEMR Facility (4 of 6)

Take Stairs down 2 floors
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Optimization of Preparation Procedure for Successful Electronic Backscatter Diffraction (EBSD) of Multi-Layer Specimen

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Abstract
Electron backscatter diffraction (EBSD) is a powerful technique that provides a wide range of analytical data, such as crystallographic orientation, phase identification, and grain size measurements. The quality of diffraction pattern, which influences the quality of the indexing of the diffraction pattern, mainly depends upon the successful achievement of a very flat and fully distortion-free sample surface. Many studies have reported guidelines on specimen preparation for EBSD of a variety of materials such as metals and ceramics. However, limited studies have been documented in literature for optimized preparation of multi-layer specimens. In this study, a series of mechanical polishing procedures have been developed, which will be adequate for producing damage-free surfaces in multiple-layer specimens for EBSD. The proposed method was used to prepare solder joint specimen as an example of multiple-layer specimen, and has produced the best possible surfaces. The results suggested that using automated grinder-polisher followed by vibratory-polisher, with standard consumable products were a sufficient surface preparatory technique for producing quality EBSD patterns for microstructure of Sn-3.5Ag/Cu solder system, in a reasonable amount of time and at low cost. Vibratory polishing was found to be the key step in the preparation process. A poor pattern quality occurred, in particular, in copper regions, when vibratory polishing step was skipped or was conducted shorter than a certain amount of time.
Case Studies on the Application of EBSD for Phase Discrimination of Asbestiform Mineral Particulate

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Application of EBSD for phase discrimination of crystalline phases in bulk specimens has been commonplace since the advent of the technique. However, there are very few published studies that exploit EBSD as a tool for phase discrimination of unpolished particulate specimens. The use of scanning electron microscopy (SEM) for asbestos analysis has been limited due in part to the inability of most instruments to provide crystallographic information useful for phase identification of small particles. Given the ability of transmission electron microscopy (TEM) to image small particles, obtain chemical information through energy dispersive x-ray spectroscopy (EDS) and obtain crystallographic information through selected area electron diffraction (SAED) it has been the predominantly used tool for the identification of respirable asbestos particles. We will present three case studies that show how SEM/EDS/EBSD can be used for the discrimination of asbestiform particulate. The first case will present EBSD and transmission EBSD (tEBSD) results from regulated asbestos minerals. This case will demonstrate that SEM/EDS/EBSD can be used to accurately discriminate these minerals on a scale comparable to TEM/EDS/SAED. The second case will present results from particles of an asbestiform mineral associated with a precious metal deposit. This case is representative of the problem of natural occurrences of asbestos that are very difficult to accurately assess using standard methods originally designed for the analysis of industrial asbestos containing materials. The third case will present preliminary results of a study to characterize particulate found in the lungs of industrial workers. The particles were previously analyzed by SEM/EDS alone and found to be an aluminosilicate polymorph, but without crystallographic information it was impossible to determine the identity of the material. This case is representative of the limitations of EBSD as a tool for phase discrimination from particulate materials.
Seismic anisotropy in subduction zones from electron backscatter diffraction (EBSD) measurements of antigorite crystal-preferred orientations (CPO)

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Seismic anisotropy is often used to infer flow directions in the mantle, but anisotropy in the tip of a subduction zone mantle wedge might more likely be related to mantle alteration (e.g., hydration of olivine). Understanding the mechanisms responsible for anisotropy in subduction zones provides a link between seismic observations and the processes occurring in subduction zones. Kneller et al. [2008] suggested that a type-B olivine crystal preferred orientation (CPO) in the tip of a mantle wedge can explain some, but not all, of the observed trench-parallel shear-wave splitting observations, and suggested that the growth of antigorite might explain the remainder. We investigate the potential contribution of antigorite to subduction zone anisotropy by measuring the CPO of antigorite in 7 serpentinites from central Guatemala using electron backscatter diffraction (EBSD) and calculating the seismic properties using the single-crystal elastic constants of antigorite from Bezacier et al. [2010].

Because antigorite can be quite difficult to prepare for EBSD, we polished each sample by hand in a stepwise fashion. We achieved the highest quality diffraction patterns when the samples were measured uncoated in low vacuum (70 Pa), using a working distance of 10–15 mm, and accelerating voltage of 20 kV. Each sample was mapped twice, once with a fine step size (1-2 µm) to image the grain size and microstructure of a small region (1 mm²), and once with a coarse step size (50-100 µm) to obtain 1-point-per-grain measurements to characterize the antigorite CPO over most of each thin section (1-3 cm²). Fine-scale band contrast images show a range of microstructures, from slightly elongated plates to submicron needles. Our results show two types of antigorite CPO - one related to deformation and one related to the CPO of the parent material - which result in calculated anisotropies of 6-28% in Vp and 5-33% in Vs. Depending on the distribution of antigorite within a subduction zone mantle wedge this anisotropy is sufficient to explain the discrepancy between observed seismic anisotropy and that predicted by type-B olivine CPO.
An investigation of grain orientation and strain fields in BCC tantalum

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Body centered cubic (BCC) metals have more complex slip systems than face centered cubic (FCC) metals. Consequently, the relationship between local strain and microstructure in BCC metals is not well understood. Experimental measurements of grain level deformation have the potential to provide valuable insight into crystallographic slip in BCC metals.

In this work, a powerful experimental technique combining electron backscatter diffraction (EBSD) and digital image correlation (DIC) was developed to measure grain-level material deformation with extraordinary detail. This technique was then used to investigate the relationship between deformation and microstructure in a tantalum oligocrystal (14 grains). Millimetric-sized grains were achieved through annealing to generate a pseudo-two-dimensional grain structure. Grain orientation and strain accumulation were measured at several levels of applied strain throughout a region of interest measuring 5.0 x 1.6 mm. By stitching several (48) DIC strain fields together, excellent sub-grain level resolution was achieved over a relatively large region of interest. Similarly, the results of several (12) EBSD scans were stitched together to obtain the microstructural orientation throughout the entire region at each strain level. These sub-grain-level, full field DIC and EBSD measurements were then spatially linked to one another using fiducial markers on the specimen surface.

The experimental measurements obtained through this technique were used to evaluate several microstructure-slip relationships in tantalum. The relationships between grain orientation, grain rotation (and the resulting local misorientation), and local strain accumulation were examined. Additionally, the relationships between Schmid and Taylor factors to deformation within grains were investigated. Although there is debate concerning the usefulness of these stress projection factors in BCC metals due to the tension-compression asymmetry, a significant correlation was found between Schmid factor and local strain accumulation within grains.

As this work progresses, more refined stress projection measures will be compared to material deformation. This study provides a better understanding of the behavior of BCC metals at the grain level and highlights the importance of neighboring grains in local deformation. Experiments such as those shown in this work are currently being used to develop and evaluate the fidelity of crystal plasticity models for BCC metals.
The strain distribution on the edge cutting of steel sheets is important in order to explain the stretch-flangeability performance. In this study, electron backscatter diffraction (EBSD) in conjunction with scanning electron microscopy was applied to measure the plastic strain distribution imposed by cutting or machining of an interstitial free (IF) steel. The effect of the shearing clearance on the strain distribution was evaluated and correlated to the stretch-flangeability performance. The results show that smaller shearing clearances provide lower level of strain near the sheared edges and better hole expansion performance.
Preparation of Materials for EBSD using an Adjustable Broad Beam Ion Source

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Analysis by Electron Backscatter Diffraction (EBSD) requires that a sample surface be free of deformation, oxidation and hydrocarbon contamination. The options for creating such a surface include mechanical methods, electro-polishing and ion based techniques. [1-2]

Sample preparation protocols will be presented as optimal combinations of mechanical grinding and broad beam ion milling techniques.

The initial goal is to produce a parallel - sided sample with a surface finish of less than 1 µm. This sample geometry is required so that tilting may occur eucentrically with respect to an ion source. Precise tilting permits large surface areas to be ion milled at low incident angles, in the minimum amount of time. Reducing the processing time enhances sample quality while optimizing throughput.

To advance this research, ion milling was conducted in an instrument equipped with a novel Ar ion source designed with an adjustable ion beam diameter. The ion source voltage ranged from 6 keV to 100 eV, while the ion beam current was independently variable from 10’s of uA to 100’s of nA. Application examples will be presented that depict the effects of adjustment of the ion beam diameter. Increased sample quality will be established based on improvements in SEM imaging and a quantitative enhancement of the EBSD results.

References

Daguerreotype is the first viable photographic process that was developed in the 19th century. Unlike other silver-based black and white photographic process, the daguerreotype is an image that rests on the surface of a highly polished silvered copper plate. The final stage of the daguerreotype process is to affix the silver-mercury amalgam particles to the plate by passive electrochemical coating with a thin gold film. Even though gold is a noble metal, tarnishing readily occurs on the image particles and the background surface of the daguerreotype. This work presents a study of 19th century and modern contemporary daguerreotypes using EBSD to determine the crystallographic orientation of the silver and gold grains that are tens of nanometers in size on the background surface. The microstructural and crystallographic information can potentially explain the occurrence of tarnish as corrosion in the intergranular boundaries. Challenges involved in performing EBSD on the daguerreotypes will also be discussed.
In-situ Analysis of the Tensile Deformation Mechanism in Rolled AZ31 and Extruded MN11 Magnesium Alloys

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In-situ tensile experiments were performed on a rolled AZ31 (Mg-3Al-1Zn wt.%) and an extruded MN11 (Mg-1Mn-1Nd wt.%) magnesium (Mg) alloy in a scanning electron microscope at 323K (50°C), 423K (150°C), and 523K (250°C). Electron backscatter diffraction was performed on selected areas of the specimens both before and after the deformation. The deformation system corresponded to the slip traces or twinning observed during the in-situ experiments was determined based on EBSD analysis which gives information on the orientation of individual grains.

