The 5th International Congress on

3DMS

3D Materials Science 2021

VIRTUAL EVENT
June 29–July 2, 2021

FINAL PROGRAM

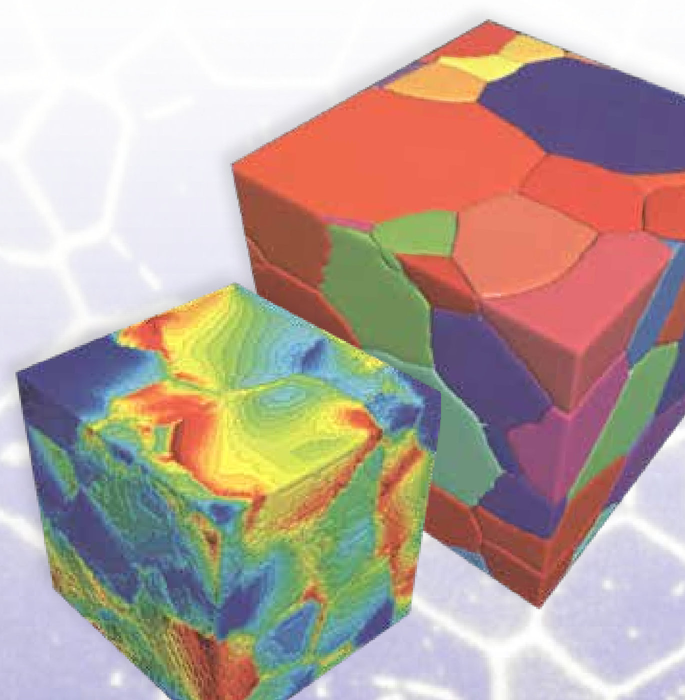
Current as of June 28, 2021

Sponsored by:

TMS

This congress is sponsored by the TMS Structural Materials Division (SMD) and the Advanced Characterization, Testing, and Simulation Committee.

www.tms.org/3DMS2021



SCHEDULE

Tuesday, June 22, 2021		
Virtual Platform Access Opens for Registrants Contributed oral and poster presentations available to view on-demand		On-Demand
Tuesday, June 29, 2021		
8:00 - 8:10 AM EDT	Introduction	Live
8:10 - 9:10 AM EDT	Plenary Presentation with Webinar-style Q&A	Live
9:10 - 9:20 AM EDT	Break	
9:20 - 9:50 AM EDT	Invited presentation with Webinar-style Q&A	Live
9:50 - 10:50 AM EDT	Breakouts: Q&A Discussions for On-demand Sessions	Live
Wednesday, June 30, 2021		
10:00 - 11:00 AM EDT	Plenary Presentation with Webinar-style Q&A	Live
11:00 - 11:10 AM EDT	Break	
11:10 AM - 12:10 PM EDT	Invited presentations with Webinar-style Q&A	Live
12:10 - 1:10 PM EDT	Breakouts: Q&A Discussions for On-demand Sessions	Live
Thursday, July 1, 2021		
12:00 - 1:00 PM EDT	Plenary Presentation with Webinar-style Q&A	Live
1:00 - 1:10 PM EDT	Break	
1:10 - 2:10 PM EDT	Invited presentations with Webinar-style Q&A	Live
2:10 - 3:10 PM EDT	Breakouts: Q&A Discussions for On-demand Sessions	Live
Friday, July 2, 2021		
8:00 AM - 12:00 PM EDT	Informal Networking by Topic	Live
Friday, July 2 to Saturday, July 31, 2021		
3DMS 2021 Virtual Platform Access Continues for Registrants		On-Demand
Saturday, July 31, 2021		
3DMS 2021 Virtual Platform Closes		On-Demand

The schedule is in Eastern Daylight Time (UTC-4:00).
Use the Time Zone Converter **to translate event times into your local time zone.**

TABLE OF CONTENTS

WELCOME

The 5th International Congress on 3D Materials Science (3DMS 2021) seeks to provide the premier forum for presentations of current interest and significance to the three-dimensional characterization, visualization, quantitative analysis, modeling, and development of structure–property relationships of materials, as well as big data and machine learning issues associated with 3D materials science. The virtual format of this year's meeting enables scientists, engineers, and researchers to engage in discussions and network with colleagues, as the COVID-19 pandemic presents obstacles to in-person meetings. The scientific community continues to innovate impressively during this challenging year. Your participation is appreciated as we explore intriguing topics in 3D materials science and further advance our knowledge and research. Thank you for being a part of 3DMS 2021.