For AZ31 alloy, extension twinning, basal, and non-basal slip were active at 323K (50°C). Extension twinning was not observed at 423K (150°C), suggesting that the critical resolved shear stress (CRSS) of non-basal systems becomes less than that of extension twinning at T<423K (150°C). From 423K (150°C) to 523K (250°C), a transition in the dominant deformation mechanism from basal + prismatic <a> to mainly prismatic <a> slip was observed. This is consistent with the decrease of the CRSS of non-basal slip systems with increasing temperature.

For MN11 alloy, extension twinning, basal, and non-basal slip were active at 323K (50°C). At 423K (150°C), much less extension twinning was observed, while basal slip and prismatic <a> slip were dominant and presented similar activities. At 523K (250°C), twinning was not observed, and basal slip became dominant. It is therefore suggested that at low temperatures, the CRSS of basal slip in MN11 alloy is increased with respected to that of pure Mg and convention Mg alloys such as AZ31, possibly due to the preferential location of rare earth (RE) atoms along basal planes. With increasing temperature, the faster diffusion of RE atoms lead to a decrease of the CRSS of basal slip, which leads ultimately to easier slip along basal planes.
EBSD ANALYSIS OF PT-20IR WIRE AS LEAD CONDUCTOR IN IMPLANTABLE MEDICAL DEVICE

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Abstract

Pt-20Ir cold drawn wire has been used as lead conductor in implantable medical devices. The lead is a device to transfer the electrical signal to the area for stimulating such as brain and neurological nerves. The Pt-20Ir wire demonstrated the good biocompatibility and stability. Although Pt-20Ir wire has been used in leads for a long time, there is lack of published data in literature on its microstructure. Due to inert nature the Pt and its alloys are difficult to be prepared as metallograph for grain structure analysis. By using EBSD technique, the Pt-20Ir sample does not need to be etched and its high atomic number is able to generate high quality backscattered pattern. In this study, the Pt-20Ir wire was analyzed for its grain structure, grain boundary and texture.
A characterization method by 3D EBSD for grain boundary (GB) triple junctions (TJ) has been developed. In a 3D EBSD dataset each data entry corresponds to the crystal orientation at a “voxel” at a location described in the sample frame by 3 orthogonal axes.

The method defines a TJ "segment" as a unit straight line along one of the axes and is situated in between four adjacent voxels. The length of a TJ segment equals the distance between adjacent voxels along the axis. A TJ segment satisfies the condition that its nearest voxels are occupied by three different grains (a grain triplet). Segments surrounded by the same grain triplet are regarded as in the same TJ. A TJ, designated by the same grain triplet, can be in a single continuous line or in several continuous lines, separated by other TJ. All TJs in the 3D dataset can be calculated by going through grain triplets in the dataset in turn. For a sufficiently long TJ (> 15 segments), a convolution procedure can be applied to obtain the approximate TJ line directions at different segments. The convoluted directions of TJ segments are placed to a stereographic projection in the sample frame (a pole figure) as "points". The positions of these points can be compared with the projected positions of the major crystal directions in the grain triplet, measured by EBSD mapping, in the same pole figure.

This method has been applied to a nickel superalloy 3D dataset. A TJ in between two $\Sigma 3$ boundaries and a $\Sigma 9$ boundary has been studied in detail. In this example a small and near flat $\Sigma 9$ boundary is enclosed in a large and flat $\Sigma 3$ boundary. The other $\Sigma 3$ boundary is highly curved. The TJ line is with an elliptical shape and in a plane shared by the flat $\Sigma 3$ and the $\Sigma 9$ boundaries. The convoluted directions of TJ segments in the sample frame form a band of points in the pole figure. The points in the band are distributed non-uniformly but densely populated along a stretch, in agreement with the fact that the TJ is in a plane and has an elongated elliptical shape. Compared with projected major crystal orientations of the 3 related grains in the pole figure, it is clear that the elongated direction of the elliptical TJ line is nearly parallel to a [110] direction common to the relevant grain triplet, and spread to the other [110] direction common to two grains in the triplet.

By using this method comprehensive information on TJ lines in a sample can be obtained.
Potential Applications of EBSD to Extraterrestrial Samples

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Extraterrestrial samples, mainly meteorites, are precious and rare whether originating from a differentiated body (e.g. Mars, Moon, some asteroids) or an undifferentiated body (e.g. carbonaceous chondrites). Minimally destructive sample preparation and analysis techniques that yield chemical and textural information are continually employed and sought after. Electron backscatter diffraction (EBSD) has the capabilities to add a microstructural component to the already existing extraterrestrial data for thick sections. With few exceptions (MNH-Imperial U.; petrofabric relationships between clasts and matrix in carbonaceous chondrites) the technique of EBSD has not been applied to the analysis of meteorites. Ongoing research at AMNH requires the use of EBSD to successfully address the following research questions. Distinguish between accretion and crystallization in chondrules by analyzing the orientation of olivine in the outermost regions and rim-like structures associated with olivine-rich chondrules. Use the orientation of grains in calcium-and aluminum-rich inclusions, the most primitive solar system solids, to gain insight into how they grew in the solar nebula. Finally, relative orientations of individual grains or objects could be used to better quantify the extent of alteration, whether aqueous or metamorphic. Thus, the potential applications of EBSD as a non-destructive analytical tool for analyzing meteorites are significant. However, the expertise to carry out these investigations among meteoricists is lacking, making insights from EBSD experts in the materials and terrestrial sciences essential to the successful application of EBSD to extraterrestrial samples.
TEM Alternative to SEM-EBSD Based Orientation Imaging

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We review a recently developed technique known as ASTAR™, which is the counterpart of automated orientation imaging using scanning electron microscope (SEM) - electron backscattered diffraction (EBSD) analysis in the transmission electron microscope (TEM). Orientation imaging via SEM-EBSD analysis, though well established, is not suitable for investigating polycrystalline materials with average grain size in the 100 nm range due to the limitations in the spatial resolution of this technique. This necessitates the need for orientation imaging in the transmission electron microscope (TEM). In ASTAR™, a nanosized quasi-parallel electron probe is scanned across the sample and the spot diffraction patterns are collected from each point in the scan. The diffraction patterns are then indexed automatically using a pattern matching algorithm to generate the orientation map. In ASTAR™, the electron probe is precessed about the optic axis as it is scanned to reduce the strong dynamical effects. The use of precession enhances the quality of diffraction patterns collected and the reliability of the orientation solutions is greatly improved. We present several examples of the use of ASTAR™ for orientation imaging to investigate various microstructures. Besides orientation mapping, ASTAR™ is also capable of identifying different phases similar in composition but different crystal structures. We also show the application of various computational tools employed routinely on SEM-EBSD orientation maps to ASTAR™ maps to extract quantitative information on microtexture and crystallography of internal interfaces.
EBSD of lunar zircon and baddeleyite grains in meteorite NWA 2200 – witnesses to the Late Heavy Bombardment?

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Meteorites from the Moon, Mars and the asteroid belt offer tremendous insights into the evolution of our Solar System. They bear witness to the formation and differentiation of planets, as well as intensive bombardment of planetary surfaces that is predicted to have peaked early in Solar System history (e.g. Tera et al., 1974). In order to fully understand the record that these precious rocks provide, it is critical to resolve the effects of extreme compression and heating that occurs during planetary collisions from the original characteristics of the sample. Such “shock metamorphism” affects all meteorites on, and during their ejection from, planetary surfaces, and can produce recrystallisation, deformation and pathways for chemical exchange amongst their constituent mineral phases.

In this study, we focus upon the effects of shock metamorphism on uranium bearing minerals that are important chronometers in meteorites: baddeleyite (ZrO$_2$) and zircon (ZrSiO$_4$). These minerals are among the earliest to have crystallized in our solar system; however the microstructural effects of shock metamorphism, and the degree to which these effects cause diffusion and alteration of U-Pb isotopic dates, are in the early stages of understanding. This is particularly the case for baddeleyite (ZrO$_2$) where the ability to use EBSD to assess crystallinity, and map crystal orientation at the nano-scale is particularly useful in identifying potential diffusion pathways and grain boundary migrations.

Here we show that EBSD is a powerful tool for resolving the timing of solar system events when combined with chemical analysis (Secondary Ion Mass Spectrometry, X-ray spectroscopy) and other FEG-SEM imaging techniques (e.g. CL, BSE). Examples are provided of the application of these tools to both unshocked (terrestrial) and highly shocked (Martian) baddeleyite and zircon, providing a framework for ongoing investigation of the lunar meteorite NWA 2200. This sample is the product of ancient impact melting and deformation on the Moon, which is hypothesized to have experienced high bolide flux and Late Heavy Bombardment 3.9 billion years ago. NWA 2200 contains baddeleyites (< 20 microns) and zircons (< 30 microns), and EBSD orientation mapping of these grains reveals lattice distortion and twinning that is related to shock metamorphism. Isotopic dating results for baddeleyite and zircon in this sample are consistent with their having witnessed a major shock metamorphic event ca. 3.9 billion years ago, with zircon preserving evidence for crystallization as early as 4.4 billion years ago. The co-existence of these phases allows comparison of their microstructural response to shock metamorphism, and aids our ongoing EBSD and U-Pb dating of other meteorite suites.

References:
Grain and Grain Boundary Corrosion Resistance of Ni-Cr-Mo Alloys

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Ni-Cr-Mo alloys exhibit exceptional corrosion resistance and are widely used in harsh industrial environments. This resistance can be attributed to a combination of alloy composition and microstructure. We have been studying these features on the C-series of alloys supplied by Haynes International (Kokomo, IN). In this study, the susceptibility to corrosion of mill-annealed, polycrystalline Ni-Cr-Mo alloys, in hot, low pH, chloride solutions is being investigated using Electron Backscatter Diffraction (EBSD). A number of electrochemical and natural corrosion experiments are being conducted to determine the influence of alloy microstructure on the initiation of corrosion sites.

Corrosion in the form of pits can be linked to the crystallographic features on the grain and grain boundaries. Grain boundaries exhibiting coincidence site lattices, especially $\Sigma 3$, were most resistant to corrosion pitting, whereas non-coincidence site lattice grain boundaries ($\Sigma > 29$) were more susceptible.

Pitting attack occurred also preferentially at small zones within individual grains where a 5 – 10 degrees lattice misorientation was observed, Figure 1, suggesting that ‘sub-grain boundaries’ were also susceptible to corrosion.

Figure 1: Orientation map of corroded Alloy-22 surface. Corroded sites are shown as dark black.
Femtosecond Laser Ablation in the TriBeam System for 3-D EBSD Data Collection

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Femtosecond pulsed lasers can ablate material with extremely limited collateral damage, particularly when operating at low fluences. Various metals have been serially sectioned using a femtosecond laser that is incorporated into the TriBeam system, an instrument that couples the laser with a DualBeam FIB-SEM. Sample surfaces are laser machined in situ, within the vacuum chamber, and then subsequently imaged using any or all of the following detectors: SE, BSE, FSD, EBSD, and EDS. The detector selection is dependent upon the material being sectioned and the type of data required for segmentation. Quantification of laser damage is shown using optical microscopy, AFM, EBSD, and optical and laser profilometry. Reconstructions of 3-D EBSD datasets are shown for several materials, including polycrystalline Ni, CuNb nanolaminates, and a CuW composite, which have been subjected to large amounts of laser irradiation during serial sectioning experiments.
Correlating planar microstructures on 3D grain exteriors and 2D polished interiors of zircon from the Vredefort dome, South Africa

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Meteorite impacts form unique microstructures within the minerals of target rocks as a consequence of rapid compression, extension and heating by the shock wave; referred to broadly as shock metamorphism. Impact microstructures form in the mineral zircon at shock loading pressures >20 GPa and range from crystallographically oriented planar microstructures to granular (polycrystalline) textures at higher pressures (>50 GPa). Impact craters on Earth are susceptible to erosion and tectonic modification; however zircon is refractory and the shock microstructures are resistant to annealing. Zircon thus can preserve evidence of impact cataclysm over long geologic time scales and through complicated geologic histories postdating the impact event.

We examined 32 subhedral zircons from sediment samples containing detrital shocked minerals derived from the core of the Vredefort dome impact structure in South Africa. One goal was to fully document the absolute 3D orientation and distribution of planar shock features. The grains were mounted for SEM analysis with their c-axes parallel to the SEM adhesive stub and imaged. Grains were then rotated in increments of 90° about the c-axis to image all four \{100\} faces. This 360° exterior imaging technique provided a three-dimensional record of the intersection of the planar microstructures on all crystal prism faces. We then used three dimensional digital modeling to identify seven crystallographic orientations of planar microstructures in zircon: \(010\), \(100\), \(112\), \(\overline{1}2\), \(\overline{1}1\), \(\overline{1}\) and \(011\).

Grains were then cast in epoxy and imaged internally with low kV (5 kV) back scatter electron (BSE), cathodoluminescence (CL), and electron backscatter diffraction (EBSD). EBSD mapping reveals microtwins along two \{112\} orientations with 65° of apparent rotation about \(<110>\). The \{112\} microtwins range in apparent thickness from 100 to 500 nm and offset one another by as much as 100 nm. PFs in \(011\) orientations show significant (>5 μm) sinistral offset of original igneous zonation in CL and up to 10° of lattice misorientation in EBSD. Although common on grain exteriors, c-axis parallel PFs \([010\) and \(100]) were rare in polished section. Where present, the \(010\) and \(100\) PFs form low angle grain boundaries with 1 - 10° of misorientation. Imaging crosscutting relationships of the microstructures facilitated understanding the chronology of planar microstructure formation, whereby \(010\) and \(100\) PFs form first, followed by \{112\} PFs and finally \(011\). This 3D external grain and 2D internal microstructure dataset serves as a frame of reference for interpreting ancient shock micro-records.
Measuring Lattice Strain with High Resolution EBSD

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Recent advances in high resolution EBSD techniques have facilitated the extraction of precise measurements of relative lattice distortion (i.e. distortion gradients). This information is crucial for accurate characterization of GND content, for example, but is inadequate for the determination of absolute local strain. In order to resolve absolute strain, reference EBSD patterns that are fixed relative to some global reference frame are required, along with knowledge of the associated pattern center (microscope geometry). The simulated pattern method provides an appropriate library of EBSD patterns in the global frame, and recently published pattern center resolution methods appear promising. Nevertheless, the measurement of absolute strain is a challenge that pushes these techniques to their limits. This paper will review the application of high resolution EBSD methods for assessing absolute strain, and discuss challenges and future opportunities.
High resolution analysis with the aid of the latest SEM and EBSD systems

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Advances in electron optics from FEI-Magellan have resulted in a FEGSEM capable of sub-nanometer resolution at both high and low voltages. In parallel with this development Oxford Instruments have produced a new generation EBSD detector with greatly increased sensitivity together with a new and improved software package.

Research has already shown the advantages of low voltage analysis for improved analytical spatial resolution and our current work is aimed at establishing the optimum performance for our combination of SEM and EBSD system and relating the attainable spatial resolution and angular resolution to accelerating voltage, probe current and sample atomic number effects. Typical applications exploiting the improved performance of the system will also be presented.
Dealing with the limitations of “live” orientation contrast imaging and EBSD analysis on materials deformed by in-situ compression and tensile testing

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The evolution of texture and microstructure during plastic deformation governs the bulk mechanical properties of virtually all materials used to design engineering components and equipment. Plastic deformation has been extensively studied for decades and is a well understood phenomenon. There are details though that could still be refined which will help improving present simulation models. Most publications have focused on comparing the initial with the final state and try to understand the phenomena taking place during plastic deformation. A few studies have tried to observe also the intermediate steps using in-situ plastic deformation experiments.

Although they are a big step forward into refining our understanding of the plastic deformation phenomenon, in-situ experiments are quite difficult to run and observe due to a series of challenges related to the technique. For instance, keeping the area of interest in the field of view is very difficult as the sample and/or stage drift is quite significant. The task is even more challenging when dealing with samples prepared for EBSD analysis which are well known for delivering low contrast in secondary electron (SE) images. The relatively large size of a typical tensile stage combined with the setup geometry needed for EBSD analysis is another factor making the in-situ studies challenging. The user is “forced” to run the experiments at very large working distances (WD), e.g. 30 mm in order to avoid collision with the SEM pole piece or other detectors placed inside the SEM chamber. As most EBSD detectors are designed to work at WDs between 15 mm and 20 mm when scanning samples at much larger WDs a significant part of the EBSD signal will not be used due to the non-optimum phosphor screen positioning.

The current study will present new hardware developments and discuss the experimental setup used for trying to minimize or completely solve the issues related with these challenging factors. Among other features, a special ForeScatter (FSE) imaging system capable of delivering high detail color coded orientation contrast images will be discussed. The capability to place the phosphor screen in an optimum position for acquiring EBSD signal over a large range of WDs will also be described. Qualitative and quantitative results acquired during in-situ plastic deformation of different materials will be shown to demonstrate the advantages brought by these new developments.
Zirconium alloys are used in nuclear reactors owing to their low capture cross-section for thermal neutrons and good mechanical and corrosion properties. However, they do suffer from delayed hydrogen cracking (DHC) due to formation of hydride particles [1]. This study shows how the EBSD techniques can be used to characterise hydrides and their orientation relationship with the matrix.

Hydrided EB weld specimens were prepared by electro-polishing and characterised using Oxford Instruments AZtec-HKL EBSD apparatus and software attached to a FEG SEM. Hydrides were found to exist as fine intra granular plates and having the Blackburn orientation relationship, i.e. (0002)Zr//(111)hydride and [1120}Zr//[1-10]hydride. The hydrides were also found to contain sigma 3 boundaries as well as local misorientations.

References

1. Delayed hydride cracking in zirconium alloys in pressure tube nuclear reactors, IAEA, VIENNA, 2004
Today ion milling is the most common method to prepare samples for Electron Microscopy. The method can be used to modify sample surfaces through ion polishing, cleaning or contrast enhancement. Preparation artifacts can be reduced by using the optimum parameters for milling angle, ion energy and milling time. Surface sensitive methods like EBSD need a perfect sample surface, free of any damage or contamination. Consequently, these methods need a sample preparation that meets this requirement. Many conventional preparation techniques deform the crystal lattice. Even a final ion polishing is often not able to remove the induced damage completely.

To overcome this problem we have used ion beam slope cutting. This is a method to prepare high quality sample surfaces with almost no limitation. Even brittle materials or very difficult material combinations can be easily prepared. The sample surface is partly protected by a sharp edged mask. The unprotected part of the sample will be milled by a high energy Ar ion beam. The result is a flat and polished surface almost free of any preparation artifacts.