TIPS FOR PARTICIPATING IN THE VIRTUAL EVENT

1) **Remove Distractions.** Notify others that you are at a conference. Set your out-of-office message in your e-mail client. Actively listen and take notes during the presentations, as if you were attending in person.

2) **Attend in Real Time.** Whenever possible, listen to presentations at their scheduled time so that you are able to ask questions and engage in discussions.

3) **Relax, and Enjoy!** If you miss something, don't worry. One of the benefits of a virtual event is the ability to access recordings of the presentations. You have access to the recordings and the proceedings through the end of July 2021.

ORGANIZING COMMITTEE

LEAD ORGANIZER:

Dorte Juul Jensen,
Technical University of Denmark, Denmark

PROGRAMMING CHAIR:

Erica Lilleodden,
Helmholtz-Zentrum Geesthacht, Germany

ADDITIONAL ORGANIZERS:

Scott Barnett, Northwestern University, USA
Keith Knipling, Naval Research Laboratory, USA
Matthew P. Miller, Cornell University, USA
Akira Taniyama, Nippon Steel, Japan
Hiroyuki Toda, Kyushu University, Japan
Lei Zhang, Chinese Academy of Sciences, China

SAVE THE DATE

The 6th International Congress on 3DMS
3D Materials Science 2022

June 26–June 29, 2022
Hyatt Regency Washington on Capitol Hill, Washington, D.C., USA
www.tms.org/3DMS2022

CONGRESS DETAILS

PLENARY AND INVITED SPEAKER PRESENTATIONS

Plenary and invited speakers will give live presentations with webinar-style question-and-answer sessions. Refer to the schedule to participate.

CONTRIBUTED PRESENTATIONS

The contributed presentations are pre-recorded and available on-demand. Access ends on July 31, 2021. To participate in the Q&A discussions for on-demand sessions, please watch the presentations in advance and come prepared with questions. We do not intend to play the presentations during these sessions. Refer to the schedule to participate.

VIRTUAL MEETING SETTINGS

The following practices are recommended for optimal participation:

- Google Chrome browser; use the latest version.
- Mute your audio settings before joining a session.
- Minimize distractions; consider an out-of-office response for e-mail.

For additional technical support, contact support@whova.com. For questions about programming, contact programming@tms.org.

GETTING STARTED

To participate in 3DMS 2021, you will need to log on to the virtual event platform. Upon registering, you will be e-mailed a link. Use the link to log on with your e-mail address and create a password. Enter your e-mail address and password to access the virtual platform.

TECHNICAL SUPPORT

3DMS 2021 is hosted on the Whova virtual event platform. If you need assistance using the virtual platform, contact the Whova support team by e-mailing support@whova.com.

CLOSED CAPTIONING

Access closed captioning by using Google Live Caption in the Chrome browser.

Open Settings >> Advanced >> Accessibility. Toggle Live Caption to On. Open a new tab with video content and play a video. Captions should appear once speaking begins.

To stop Live Caption, open Settings >> Advanced >> Accessibility and toggle Live Caption **Off**.

PUBLISHING PLAN

The TMS journal *Integrating Materials and Manufacturing Innovation* will be publishing a topical collection dedicated to the 3DMS 2021 meeting. This collection will take the place of a traditional conference proceedings publication.

The journal submission deadline is August 3, 2021. Visit the Publishing Plan page of the website for more information.

Jaafar El-Awady,
Johns Hopkins University, USA
"Acoustic Emission Measurements of Damage Accumulation and Crack Initiation in Metals at the Micron-scale"

Wednesday | 10:00 AM EDT | June 30, 2021

Recent advances in small scale experiments have significantly improved the ability to quantify the mechanisms that control deformation and failure in metals. In this talk a new methodology coupling acoustic emission measurements and high frequency in-situ scanning electron microscopy experiments to quantify the evolution of damage and crack initiation in nickel microcrystals will be presented. Mechanical properties are continuously monitored during the cyclic loading through analysis of the different instrument signals. Through the in-situ observations the nucleation and propagation speeds of persistent slip bands (PSBs) was quantified, while postmortem characterizations have led to an unprecedented understanding of the origin of these PSBs. Additionally, quantification of crack initiation and propagation was also analyzed from acoustic emission measurements, and the statistics of these events at different stages of crack propagation (i.e. short crack and long crack regimes) are quantified. Challenges and limitations of the methodology will also be discussed.