Normally ion beam slope cutting is carried out with one ion beam and an oscillated sample. We have developed a unique sample preparation method. Instead of one ion beam and sample oscillation we use three ion beams hitting the sample from 3 different directions (Fig.1). This method provides a lot of advantages: The sample can be observed under perfect conditions while milling. The heat transfer between sample and stage is much better compared to that of an oscillated sample. Re-deposition and shadowing effects can be almost completely suppressed.

We will show the advantages of ion milling over mechanical polishing. Several preparation examples show the surface quality.

FIG. 1: Unique triple ion beam technique for ion beam slope cutting
Analysis of Deformation Behavior of Commercially Pure Titanium and Ti-5Al-2.5Sn (wt.%) Using in-situ Scanning Electron Microscopy and Electron Backscattered Diffraction

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The 296K tension deformation behaviors of a fully-α phase commercially pure (CP) Ti microstructure and a near-α Ti-5Al-2.5Sn (wt.%) microstructure were investigated and compared. In-situ tensile experiments were performed inside a scanning electron microscope. Electron backscattered diffraction was performed both before and after the deformation. Slip trace analysis was used to determine the active slip systems and the associated global stress state Schmid factors. Prismatic slip was the most frequently observed deformation mode in both microstructures. However, the basal slip activity in Ti-5Al-2.5Sn was significantly greater than that observed in CP Ti. Deformation twinning systems were determined by comparing the twin orientations with their parent orientations as each type of twin has a unique misorientation angle and rotation axis with respect to the parent orientation. Although twinning was an active deformation mode in CP Ti at 296K, it was almost completely suppressed in Ti-5Al-2.5Sn(wt.%) due to the presence of Al. In-situ elevated-temperature tensile and tensile-creep experiments were also performed, and will be compared for each material.
Factors Controlling the Crystal Morphology and Orientation of Carbonate Minerals Precipitated in the Pipe of hot spring, Taiwan

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The carbonate sinters of this study are aragonite and calcite which collected from the Ho-Ya and Dung-Mei hot spring hotels, located in Rui-Shui of Hualien County and Chi-Pen of Tai-Tung County, Taiwan, respectively. The former is predominantly composed of aragonite (>99.4%), while the latter is calcite (>94%) by Electron Backscatter Diffraction (EBSD). Meantime, crystal morphologies and orientations of sinter are also collected to discuss what kinds of factors are dominated and to reconstruct the pumping history of hot spring. Two particle morphologies of aragonite in the Ho-Ya SPA sinter can be identified. One is small and rounded crystals gathered as the strips, while the other is prism. However, crystal form of calcite in the Dung-Mei Hot Spring sinter is characterized by mosaic granulated grains. The peaks of misorientation angle in aragonite of the former are <15°, 50°-60°, and 60°-70°, while in calcite of the latter is usually less than 20°. The porosity of aragonite range from 0% to 25%, but for calcite is very low to zero.

Holcomb et al., (2009) simulated the coral’s growth and concluded that the small and rounded crystal which called centers of calcification (COC) is owing to high pH and saturation state in the solution. We, therefore, propose that the higher pumping rate causes the rapid depressurization of CO₂ to over-saturate bicarbonate concentrations quickly, and then crystals nucleate and grow. This process may generate higher porosity and rounded shape of aragonite. In low season with low pumping rate in the Ho-Ya hotel, the aragonite grows the fine and elongate crystals inside the pipe due to slow supply of ions from hot spring. However, in peak season with high pumping rate it causes the quick degassing of CO₂ to over-saturate ions in solutions, then precipitate aragonite rapidly and generate prismatic crystals with larger porosity. The crystal morphology, therefore, of the colorful rings implies that the sinter of Ho-Ya hot spring grows at the summer-winter-summer cycle during one and a half years.

Keyword: hot spring, sinter, aragonite, calcite, crystal morphology,
Determination of the stress and strain states at dislocation channel-grain boundary intersections in irradiated stainless steels.

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The objective of this project is to quantify the stress and strain states that develop due to localized deformation in irradiated austenitic stainless steels. Irradiation causes several changes in the microstructure of stainless steel, such as the segregation of elements, increased hardness, constraint of deformation into coarse channels, referred to as dislocation channels, and an increased susceptibility to intergranular stress corrosion cracking. The complex irradiation effects make it difficult to determine the exact source of increased susceptibility to cracking, however, there appears to exist a connection between it and localized deformation.

The dislocation channels cause strain to be highly localized and heterogeneous. If the strain of the dislocation channel is unaccommodated at the grain boundary, it can cause areas of high normal and shear stresses. While there is a strong connection between localized deformation and the increased susceptibility to cracking, an exact mechanism is still unknown. It is known that dislocation channeling concentrates strain so that there exist localized areas of high stress and strain. Little is known, however, on a quantitative level of the stress and strain states induced by these channels. This is due to the difficulties of experimentally measuring local strain states and of simulating the straining of a sample with complex irradiation effects in the microstructure. For the exact mechanism that connects localized deformation and cracking to be understood, it is important to be able to experimentally quantify the stress and strain states in the material. This project is in its early stages, but will use high resolution electron backscatter diffraction (EBSD) analysis, combined with digital image correlation (DIC) and atomic force microscopy (AFM) to quantify plastic strain through DIC and AFM, as well as elastic strain and stress through EBSD analysis.
High Throughput Characterization of Structure across Composition Spread Pd-Alloy Libraries by EBSD

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Pd alloys can be used for separation of H₂ from mixed gas streams with nearly perfect selectivity. Performance of the alloy in the separation application is a complex function of surface composition, bulk composition and bulk structure. However, optimization and understanding of membrane alloys is complicated by the large number of components—and their relative amounts—that are of potential interest.

To address this challenge, we have developed composition spread alloy films (CSAFs) as libraries of model separation membrane alloys. CSAFs are thin (~ 100 nm in this work) films with continuously varying lateral compositions that are deposited onto compact (~1 cm² in this work) substrates. When characterized for composition, structure, and functional properties with appropriate spatial resolution, CSAFs enable rapid construction of composition-structure-property relationships.

In this work we demonstrate preparation and structural characterization of CuₓPd₁₋ₓ and CuₓPdᵧAu₁₋ₓ₋ᵧ composition spreads deposited onto Mo substrates. We annealed a CuₓPd₁₋ₓ CSAF at 800 K and characterized its local structure by EBSD. The phase behavior that we observe across the CSAF matches that of the CuPd bulk phase diagram at 800 K: FCC above x = 0.68 and below x = 0.51; BCC between x = 0.56 and x = 0.65; and mixed FCC-BCC phases elsewhere. We also characterized phase behavior of a CuₓPdᵧAu₁₋ₓ₋ᵧ CSAF annealed at 700 K. Along the CuPd binary (Au = 0) BCC order is observed at the expected compositions. By 20% Au content, however, the BCC phase is no longer present. High Cu and high Au ternary compositions generally display FCC order; no order is detectable at high Pd ternary compositions.

Results of spatially-resolved characterization of the surface activities of the CuₓPd₁₋ₓ and CuₓPdᵧAu₁₋ₓ₋ᵧ libraries for H₂ dissociation—a key functional property of H₂ separation membranes—will be presented and interpreted from the perspective of the libraries’ structure.
EBSD analysis in the geosciences: high temperature mylonites

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We use microstructural and electron backscatter diffraction (EBSD) analyses to evaluate the deformation conditions under which the Earth’s oceanic crust and continental crust are deformed in high-temperature shear zones associated with extensional fault systems. We examine both high-temperature (670-750°C) gabbro mylonites from lower oceanic crust exposed in the footwall of the Atlantis Bank oceanic core complex fault system, and high-temperature (500-500°C) granodiorite mylonites from continental crust exposed in the footwall of the South Mountains, Arizona, continental core complex fault system. In both fault systems, the polyphase crustal rocks are heterogeneously deformed; strain is partitioned into rheologically weaker minerals. In these heterogeneously-deformed crustal rocks, we use microstructures to interpret the relative strengths of the constituent minerals. For those minerals that exhibit microstructural evidence of crystal plasticity, we use EBSD analyses to 1) identify the locations and shapes of grain and subgrain boundaries for each constituent mineral, 2) measure the crystallographic orientation of individual grains and subgrains within a constituent mineral, and 3) measure lattice preferred orientation and use it to interpret the deformation mechanism(s) by which that mineral deforms during flow.