Satoshi Hata,
Kyushu University, Japan
"Toward Dynamic 3D Visualization of Dislocations by Electron Tomography"

Tuesday | 8:10 AM EDT | June 29, 2021

Observation of dislocation dynamics in three-dimensions (3D) is still challenging in the 3DMS research field. The authors' group recently obtained a preliminary result of subsequently repeating in-situ specimen straining and electron tomography (ET) observation for a steel specimen in which a dislocation was interacting with a spheroidized cementite [1]. Although the observed magnitude of the dislocation movement was small, a few tens nm, the observation demonstrated the importance of 3D observation: the dislocation movement was recognized from some directions while not from the other directions. This different visibility of the dislocation movement is due to the geometrical relationship between the active slip plane and the viewing directions. In other words, a 3D imaging technique is indispensable for visualizing arbitral dislocation movements in such an in-situ straining and observation experiment. There are technical issues to be resolved toward a complete establishment of a 3D dislocation dynamics imaging method using ET. For example, diffraction alignment is an essential part of visualizing dislocations in ET. If we can predict how dislocations move in a specimen, we could keep the diffraction condition during the dislocation movement by precisely setting the crystallographic orientation regarding the loading direction. However, the aligned diffraction condition actually can change during the deformation of the specimen with applied stress. Therefore, the authors propose an alternative way to visualize 3D dislocation dynamics "without" keeping particular diffraction conditions during ET data acquisition. A procedure of the alternative way will be discussed.

Helena Van Swygenhoven, Paul Scherrer Institute & Swiss Federal Institute of Technology Lausanne, Switzerland
"*Operando* and *Insitu* Synchrotron Experiments Following Microstructural Evolutions"

Thursday | 12:00 PM EDT | July 1, 2021

Thanks to the high brilliance and the fast detectors available at synchrotrons, operando and insitu experiments have become possible, adding the timescale to microstructural characterization techniques. Such experiments allow identification of operating mechanisms that are responsible for microstructural evolutions. This talk will demonstrate the potential of such experiments. Addressing microstructures of superelastic NiTi alloys, insitu xray diffraction experiments provide understanding why some microstructures can accommodate for changing strain paths, and why degradation during fatigue loading depends strongly on the strain path (Acta Materialia 144(2018)68 and 167(2019)149). Addressing 3D printing, operando selected laser melting of Ti6Al4V during xray diffraction, allows to track with a time resolution of 50µs the dynamics of the á and â phases during fast heating and solidification, providing the cooling rates of each phase and the duration the â phase exists during processing. Such an experiment reveals for instance the link between the cooling rate, the scan vector length and the resulting microstructure (Materials Today, November 2019). This research is performed within the ERC Advanced Grant MULTIAX (339245), PREAMPA and the CCMX-AM3 challenge projects financed by the ETH board and the Swiss watch and precious metal industry.

TMS would like to thank the following sponsors for their support of the event:

ThermoFisher Scientific
www.thermofisher.com
3D visualization and analysis of your imaging data allows you to better understand your materials structure, properties and performances. No matter what scale and data modality you use, Thermo Scientific Avizo Software provides optimized workflows for advanced materials characterization and quality control.

Zeiss
www.zeiss.com
Carl Zeiss Microscopy has recently introduced the CrystalCT – the world's first X-ray diffraction contrast tomography system based on a laboratory microCT platform. CrystalCT advances your research into engineering material systems by nondestructively mapping and characterizing three-dimensional grain structures in polycrystalline materials. Furthermore, ZEISS is a provider of correlative workflow solutions spanning lengthscales in 3D, including transfer of samples from X-ray to Crossbeam laser to utilize fs-laser ablation for site-specific targeting, access, and interrogation of nanoscale features buried deep within bulk specimens.

Carl Zeiss Microscopy is the world's only manufacturer of light, X-ray and electron/ion microscopes, offering dedicated systems for industry, materials research, and life science. A dedicated and well-trained sales force, an extensive support infrastructure and a responsive service team enable customers to use their ZEISS microscope systems to their full potential.

Xnovotech
www.xnovotech.com
Xnovo Technology ApS delivers novel, high-performing, and user-friendly 3D imaging tools for applications within the fields of engineering, materials and geosciences. The company is an offshoot of more than 20 years of synchrotron and diffraction imaging research at the Technical University of Denmark.

By registering for this meeting, attendees accept the terms of the TMS Privacy Policy and agree to abide by TMS policies, including the Meetings Code of Conduct and the TMS Anti-Harassment Policy. For additional information on policies related to TMS events, visit the TMS Meetings Policies page and the Code of Conduct portal. A complete listing of Society policies can be accessed through the Society Bylaws & Policies section of the TMS website.

REFUNDS

The deadline for all refunds was May 31, 2021. No refunds will be issued at the congress.

TIME ZONES

Unless otherwise noted, all times for this congress and related events will take place in the local time zone, EDT (UTC/GMT -4 hours). This is the local time for TMS headquarters in Pittsburgh, Pennsylvania, USA. Use a tool like the Time Zone Converter to translate event times into your local time zone.

LANGUAGE

The meeting and all presentations and program materials will be in English.

CURRENCY

All meeting fees are expressed in U.S. dollars (USD).

ACCESS TO RECORDED PRESENTATIONS AFTER THE CONGRESS

Please note that registrants will have access to all recorded presentations from 3DMS 2021 through July 31, 2021. Log in to the virtual event platform at any time after the congress has ended to view content.

The 5th International Congress on

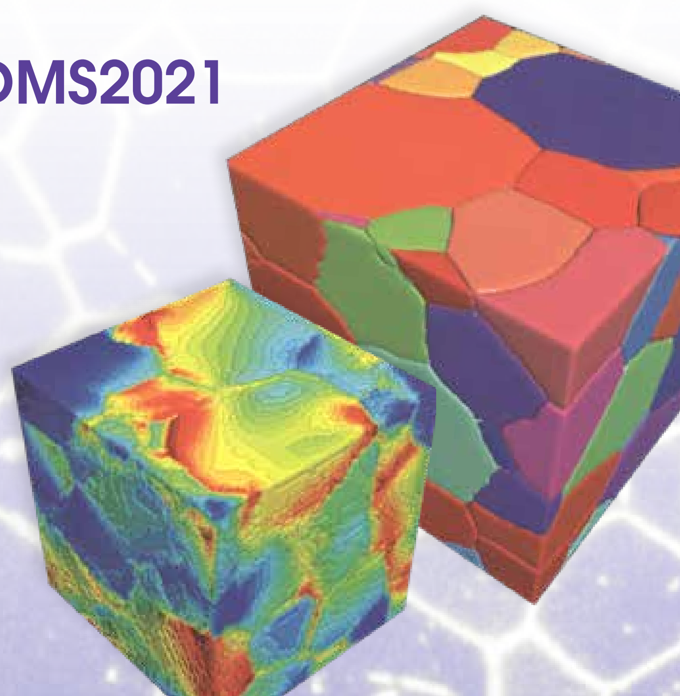
3DMS

3D Materials Science 2021

VIRTUAL EVENT
June 29–July 2, 2021

TECHNICAL PROGRAM

www.tms.org/3DMS2021



Tuesday Plenary

Tuesday AM | June 29, 2021

Session Chair: D. Juul Jensen, Technical University of Denmark

8:00 AM Introductory Comments

8:10 AM Plenary
Toward Dynamic 3D Visualization of Dislocations by Electron Tomography: *S. Hata*[1]; H. Saito[1]; M. Murayama[2]; [1]Kyushu University; [2]Virginia Tech; Pacific Northwest National Laboratory; Kyushu University

Observation of dislocation dynamics in three-dimensions (3D) is still challenging in the 3DMS research field. The authors' group recently obtained a preliminary result of subsequently repeating in-situ specimen straining and electron tomography (ET) observation for a steel specimen in which a dislocation was interacting with a spheroidized cementite [1]. Although the observed magnitude of the dislocation movement was small, a few tens nm, the observation demonstrated the importance of 3D observation: the dislocation movement was recognized from some directions while not from the other directions. This different visibility of the dislocation movement is due to the geometrical relationship between the active slip plane and the viewing directions. In other words, a 3D imaging technique is indispensable for visualizing arbitral dislocation movements in such an in-situ straining and observation experiment.There are technical issues to be resolved toward a complete establishment of a 3D dislocation dynamics imaging method using ET. For example, diffraction alignment is an essential part of visualizing dislocations in ET. If we can predict how dislocations move in a specimen, we could keep the diffraction condition during the dislocation movement by precisely setting the crystallographic orientation regarding the loading direction. However, the aligned diffraction condition actually can change during the deformation of the specimen with applied stress. Therefore, the authors propose an alternative way to visualize 3D dislocation dynamics "without" keeping particular diffraction conditions during ET data acquisition. A procedure of the alternative way will be discussed.