In the Atlantis Bank oceanic core complex, microstructural analyses demonstrate that strain localization associated with oceanic detachment faulting is dominated by crystal plastic deformation of plagioclase. EBSD analyses reveal that strain localization is achieved in part by dynamic recrystallization of plagioclase, resulting in the transition from dislocation to diffusion creep deformation mechanisms. In contrast, microstructural analysis of the South Mountains granodiorite mylonites shows that the crystal plastic deformation of quartz dominates the strain localization process during mid-crustal shearing. EBSD analyses reveal that dynamically recrystallized quartz grains exhibit lattice preferred orientation (LPO) indicative of basal \(<a>\) and rhomb \(<c>\) slip. Based on the microstructures and the interpreted quartz slip systems, the mylonitic fabric was developed at temperatures ~500-550°C. Together, the microstructural and EBSD data suggest that dislocation creep is the operative deformation mechanism in the interconnected network of abundant quartz.
Lattice preferred orientation of quartz from an Appalachian shear zone: testing numerical models of tectonic strain with optical and EBSD measurements

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Quartz (SiO2) undergoes ductile creep under tectonic rates of strain, and numerical modeling based on micromechanics predicts specific lattice preferred orientation patterns depending on the relative activity of various slip systems in quartz and the flow field. Combining characterization of quartz lattice fabrics from natural tectonites with numerical modeling has the potential to constrain the ancient flow field and deformation boundary conditions, which in turn helps to unravel the tectonic evolution of an area. Conventional optical crystal orientation measurements of quartz are made with a petrographic microscope and U-stage whereas EBSD stage mapping offers the ability to collect and analyse quartz data sets that are orders of magnitude greater. Here we present our first EBSD stage mapping results of an oriented thin section from the Roper Lake shear zone in northeastern Cape Breton Island in the Canadian Appalachians (Lin et al., 1998). The shear zone is developed along the southeastern contact of the Middle Devonian Black Brook granite (U-Pb monazite age 375±5 Ma, Dunning, et al., 1990). The shear zone formed in Late Devonian to Early Carboniferous due to oblique boundary convergence. Shape fabrics in the forms of foliations and lineations are well developed in the shear zone and the strain geometry suggests a triclinic flow field (Lin et al. 1998; Jiang and Williams, 1998). We made EBSD measurements in stage mapping mode over a 14 mm by 4.2 mm area of a vibratory polished petrographic thin section using a step size of 20 μm. Acquisition time per point was 5 ms, using 8x8 binning and high gain. Four to five bands were used for indexing and no noise reduction was used on the final data set. Over the entire area, there was a 63.9% successful indexing rate, with quartz having a mean Band Contrast (BC) of 77.04, and mean Mean Angular Deviation (MAD) of 0.8587, albite having a mean BC of 76.11 and mean MAD of 0.9373, and orthoclase having a mean BC of 75.65 and mean MAD of 0.945. We will present a comparison of numerical modeling, optical crystal orientation data and EBSD data for quartz domains in this high temperature ductile strain fabric at cm to micron scales to better elucidate material behavior during triclinic flow, and the associated tectonic boundary conditions during plate collision in this period of the evolution of the Appalachian orogeny.

References


Fast Orientation Projection using OpenGL

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Pole figures are often chosen for the presentation of orientation data, where each orientation is often represented by numerous points due to symmetry. In case of a cubic phase, due to their multiplicity, at least 3 {001}, 4 {111}, and 6 {011} must be drawn. For an orientation map consisting of 800x600 single orientation measurements about 7.5 million dots must be calculated and projected. Using standard computers, even for such a small map, a real-time rotation of the resulting big number of data points seems to be impossible, especially if one considers that for any rotation the complete calculations must be repeated. Moreover, a requested change from a stereographic to an equal-area projection needs an additional calculation of the projected points.

The presentation will demonstrate that there is no need for an extra-calculation of the stereographic projection. It will be shown how gnomonic, stereographic, equal area, equidistant and spherical projections are related to each other and why they can be generated in a few Milliseconds, independently of how many data points or which kind of radial distribution must be presented.
Thermoelectric materials convert thermal energy into electrical energy in a power generation role, and convert electric energy into thermal energy in a heat management application. These materials can be used to recover useful work from waste heat and provide a solid-state alternative for refrigeration. However in order for continued commercial development and deployment of these materials, the conversion efficiency of these materials needs to improve. Thermoelectric materials require high electrical conductivity and low thermal conductivity. One approach to obtain improvement is reducing the grain size to increase the scattering of phonons and reduce thermal conductivity. Another approach is to take advantage of the inherent anisotropy of thermoelectric materials by producing a favorable texture. In either case, EBSD is a well suited tool for characterization of this class of materials. In this work, a nanostructured Bi$_2$Te$_3$ powder precursor was produced using a gas atomization process. EBSD was then used to characterize the microstructure of the power particles and verify the nanoscale grain structure. The powders were then consolidated into bulk thermoelectric material using both hot pressing and explosive compaction. The microstructures produced with both fabrication methods are then compared, and the nanostructure verified to be retained. The information provided by both the average grain size and the grain size distribution will be discussed.

Figure 1: Orientation map of compacted Bi2Te3
Using EBSD to aid understanding of irradiation creep in CVD-3C SiC

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Experiments conducted in the High Flux Isotope Reactor (HFIR) indicated that CVD-3C SiC received from different vendors (V), V1 and V2, showed measurably different irradiation creep responses. This work examined pre-irradiated material to help understand the creep behavior. EBSD analysis of V1 SiC showed columnar grains in the growth direction and dense "checkerboard" artifacts, identified as mostly Σ3 boundaries (Figure). V2 SiC exhibited mixed columnar and equiaxed grains, no checkerboarding, and discrete Σ3 boundaries. Samples were ion polished (Gatan Ilion+) mapped at 10 kV and 2~3 nA, with 250 nm steps, 25~30 Hz, with an EDAX Hikari camera binned to 106×80 pixels. Experiments at 20 kV showed the checkerboard locations to be repeatable from one scan to the next, implying systematic rather than random results. HR-TEM analysis showed the V1 SiC to have a higher stacking fault density, which may cause the checkerboarding. The columnar and defect-dense structures likely cause the different SiC creep properties.

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In multiphase materials, orientation determination by EBSD presupposes a correct phase assignment in addition to correct identification of the diffracting lattice planes. The existence of phase-specific interplanar angles make this possible in materials containing multiple phases with low symmetry; however, in materials with multiple cubic phases, phase assignment is only possible when phase-specific minor bands are visible because the major reflectors are all identical. In difficult cases, the band width can be applied as additional source of information, but this automatically results in comparatively slow measurement speeds and requires well-visible band edges -- i.e. the material can only be deformed minimally, if at all. For isostructural phases like Cu and Ni, or for the numerous spinel-like phases (e.g. Fe₃O₄, FeCr₂O₄, FeCr₂S₄), even using band widths will be insufficient for differentiating between the phases since the lattice parameters are very close to one another. The use of band width also often fails for superstructures like ZnS and CuFeS₂. The relatively recent introduction of simultaneously acquired and analyzed chemical composition by EDS provides significant additional phase assignment capability, but the quality of the phase assignment is limited by the relatively high required speeds of EBSD data collection. Furthermore, the comparatively big difference in the interaction volumes between the two techniques limits the resolution to particle sizes larger than ~1 µm. Moreover, for certain phases like FeO and Fe₃O₄, the phases cannot generally be differentiated by EDS alone. The segmentation of γ from γ' in Ni-based superalloys is extremely challenging since the crystal structures are described by essentially the same atomic positions (with γ' having an ordered superlattice) and in the case of tertiary and secondary γ' particles, the precipitates often have a mean size under 500nm. In the present work, a simple approach will be presented which immediately solves many of the aforementioned problems and offers additional applications. It is well-known that the backscattered electron (BSE) signal in SEM is sensitive to atomic number and is therefore useful for imaging of different phases; cf. mineral liberation analysis. Acquisition of the BSE signal using detectors mounted above the EBSD camera screen allows for the collection of additional information that can be used for segmentation of the phases in EBSD scans. The simultaneous acquisition of EBSD patterns, EDS spectra, the BSE signal, and the forward scattered electron signal (which shows greater sensitivity to crystal orientation than may be quantified by automated EBSD) would provide much new potential for advancing multiphase material characterization using EBSD.
Effect of Hot Compression Conditions on Microstructure and Texture of TX32 and TXA321 Magnesium Alloys

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Wrought magnesium alloys are of considerable interest for structural components due to their light weight. Good ductility, corrosion and creep are the properties considered while employing magnesium wrought alloys. Newer Mg-Sn-Ca (TX series) alloys are being developed to meet these requirements. The current paper reports the effect of test conditions on the hot deformation of TX32 and TXA321 magnesium alloys by studying the microstructure and texture of compressed specimens. Compression tests were carried out at various deformation conditions of temperature (250-500 °C) and strain rate (0.0003-10 s⁻¹). The occurrence of dynamic recrystallization (DRX) was confirmed from the microstructure observations, and the most suitable conditions for processing were identified as domains in the processing maps of these alloys. The resulted texture characteristics of the deformed alloys were analyzed with electron backscattered diffraction (EBSD) technique. Schmid factor analysis was used to find the activation of different slip systems, and the contribution of pyramidal (non-basal) slip system is found to be significant for deformation at higher temperatures.
Deformation characterization of Zircaloy-4 sheet deformed by plane strain compression

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To study the deformation mode activation in Zircaloy-4 material in plane strain compression condition the method of calculating the in-grain misorientation axes (IGMA) \cite{1} was applied. In order to evaluate the dependence of activated slip and twin systems on the strain level cold rolling of Zircaloy-4 sheets was performed at room temperature. To achieve different strain levels and to cover all possible grain orientations in the specimens the sheets were cold rolled to thickness reductions between 5\% and 80\% in three different rolling direction: first along, second transverse to the original cold rolling direction, third type of sheets were cross rolled (rolling direction was changed alternately from RD to TD).

The aim of the work was i) to study the limits of the method as it is applied to different strain levels on a fine-grained microstructure (mean grain size \(\sim\) 4\(\mu\)m); ii) to study the grain orientation dependence on slip and twin system activation.

It was shown that one can clearly separate between activation of prism \(<a> slip and systems with \(<c+a> slip using IGMA distributions. In addition it is possible to limit the number of possible activated \(<c+a> slip systems in particular grains depending on their orientation using Schmid factor distribution analysis.