9:10 AM Break

Tuesday Invited

Tuesday AM | June 29, 2021

Session Chair: A. Taniyama, Nippon Steel Corporation

9:20 AM Invited
Development of Comprehensive Evaluation Techniques for Extraterrestrial Materials: *M. Uesugi*[1]; A. Takeuchi[1]; K. Uesugi[1]; [1]Japan Synchrotron Radiation Research Institute

Synchrotron radiation (SR) computed tomography (CT) is one of the important tools for the analysis of extraterrestrial materials, such as meteorites and cosmic dusts and especially returned samples by spacecraft missions, because of its non-destructive feature. Internal structure of the extraterrestrial material can be obtained with spatial resolution less around 1µm before the destructive analyses. However, materials included in the samples cannot be determined uniquely, because the range of the X-ray absorption coefficient of constituent materials is significantly overlapped in previous system. A multi-mode CT system for the comprehensive analysis of extraterrestrial materials has been developed at BL20XU/SPring-8. The system composed of multiscale CT, XRD-CT and phase contrast CT setup, to identify variety of components in extraterrestrial materials, such as silicates, iron-Ni-sulfur alloys, carbonates, and organic materials. The system will be applied for the analysis of returned samples of asteroid Ryugu, after return back of "HAYABUSA2" spacecraft in late 2020.

Characterization Methods I

Tuesday AM | June 29, 2021

Session Chair: M. Miller, Cornell University

A New Generation of Synchrotron-based Design Tools at MSN-C (The Materials Solution Network at CHESS): *M. Miller*[1]; P. Shade[2]; [1]Cornell University; [2]Air Force Research Laboratory

Over the past decade at high energy synchrotron light sources around the world, a broad range of x-ray scattering experiments have been transitioning to materials characterization measurements, thereby shifting the focus from the x-rays and photon science to the materials themselves and their applications in engineering design. This talk describes several examples examining the processing and performance of structural alloys within the new Materials Solution Network at CHESS (MSN-C) funded by AFRL. MSN-C provides beamtime to DoD researchers on two x-ray beamlines – one at high energy for metals and the other at lower energy for polymers and composites - to utilize these developing tools on some of the most pressing DoD materials challenges.

Advanced In-situ Loading Environments for Synchrotron X-ray Diffraction Experiments: *P. Shade*[1]; B. Blank[2]; M. Obstalecki[1]; W. Musinski[1]; P. Kenesei[3]; J. Park[3]; J. Almer[3]; D. Pagan[4]; K. Nygren[4]; P. Ko[4]; C. Budrow[5]; D. Menasche[6]; J. Bernier[7]; R. Suter[8]; T. Turner[1]; [1]Air Force Research Laboratory; [2]PulseRay; [3]Advanced Photon Source; [4]Cornell High Energy Synchrotron Source; [5]Budrow Consulting; [6]Hamiltonian Group; [7]Lawrence Livermore National Laboratory; [8]Carnegie Mellon University

High energy x-ray characterization methods hold great potential for gaining insight into the behavior of materials and providing comparison datasets for the validation and development of mesoscale modeling tools. A suite of techniques have been developed by the x-ray community for characterizing the 3D structure and micromechanical state of polycrystalline materials; however, combining these techniques with in situ mechanical testing under well characterized and controlled boundary conditions has been challenging due to experimental design requirements. In this presentation, we describe advanced in situ loading environments that have been developed for communal use at the Advanced Photon Source and the Cornell High Energy Synchrotron Source. Example 3D datasets that have been collected using this hardware and their application for materials modeling efforts will be discussed.

Application of High Energy Imaging CT to Investigate Local 3D Short Fatigue Crack Closure Behavior in Ti-6Al-4V Alloy: *V. Tubei*[1]; H. Toda[1]; K. Hirayama[1]; M. Hassanipour[1]; A. Takeuchi[2]; M. Uesugi[2]; [1]Kyushu University; [2]Japan Synchrotron Radiation Research Institute

High energy imaging CT has been utilized for the in-situ observation of local 3D short fatigue crack closure behavior in Ti-6Al-4V alloy at SPring-8 synchrotron radiation facility in Japan. In recent years, projection CT with a resolution of ~1 µm has been employed to non-destructively observe crack closure. However, the short crack tip opening displacement is usually below 1µm and thus this technique cannot be used to accurately visualize crack front closure behavior. Recent CT technology advances have led to the achievement of ultra-high resolution imaging that is realized by focusing the high energy X-ray beams using the apodization Fresnel Zone Plate. This imaging CT was utilized in this study with X-ray energy of 30 keV. In combination with the high contrast attained by using the Zernike Phase Plate, the internal microstructure, crack and crack closure occurrence at the crack front were readily visualized at a resolution of 130 nm.

Phase Change of Pyrolitic Material: In-situ Transformation and Induced Microstructures at 660 km Depth: *J. Gay*[1]; E. Ledoux[1]; M. Krug[2]; A. Pakhomova[3]; I. Kupenko[2]; J. Chantel[1]; C. Sanchez-Valle[2]; S. Merkel[1]; [1]University of Lille; [2]University of Münster; [3]DESY, Hamburg, Germany

Phase transformations within the Earth's mantle can cause crystal growth in preferred orientations among other phenomena. These phase changes are a result of changes in pressure and temperature with depth. At 660 km, ringwoodite and garnet decompose to form bridgmanite and ferropericlase. These transformations generate microstructures, cause seismic reflectors, and viscosity changes that impact mantle dynamics. Understanding these transformation microstructures will hence better constrain processes in the Earth's mantle. These transformations are not martensitic, but grains grow with preferred orientations through diffusive processes. H e r e we implement synchroton multigrain x-ray diffraction in a laser heated diamond anvil cell. We are able to track individual grains, crystal structures, and orientations, while being compressed in-situ at pressures ranging from 18 to 55 GPa and temperatures of ~1800 K. Using this experimental approach, we can then use obtained transformation textures to refine current seismic models to better understand seismic observables in the Earth's mantle.

Redefining ESRF's Tomography Ecosystem for the EBS Upgrade: *N. Viganò*[1]; [1]ESRF - The European Synchrotron

The scheduled ESRF EBS upgrade will offer unprecedented opportunities for tomographic applications. The higher available X-ray photon flux and coherence will allow to overcome current limitations in imaging and microstructure characterization techniques like: X-ray phase contrast tomography (XPCT), X-ray diffraction computed tomography (XRD-CT) and X-ray fluorescence computed tomography (XRF-CT). The increased X-ray flux and the consequent reduction of acquisition times allow to increase the acquisition rate and data throughput. This will unlock a number of time-lapse observation studies and multi-modal techniques that were not possible before, but it will also put more strain on the current data processing pipeline.In this oral presentation, I will present the undergoing restructuring of the ESRF tomography acquisition and processing ecosystem. I will also discuss the plans for improving the currently offered tomographic reconstruction tools, by leveraging advances in multi-channel/multi-modal reconstructions and machine-learning.

Three-dimensional Sub-grain Mapping of Lattice Strains and Orientations in Polycrystals: *P. Reischig*[1]; W. Ludwig[2]; [1]InnoCryst Ltd; [2]CNR / ESRF

We demonstrate the capability of efficiently mapping the complete local strain and orientation tensor field at the sub-grain level in three dimensions inside a polycrystal, in a non-destructive way. Diffraction Contract Tomography can provide detailed 3D grain maps of moderately deformed polycrystals at the micrometre scale, utilising high-energy, monochromatic synchrotron X-rays. Using full-beam illumination and a single sample rotation enables fast and efficient scans. The ESRF source upgrade will bring scanning times of the order of minutes in in-situ experiments within reach. The highly convoluted experimental data pose a large-scale, 12-dimensional, ill-posed, non-linear reconstruction problem. A forward model and iterative solver has been developed to infer the grain shapes, sub-grain orientation and strain fields (order of 10^{-3} to 10^{-4}). Furthermore, the single crystal elastic moduli are fitted from the strain data. The data acquisition and processing method, and experimental validation on a Gum metal sample under tensile load will be discussed.

Materials Dynamics in 3D

Tuesday AM | June 29, 2021

Session Chair: H. Friis Poulsen, Technical University of Denmark

3D Mapping of Type-III Stress in Plastically Deformed Steel: *Y. Hayashi*[1]; [1]RIKEN SPring-8 Center

Materials modeling for the prediction of mechanical behavior of alloys requires advances in multiscale 3D measurement of stress and its evolution produced by mechanical events. We show non-destructive 3D mapping of intragranular local (type-III) stress in plastically deformed multiple grains embedded in mm-sized bulk steel using scanning 3DXRD. The grains were illuminated by a high-energy X-ray microbeam from various directions. Non-overlapped multiple diffraction spots per grain were detected through a conical slit. Type-III elastic strain tensors under 5% plastic deformation were determined from the differences between observed scattering angles and stress-free Bragg angles. The observed type-III stress deviated by a value as high as the yield strength from grain-averaged (type-II) stress. The type-III stress was in a triaxial stress state even under elongation smaller than the uniform elongation. Multiscale modeling of deformation and failure of steel will be facilitated using the non-destructive 3D type-III stress tensor mapping data.