Polycrystalline materials undergoing ductile dynamic deformation are known to accommodate strain through large deformation plasticity, adiabatic shear banding and void formation. This work will focus on the latter to study the process of void nucleation, growth and coalescence. Seaman and Curran studied nucleation and growth (NAG) extensively in the early 70’s and developed NAG theories, which can be improved on with the use of EBSD. One dimensional plate impact experiments were conducted in order to capture incipient “spall” in the samples. Spall occurs when two decompression waves under uniaxial strain conditions interact and produce a region of tension in the interior of the material, which if of sufficient strength causes material failure. In the absence of precipitates, grain boundaries are preferred sites for void nucleation; therefore, experiments were conducted on four grain sizes of well characterized commercially pure 1050 aluminum in order to alter the defect density. Input stress and duration were held constant in order to maintain the total volume loaded in dynamic tension constant and solely study the influence of defect distribution on the dynamic deformation process. Post mortem analysis of soft recovered samples was conducted via EBSD to correlate mesoscale features of damage to the microstructure. Results indicate that void nucleation occurs predominately at high angle grain boundaries. A critical length scale is observed where a nucleation dominated regime is transitioned to a growth dominated regime. Observed damage follows grain size dependence with increasing damage seen as grain size is increased, except in the largest grain size where intragranular void nucleation occurs and a drastic reduction in damage is observed. This phenomenon will be discussed thoroughly as well as the influence of Taylor factor on the growth and coalescence of voids.
Recent technological progress in dynamical observations of individual functional single protein molecules in living cell has been achieved with several single molecular techniques and systems. In order to improve monitoring precisions and stability of the signal intensity from single molecular units under physiological conditions, we have proposed that single molecular techniques using shorten wavelength, for example, X-rays, electrons, neutron, and other accelerated ion probes. In this work, we demonstrate three-dimensional (3D) tracking of single nanocrystals using Scanning Electron Microscope. We called Diffracted Electron Tracking (DET).

Diffracted X-Ray tracking (DXT) [1-5] has been developed for obtaining the information about the dynamics of single molecules. This method can observe the rotating motion of an individual nanocrystal, which is linked to specific sites in single protein molecules, using a time-resolved Laue diffraction technique. This method needs a very strong X-ray source, such as the SPring-8, so we began to develop a compact instrument for monitoring the rotation of the single protein molecules, using the electron beam instead of the X-ray.

Instead of the Laue diffraction using white X-ray, the Electron Back-Scattered Diffraction Pattern (EBSP) is adopted to monitor the crystal orientation of the nano-crystals linked to the single protein molecules. For this purpose, it is necessary to realize (1) wet cell with very thin sealing film, (2) EBSP system with high sensitivity, (3) damage-less electron irradiation technique. Using DET, we measured motions of gold nanocrystal that labeled on silane-coupling-agent under different conditions; in water, in argon gas and vacuum environments. From results, the motions of gold nanocrystals in water environment were over one hundred times as large as in argon gas environment. Additionally, we succeeded in observing movement of the commercial colloidal gold linked to the functional protein molecule. DET technique may develop as a very general single-molecule methodology.

References
New approach to characterizing multi-phase materials by advanced EBSD/EDS integration

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Recent software and hardware developments have greatly increased the speed at which simultaneous Electron BackScatter Diffraction (EBSD) and Energy Dispersive X-Ray Spectroscopy (EDS) mapping can be done. New developments have transformed the combination of these techniques into a powerful tool for characterizing multiphase materials with improved efficiency and data quality, introducing new ways of using the two complementary techniques to ensure data integrity.

It is well known that the information delivered either by EBSD or by EDS alone is not always enough to successfully distinguish the different phases present, e.g. phases creating similar patterns or having similar chemical composition respectively. The new approach consists of simultaneously acquiring an EBSP and a complete EDS spectrum for each point in the map at speeds of up to 500 points/sec. An automatic procedure uses the quantified EDS results in each spectrum to discriminate phases creating similar patterns and immediately after the corresponding EBSP is used for finding the crystal orientation. This automatic feature can be used online as well as offline and is capable of analyzing up to 1300 points/sec. When one or more unknown/unexpected phases are present the simultaneously acquired data can be used for offline phase identification and subsequent ultra fast re-indexing for completing the map within minutes.
In-Situ Characterization of slip transfer across α/β interfaces in equiaxed Ti-5Al-2.5Sn (wt. %) using EBSD and Microcantilever Beams

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Prior research of slip transfer across the α/β interface in Ti-5Al-2.5Sn has demonstrated that the traditional Burgers orientation relationship (OR) is not frequently observed when it has an equiaxed α microstructure with globular grain boundary β. Slip trace analysis of microstructural patches in four-point bend specimens showed that α to β slip transfer is not strongly influenced by the lack of an α/β OR. Still, it is unclear what effect random α/β ORs play in strengthening or weakening grain boundaries during deformation. EBSD provides information about the local crystal orientation of adjacent α and β phases. Microcantilever beams have been created across α/β boundaries to study the effect that different α/β ORs have on slip transfer. This effect is assessed by comparing load-displacement curves from nanoindentation tests across multiple beams with different α/β orientations coupled with observations of slip traces. This research was supported by DOE/BES grant DE-FG02-09ER46637.
An Analysis of Fatigue Crack Initiation Using 2d Orientation Mapping and Full-Field Simulation of Elastic Stress Response

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Nickel-based superalloys are important engineering materials, particularly in the demanding applications such as turbines for aerospace and, increasingly, the ground-based energy production. The continuing efficiency increases demanded by these applications requires operation at higher temperatures and expectations of longer fatigue lifetimes. Fatigue life depends not only on crack growth rates but also crack initiation, therefore lifing studies require a better understanding of the nucleation of microstructurally small fatigue cracks. The approach of the present work is to characterize nickel-base superalloy specimens. Initial work is limited to surface analysis via EBSD mapping of microcrack regions, and simulation of the material stress state, achieved using a fast Fourier transform (FFT) technique providing a full-field model of the elastic stress response. Although analysis of an already-acquired three-dimensional reconstruction is ongoing, preliminary results indicate that under these test conditions cracking initiates along coherent twin boundaries, and specifically coherent twins that are near the upper tail of the length distribution. And although the simulated elastic stress response points to high relative resolved shear stress along the crack boundaries, it appears more likely that a combination of boundary length and orientation are responsible for the susceptibility to crack nucleation.
Dependence of creep fracture on microtexture of polycrystalline Ni-based cast super-alloys

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The aim of this research is to study the relationship between crack initiation and the orientation of the grains at the crack initiation site during creep deformation of polycrystalline Ni-based cast super-alloy. It is planned to test polycrystalline cast Ni-based super alloys specimens that have been subjected to an interrupted creep tests. Determination of the grain boundary misorientations at the crack initiation sites and along the crack propagation path will be carried out using Electron Back Scattered Diffraction (EBSD). The misorientation distribution function (MDF) of the grain boundaries will be used to quantify the effect of local texture on crack initiation and propagation.

We have made EBSD measurements on a Ni-based cast super-alloy specimen in order to determine the experimental conditions and sample preparation techniques necessary for the interrupted creep test specimens and also to identify any material specific implications for better measurements. Ni-based super-alloys consist of two major phases: an f.c.c matrix (\(\gamma\)) predominantly containing nickel along with numerous alloying elements like Cr, Co, Al, Ti, Ta, Mo and an L1₂ precipitate (\(\gamma'\)) which is an ordered f.c.c structure, based on Ni₃Al, with Al atoms on cube corners and Ni atoms on face centered sites. C is intentionally added to form metal carbides with the alloying elements that have a higher affinity for C than Ni. The metal carbides and \(\gamma'\) precipitates improve the strength of the material considerably. It was observed from the optical images that the \(\gamma'\) precipitates have preferentially segregated at inter-dendritic regions (grain boundaries) and the core of the grains contained few or no precipitates, while the metal carbide particles were dispersed throughout the matrix. EBSD measurements show the presence of both large and small angle grain boundaries.
Development and application of a novel characterization system to quantify grain structures in 3D

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This talk describes the development and application of a first-of-its-kind automated serial sectioning system to characterize grain ensembles in structural engineering alloys such as polycrystalline Ni-base superalloys that have an average grain size exceeding ten micrometers. The characterization system is comprised of three primary sub-systems: a Tescan Vega SEM outfitted with a custom motorized load-lock, Bruker energy dispersive spectroscopy (EDS) and electron backscatter diffraction (EBSD), a RoboMet.3D robotic serial sectioning system for multi-step mechanical polishing, and a 6-axis Mitsubishi RD12SVL robot with custom end-of-arm-tooling to transfer samples between the other two sub-systems. Custom computer control scripts have been developed in Python and C++ to automate the entire data acquisition process. For characterization of polycrystalline Ni superalloy microstructures, the collection of both EBSD and backscattered electron image data enables computer-based segmentation procedures to automatically extract grains and porosity from the 3D data stack. A 3D reconstruction of a polycrystalline superalloy (LSHR) grain structure from an initial experiment is shown in Figure 1. Data registration and segmentation was performed using the DREAM.3D software package (http://dream3d.bluequartz.net/). Future data sets are anticipated to exceed volumes of 0.1 mm\textsuperscript{3} and contain over 10,000 grains.