4D Characterization of Solidification in Al-based Droplets: *J. Valloton*[1]; A. Bogno[1]; C. Schlepütz[2]; G. Reinhart[3]; M. Rappaz[4]; H. Henein[1]; [1]University of Alberta; [2]Paul Scherrer Institut; [3]Aix-Marseille Université; [4]Ecole Polytechnique Fédérale de Lausanne
Sub-millimeter droplets of near eutectic Al-Cu alloy were melted and solidified using the laser-based heating system available at the TOMCAT beamline of the Swiss Light Source. Cooling rates were monitored using a thermocouple placed at the base of the crucible. During cooling, radiography scans at 1 kHz frame rate were acquired in order to capture the solidification front in-situ. The corresponding field of view allowed the scanning of two droplets at once while providing sufficient resolution to observe the envelope of the solidification front (0.81 µm pixel size). Micro-tomography scans with a pixel size of 0.325 µm were then performed on the solidified samples to analyze the microstructure. Primary dendrites of either a-Al or Al_2Cu were observed, followed by a eutectic front. The low growth velocities measured by radiography (10^0-10^{-1} mm/s) suggests low undercoolings. Surprisingly, a deviation from the typical <100> growth direction has been observed for primary Al dendrites.

Characterization of Free-growing Dendrites Using 4D X-ray Tomography and Machine Learning: *T. Stan*[1]; K. Elder[1]; X. Xiao[2]; P. Voorhees[1]; [1]Northwestern University; [2]Brookhaven National Laboratory
When metallic alloys are cooled from the liquid, in almost all cases from castings to additive manufacturing, the metal freezes via the formation of dendrites. Using high temporal and spatial resolution synchrotron 4D x-ray computed tomography (XCT), we present the first in-situ observations of free-growing "hyperbranched" dendrites in Al-Zn. Convolutional neural networks were used to segment the reconstructed datasets, and interface energy anisotropy calculations combined with XCT morphological observations were used to track the crystallographic growth directions of dendrite tips through time. We show that a single dendritic root can have arms with tip morphologies ranging from nearly spherical to highly elliptical. Dendrite fragments are also observed moving both with and against gravity, indicating density changes during growth. The 4D experiments give new information regarding the evolution of dendrite tip curvatures, velocities, crystallography, symmetries, and the interface energy anisotropies of Al alloys.

Dark Field X-ray Microscopy of Recovery Annealing of Cold Rolled Fe-Si and Fe-Si-Sn Alloys: *C. Yildirim*[1]; N. Mavrikakis[2]; M. Gauvin[2]; P. Cook[3]; M. Kutsal[4]; R. Hubertt[2]; W. Saikaly[2]; H. Poulsen[5]; M. Dumont[6]; H. Zapolsky[7]; D. Mangelick[8]; C. Detlefs[4]; [1]CEA Grenoble; [2]OCAS; [3]BOCU; [4]European Synchrotron Radiation Facility; [5]DTU; [6]Universite Aix Marseille; [7]Universite De Rouen; [8]IM2NP
Numerous interesting phenomena occur at different microscopic scales during the annealing of engineering materials. Understanding these phenomena are essential not only for optimising performance of macroscale components, but also for the validation of structural models. Here, we present an in-situ study on the effect of Sn on the recovery annealing of cold rolled Fe-3%Si alloys using Dark Field X-ray Microscopy. DFXM is a recent non-destructive, diffraction-based technique that allows 3D mapping of orientation and lattice strain with 100 nm spatial and 0.001° angular resolution within embedded in bulk samples. The evolution of relative strain and orientation in alpha Fe grains upon annealing show that Sn slows down the recovery kinetics. DFXM results provide a direct observation of the strain field around dislocation loops that remain static upon annealing.These findings agree with the complementary micro-hardness and EBSD measurements. We further discuss this by an atomistic modelling on Sn-dislocation interaction.

Microstructural Stability of a Three-phase Eutectic Examined *via* 4D X-ray Nano-tomography: *G. Lindemann*[1]; V. Nikitin[2]; V. De Andrade[2]; M. De Graef[3]; A. Shahani[1]; [1]University of Michigan; [2]Argonne National Laboratory; [3]Carnegie Mellon University
Multiphase materials often operate at elevated temperatures where coarsening may cause significant microstructural changes. Therefore, a thorough understanding of coarsening mechanisms in such materials is critical to accurately predict their behavior in-service. Here, we use 4D x-ray nano-tomography (three spatial dimensions and time) to investigate microstructure evolution of an Al-Ag_2Al-Al_2Cu three-phase eutectic alloy during isothermal annealing below the eutectic temperature. With the aid of a new, *in situ* furnace and an innovative total variation regularization reconstruction technique, we visualize the evolution of eutectic lamellae and identify two unique coarsening modes that occur in parallel for the two intermetallic phases. Ag_2Al coarsens *via* 2D Ostwald ripening when the rod's initial radius is greater than a critical value while Al_2Cu evolves *via* 3D Ostwald ripening. We also consider the role of crystallographic anisotropy. Our efforts reveal the need to expand contemporary theories of eutectic coarsening to better describe multiphase and multicomponent systems.