Figure 1. Reconstruction of an initial data set (LSHR superalloy) from the multi-modal 3D characterization system. Volume is roughly 675 x 475 x 11 micrometers in size.
EBSD applications to predict nuclear fuel performance

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Development and testing of high performance fuel cladding has been identified as a high priority to support enhancement of fuel performance, reliability, and reactor safety. One of the primary technologies being developed under the Light Water Reactor Sustainability (LWRS) Program Advanced LWR Nuclear Fuel Development (ALFD) Pathway, is an advanced fuel cladding made from ceramic matrix composites (CMC) utilizing silicon carbide (SiC) as a structural material supplementing an internal Zircaloy-4 (Zr-4) tube. The ultimate objective is to utilize the SiC-CMC hybrid tube to improve safety and economics at nuclear power plants. It is expected that the SiC-CMC protective coating on the standard Zircaloy-4 cladding tube will enhance fretting resistance and decrease hydrogen uptake. The low chemical reactivity of SiC also is expected to eliminate the exothermic reaction between zirconium and water. This will prevent the generation of free hydrogen, leave the cladding intact at higher temperatures and reduce energy generated in a reactor accident. Electron backscattered diffraction (EBSD) has been reported previously as a technique to study the microstructure and crystallographic orientation of hydrides in Zircaloy-4 LWR fuel cladding. Furthermore, EBSD is a technique that is currently receiving much attention in studies exploring grain boundary-related fission product transport mechanisms. As this SiC-CMC Zircaloy 4 hybrid tube development is “first of the kind” and is testing a novel idea, not all the testing requirements are quantified fully yet. However, it is decided that in all cases, test results will be compared with those of a standard Zircaloy-4 tube as the status quo. The requirement for this LWRS-1 test is thus in general to compare favorably with the standard Zircaloy-4 fuel tube. Thus for this preparatory work, EBSD measurements are done on the SiC-CMC sleeve and Zircaloy-4 tube to form a baseline for studies and an understanding of hydride formation. It is the aim of this presentation to present preliminary characterization results using EBSD as a performance predictor for SiC, SiC-CMC and Zircaloy-4 as material of choice in nuclear fuel cladding. Sample preparation technique challenges will also be shortly discussed.
Some Advances in and Applications of Cross-Correlation Based Analysis of EBSD Patterns

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Over the last ~5 years we have been steadily improving the mapping of elastic strain variations and small lattice rotations from EBSD patterns [1-3]. The required high sensitivity (~10^{-4} rads) is achieved by measuring small shifts in the positions of features in EBSD patterns. This presentation will review some of these improvements including the significant advance achieved by adopting a pattern remapping algorithm [4].

The original method [1] uses a single pass of cross-correlation analysis which works well if lattice rotations are small and of similar magnitude as elastic strains (ie in semiconductor structures). When small elastic strain variations are accompanied larger lattice rotations, as is the case in plastically deformed metals, then although the rotations are well determined the strains are generally not. However, the initial cross-correlation analysis can be used to determine a finite rotation that allows the test pattern intensities to be remapped to generate a new test pattern for a crystal more closely aligned to the reference case. A second pass of cross-correlation analysis between the reference pattern and the remapped test pattern is then used to determine the elastic strain variation, and a correction to the lattice rotation. This approach has been tested using virtual EBSD patterns simulated using the dynamical diffraction theory of Winkelmann [5]. These simulations are invaluable for evaluating new methods as the strains and rotations are known a priori.

The improvements generated by the new algorithm will be illustrated by comparing maps obtained from various plastically deformed metallic samples with and without the second pass of cross-correlation on the remapped patterns.

Characterizing Artifacts in Local Misorientation Measurements at Grain Boundaries

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The measurement of spatially specific orientation by automated electron backscatter diffraction (EBSD) is now a well-established technique. When such measurements are made on deformed materials, local variations in orientation are often observed. These local variations provide evidence on the dislocation density in the deformed material. One method used to quantify the magnitude of these local misorientations is the kernel average misorientation (KAM). The KAM measures the average deviation in orientation from every pixel in a prescribed kernel (typically a few nearest neighbors) with respect to the pixel at the center of the kernel. Several EBSD based studies on deformed materials have shown that KAM values frequently increase in the vicinity of grain boundaries. This phenomenon would be expected due to dislocation pile-up at grain boundaries; however, some increase in the KAM may also be due to a loss of orientation precision in the EBSD measurements near grain boundaries. When the electron beam is positioned near a grain boundary, the resultant EBSD pattern will contain contributions from both lattices separated by the boundary. This study quantitatively characterizes the ability of the computer software to effectively deconvolute these mixed patterns and precisely determine the orientation of one of the contributing crystals. This work includes studies of high resolution experimental data as well as results from simulated mixing of patterns. Some of the experimental work has been performed on copper with a high density of twins to ascertain whether the inclination of the grain boundary plane plays any perceptible role. The results clearly show an effect of the pattern mixing on the orientation precision in the immediate vicinity of grain boundaries. However, the contribution is relatively small and is likely only to affect KAM measurements on lightly deformed materials.
EBSD study of dynamic recrystallization mechanisms during hot deformation in Nimonic 80A

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Microstructure evolution and dynamic recrystallization (DRX) mechanism of Nimonic 80A deformed at the temperature range of 800°C-1180°C and strain rate of 1s⁻¹ via compression tests were studied with the help of EBSD. The stress-strain curves show typical hot deformation behavior of restoration as peak stress appears followed by steady state stress. It indicates that DRX occurred during hot working as it reached critical strain. Fine grain sizes are obtained at lower temperatures of 800°C-950°C (ε at 1.1). However, the grain-structures are not fully homogeneous, which can be explained by the flow instability. An increase of temperature to 1000°C resulted at homogeneous microstructure with grain size of 4µm. This may be due to a change from continuous dynamic recrystallization (CDRX) to discontinuous dynamic recrystallization (DDRX).

Samples deformed at temperatures of 800°C-1160°C are characterized by EBSD. Inverse pole figures (IPF) (T≥1000°C) show that nucleation occurs at IGBs with boundary bulging and nuclei grow into small grains along IGBs, forming necklace structure. As strain increases, IGs are finally consumed by equiaxed DRX grains with low density of dislocation (ρ). Also, the fraction of twin boundary (TB%) at 1000°C and 1100°C was found to decrease first and then increase to a steady value with increasing strain, which is associated with the nucleation and grain boundary migration in DDRX. However, IPFs at 800°C and 950°C show recrystallized grains within initial grains, indicating nucleation without boundary bulging.

Internal statistic component (ISC) is used to identify DRX grains from deformed grains. It is found that in DRX grains at 950°C, 1000°C, 1120°C and 1160°C, the fractions of low angle boundary (LAB%) are 4.5%, 3.6%, 1.6% and 1.4%; TB% are 42.5%, 53.4%, 59.3% and 57.8%. LAB% suggests the density of dislocations. Values at 950°C show an increment by 65% in LAB% and a reduction by 26% in TB%, compared with values at 1120°C and 1160°C. DRX grains form by the growth of nuclei with the GB migration in DDRX, while they form by the rotation of subgrains turning LABs into high angle boundaries (HABs) in CDRX. Since less GB migration results in higher ρ and less annealing twins, these results indicate that CDRX is the main mechanism at T≤950°C; and DDRX at T≥1000°C. A transition in DRX mechanism from DDRX to CDRX was found in the temperature range of 950°C to 1000°C.
A Novel Nano-scale Non-contact Temperature Measurement Technique
Based on Electron Backscatter Diffraction

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Detecting nano-scale temperature and temperature distributions is important for studies of heat generation and transfer in a wide range of engineering systems, such as microelectronic, optoelectronic and micromechanical systems. While in the past few decades, much progress has been made in the area of nano-scale temperature mapping techniques, no current method adequately combines high spatial resolution, high temperature sensitivity, and an ability to work in non-contact mode (such that the local temperature distribution is not perturbed). In this presentation, we introduce a new nano-scale resolution non-contact temperature measurement technique, thermal scanning electron microscopy, ThSEM. This technique is based on temperature dependent thermal diffuse scattering in electron backscatter diffraction (EBSD) in a scanning electron microscope (SEM). Unlike conventional scanning thermal microscopy, which uses contact probes, ThSEM is a non-contact method. In contrast to optical temperature mapping techniques, ThSEM doesn’t have the spatial resolution limitation that arises from the optical wavelength and theoretically can reach a resolution of < 10 nm. The hardware setup is very similar to the EBSD system in an SEM, which makes the integration of temperature mapping into SEM relatively straightforward. Moreover, multiple signals or contrast mechanisms, such as temperature distributions, grain orientation maps, topographic images, and elemental maps could be obtained from the same sample area depending on the specific SEM capability. This technique thus adds a new channel – the temperature signal – to the collection of existing SEM signals.

Figure: Summary of nano-scale temperature measurement by EBSD analysis. Top left – experimental geometry. Top right – part of typical EBSD pattern from Si(001). Bottom left – intensity scans across (400) Si Kikuchi line at different temperatures. Bottom right – calibration of normalized peak intensity vs. temperature
In situ study of the tensile deformation and fracture modes in peak-aged Mg-11Y-5Gd-2Zn-0.5Zr alloy

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ABSTRACT

Deformation and fracture modes in peak-aged cast Mg-11Y-5Gd-2Zn-0.5Zr (wt.%) (WGZ1152) alloy strips during tensile deformation at temperatures between 298 and 623 K (0.33-0.69 Tm) have been studied in situ inside a scanning electron microscope using EBSD and slip trace analysis. Electron backscatter diffraction data collected before loading on a microstructural patch located in the center of the gage section was used to identify the orientations of the grains for slip trace analysis. At 298 K, the sample fractured at 291 MPa with 6% elongation. Near the fracture surface, basal <a> slip were dominant, and the specimen fractured by both intergranular cracking (60%) and transgranular cracking (40%). In contrast, at 523K, the sample fractured at 265 MPa with 9% elongation and 62.5% of the 40 slip traces analyzed near the fracture surface were basal <a> slip, 22.5% were prismatic <a> slip, and 15% were pyramidal <c+a> slip. At 473K, 75% of the 20 traces analyzed near the fractured surface corresponded to basal <a> slip, 15% to prismatic <a> slip, and 10% to pyramidal <c+a> slip. No obvious transgranular cracking was observed above 473K. In samples deformed above 523K, only basal slip was present at low (3%) strains and evidence of cross slip and grain boundary sliding was present at high strains. Crack nucleation sites tended to be located at large intermetallic grain boundary X phase at all temperatures and at coarsened slip bands at low temperatures.
Investigation of the microstructure in the vicinity of a fatigue crack in in-depth direction in a ferritic-martensitic steel

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The influence of the martensitic phase on the crack growth of a fatigue crack in a ferritic-martensitic steel was analyzed using Electron Backscatter Diffraction (EBSD) analysis with the purpose of understanding the interaction between the microstructure and physically small cracks.

Fatigue tests were conducted with small dogbone-shaped specimen to initiate fatigue cracks on micro-notches. Afterwards tiny specimens were cut out by spark erosion to investigate the crack growth and the crack front in a synchrotron beam line (SPring-8 in Japan). Following synchrotron tomography the crack was polished from the surface in defined steps with a subsequent EBSD-analysis. Altogether the microstructure in in-depth direction can be characterized by 28 EBSD-Maps with an interval of about 9 to 14 µm each.

Since ferrite and martensite have a very similar crystallographic structure it is very difficult to distinguish one phase from the other via EBSD. Furthermore it is almost impossible to discern martensite and plastic deformation at the crack tip possible based solely the results of the EBSD as they both contribute to reducing the IQ-values. Instead, images taken with a confocal laser scanning microscope after each polishing step are used to define the martensitic areas which have been less polished than the ferritic phase. By putting the inverse pole figure maps on top of the pictures from the confocal laser scanning microscope the martensitic areas can be highlighted in the EBSD map and therefore distinguished from the plastically deformed ferrite. Using this procedure a relation can be put up between local plastic deformation and damage accumulation around the crack tip.
Sn crystal orientation and microstructure evolution during thermal cycling in a high-stress package design

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Electron backscatter diffraction (EBSD) was used to study the microstructure and crystal orientation evolutions in lead-free solder joints during thermal cycling. With different package designs, the stress levels imposed on solder joins vary. More significant recrystallization and large opening cracks were observed in high stress wafer-level-chip-scale (WLCSP) packages. The effects of Sn crystal orientation on recrystallization, crack nucleation and growth during thermal cycling are characterized. In the early stage of thermal cycling, more slip activity and microstructural evolution was observed in solder balls that have Sn crystal orientations with [001] direction nearly parallel to the interfaces. Sn crystal orientation and misorientation distribution in selected solder balls are analyzed throughout their thermal history. By using fine-step EBSD scans, local crystal orientation gradient and deformation-induced lattice rotations are assessed, and are correlated with slip activities. The continuous recrystallization mechanism that leads to development of vulnerable high angle grain boundaries during thermal cycling is discussed.
Round Robin of EBSD Grain Size Measurements (ASTM E2627)

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The grain size of a polycrystalline material's microstructure is an engineering parameter that has a well-established relationship with many critical material properties and mechanical behaviors. Depending on the material, these include the basic Hall-Petch relationship with yield strength, complex fatigue and creep behaviors, corrosion performance, and various optical and electronic properties. Standard practices for measuring grain size, such as ASTM E112 and ASTM E1382, have been an integral part of material specifications and quality control in many engineering applications.

Recently, ASTM E2627 was established to provide a standard practice for grain size measurements using EBSD as opposed to optical methods. To assess the reproducibility of the EBSD method, a round robin study consisting of 20 laboratories was performed. A fully-annealed extrusion of a single Inconel 690 heat was used as the test material. Additional analyses outside the practice of the standard were also performed to compare ASTM E2627 with the traditional ASTM E112 methodology and engineering practices. Results, analyses, and participant feedback indicate that, while ASTM E2627 provides a good framework for basic grain size analysis using EBSD, significant improvements are needed. Moreover, the relationship with ASTM E112 measurements is not straightforward.
Zirconium alloys are used extensively in the nuclear power industry because of their unique combination of mechanical strength, corrosion resistance, and low neutron cross section. However, during exposure to a hot water environment, these alloys do develop a porous oxide corrosion film, ZrO$_2$. The oxide film has a significantly lower thermal conductivity compared to the metal and impacts thermal performance of zirconium alloy clad fuel elements. One question about the corrosion process that has drawn significant scientific interest is the influence of the metal grain orientation on the oxidation rate. Prior work in this area has relied on experiments involving large grained substrates or single crystals. While providing useful information, only a subset of possible orientations present in a specimen can be examined. The results presented here correlate the thicknesses of oxide film with the crystallographic orientation of individual grains in polycrystalline Zircaloy-4. The orientations of metal grains were obtained from a series of electron backscatter diffraction (EBSD) maps taken along the oxide metal interface prepared in cross-section. To visualize the relationship between metal grain orientation and oxide thickness, a plot was constructed using the inverse pole figure (IPF) representation of grain orientations and superimposed with the film thickness data. While absolute differences in oxide film thickness were small, the results from the combined EBSD/SEM study showed that the oxide film was thickest on grains with the basal pole oriented normal to the specimen surface. Overall, the relationship between oxide thickness and orientation was complex. The approach described in this work represents a significant improvement in the number of orientations that can be examined and compared with oxide thickness. As a result, a more comprehensive description of the relationship between oxidation rate and metal grain orientation is possible.
Evaluating Residual Plastic Strain Using SEM/EBSD

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The fabrication of many metallic engineering components involves plastic deformation of the component materials. Service conditions also sometimes produce plastic deformation in component materials. Even simple temperature excursions can sometimes produce local plastic strains in components due to coefficient of thermal expansion mismatch. This presentation demonstrates how the SEM EBSD technique can be used to estimate residual plastic strains in common commercial alloys including Ni-base Superalloys, Stainless Steels, and Zirconium alloys.
Laser-engineered net shape forming is a three-dimensional metal printing process that can be used to produce complex-shaped structures from design software. The process uses metal powder and a laser beam to build a structure layer by layer. The microstructure of such a material is also built one layer at a time, but subsequent layers necessarily influence the material that is already deposited, as it goes through thermal cycling, and perhaps some re-melting. Commercial purity Ti was manufactured both as a solid structure and with a porous framework using this technique. Parameters of the deposition were adjusted in an attempt to change the grain size and texture of the resulting material. The texture obtained is only moderately influenced with all specimens having a transformation texture that can be described from the EBSD data.
EBSD Analysis of Lunar Zircon: Resolution of the Early Impact History of the Moon

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It is thought that the early Moon was subjected to a large number of impact events during a period of time (~3.9 billion years ago) known as the Late Heavy Bombardment (LHB), sometimes referred to as the “Lunar Cataclysm”. The LHB hypothesis has shaped our current understanding of the early impact history of the solar system. The exact nature and timing of this process is disputed because the isotope systems used to determine the majority of ages of lunar rocks are known to reset readily under high pressure-temperature conditions, and thus a few late impacts and small number of sample sites could give a skewed impression of the impact history. Resolution relies on collection and interpretation of precise and robust age data. We have examined the microstructure and geochronology of the mineral zircon (ZrSiO$_4$) from lunar impact breccias collected during the Apollo 17 mission via optical microscopy, cathodoluminescence imaging, electron backscatter diffraction mapping and SHRIMP U-Pb dating. EBSD mapping shows that many zircon grains sourced from lunar igneous rocks preserve deformation microstructures that show a wide range in style and complexity, and are similar to with ‘shock’ microstructures in zircon from terrestrial impact sites and laboratory shock deformation experiments. Planar deformation features (PDFs) are documented in lunar zircon for the first time, and occur along {001}, {110}, and {112}, typically with 0.1-25 µm spacing. The widest PDFs associated with {112} contain micro-twin lamellae with 65° / <110> misorientation relationships. Deformation bands parallel to {100} planes and irregular low-angle (<10°) boundaries most commonly have <001> misorientation axes. This geometry is consistent with a dislocation glide system with <100>{010} during dislocation creep. Shock deformation microstructures in zircon are explained in terms of the interaction of strong stress (elastic, elastic-plastic and shock) waves with elastic anisotropy of zircon. PDFs form along a limited number of specific {hkl} planes that are perpendicular to directions of high Young’s modulus, suggesting that PDFs are likely to be planes of longitudinal lattice damage. Twinned {112} PDFs also contain directions of high shear modulus. A conceptual model is proposed for the development of different deformation microstructures during an impact event. This ‘shock deformation mechanism map’ is used to explain the relative timing, conditions and complexity relationships between impact-related deformation microstructures in zircon. Localized resetting of U-Pb system associated with the development of impact-related microstructures in these zircon grains provides a means of dating impact events. New zircon grown as acicular grains within pockets of quenched incipient impact melt and as impact-related reaction rims on pre-existing zircon and baddeleyite (ZrO$_2$) grains provide a means of dating impact events directly. Our data shows that these zircon grains preserve both a microstructural and isotopic record of pre-3.9 Ga impact events on the Moon. An alternative explanation to LHB hypothesis is that the lunar impact history consisted of a series of intense bombardment episodes interspersed with relatively calm periods of low impact flux.
Quantitative description of the microstructure in terms of its morphology and crystallography is widely used to link final properties to the processing path. From grain size and crystallographic texture, properties such as the anisotropy in strength, magnetism or elastic modulus can be predicted. Other properties related to the local orientation and morphology are surface roughness, sensitivity to Stress Corrosion Cracking, or electro-migration characteristics. In understanding the microstructure-property relation ship of polycrystalline materials, Orientation Imaging Microscopy (OIM) has emerged as the tool of choice to completely characterize a microstructure for both its stereological as well its crystallographic orientation aspects.