Observation of Plastic Slip Localization in a Ti-7Al Alloy Using X-ray Topotomography: *P. Callahan*[1]; J. Stinville[2]; M. Echlin[2]; W. Ludwig[3]; H. Proudhon[4]; T. Pollock[2]; [1]Naval Research Laboratory; [2]University of California, Santa Barbara; [3]ESRF - European Synchrotron Radiation Facility; [4]MINES ParisTech, PSL Research University, MAT-Centre des Materiaux, CNRS
Specimens of the titanium alloy Ti-7Al were studied with high-resolution digital image correlation (HR-DIC) in an SEM along with x-ray diffraction contrast tomography (DCT) and x-ray topotomography (TT) at a synchrotron facility. Several of the samples were pre-strained near the elastoplastic transition before DCT and TT was performed. This enabled regions of localized plasticity, or slip localization, observed in 3D within both bulk and surface grains using TT to be correlated with HR-DIC observations of plasticity at the surface. In another sample, a number grains of interest were identified prior to deformation using DCT. TT data was collected from these grains before deformation, and then at several deformation steps in order to study the evolution of slip localization in 3D. These observations can provide new insights into plastic localization as it relates to microstructural configurations and on plastic transmission between grains both at the surface and internally in 3D.

The Effect of 3D Microstructure on Deformation-induced Martensitic Transformation in Austenitic Fe-Cr-Ni Alloys: Y. Tian[1]; M. Guan[1]; H. Liu[2]; R. Suter[2]; P. Kenesei[3]; J. Park[3]; *T. Hufnagel*[1]; [1]Johns Hopkins University; [2]Carnegie Mellon University; [3]Argonne National Laboratory
Deformation-induced transformation of individual grains in Fe-Cr-Ni alloys from austenite (fcc) to a'-martensite (bcc) is strongly influenced by the local stress state, which in turn is a function of the local microstructure. To investigate these effects, we have performed near-field and far-field high-energy diffraction microscopy (HEDM) to characterize the deformation-induced martensitic transformation in high-purity Fe-Cr-Ni alloys during in situ tensile loading. For the range of Fe-Cr-Ni alloys studied, martensite start () temperatures are close to room temperature, where both stress-assisted and strain-induced formation of martensite are possible during deformation. Three-dimensional (3D) in situ reconstructions were performed including austenitic substrate and newly formed a'-martensite. Both the strain tensor evolution and neighboring environment of individual austenite grains were tracked during the loading, and correlated with martensite formation. We discuss the transformation mechanism discussed in light of the stacking-fault energy of the alloys and the grain-averaged stress state.

Time Evolution of Dislocations and Defects with Dark-field X-ray Microscopy: *L. Dresselhaus-Cooper*[1]; S. Ali[1]; S. Breckling[2]; P. Cook[3]; C. Detlefs[4]; J. Eggert[1]; E. Galtier[5]; L. Gavilan-Marin[6]; A. Gonzalez[2]; M. Howard[7]; K. Katagiri[8]; H. Kim[9]; S. Kim[10]; S. Kim[10]; S. Kim[9]; S. Kim[9]; S. Kuschel[5]; H. Lee[5]; C. Lin[11]; R. McWilliams[12]; D. Nam[10]; N. Ozaki[8]; R. Pedro[13]; H. Poulsen[14]; A. Saunders[1]; F. Schoofs[14]; T. Sekine[15]; H. Simons[14]; B. Wang[11]; W. Yang[11]; G. Winthers[14]; C. Yildirim[16]; [1]Lawrence Livermore National Laboratory; [2]Nevada National Security Site; [3]BOKU; [4]ESRF; [5]SLAC National Accelerator Laboratory; [6]NASA Ames; [7] Nevada National Security Site; [8]Osaka University; [9]Sogang University; [10]Pohang Accelerator Laboratory; [11]HPSTAR; [12]University of Edinburgh; [13]Massachusetts Institute of Technology; [14]Technical University of Denmark; [15]U.K. Atomic Energy Authority; [16]CEA

A material's response to its surroundings depends on both its native properties and the imperfections (defects) in its structure. While techniques exist to probe material defects, they are mainly limited to surface measurements or rastered scans that cannot measure the dynamics of irreversible processes. Dark-field X-ray microscopy (DFXM) can now directly image defects in single- and poly-crystals, resolving the lattice tilt and inclination with high sensitivity over long length-scales. We have extended DFXM to synchrotron and X-ray free electron laser experiments to directly image the time evolution of defects in materials—focusing on defects at the mesoscale (e.g. dislocations). These movies resolve the dynamics of defects in single crystals, directly measuring their mobility and interactions by measuring the strain with 10-5 resolution over hundreds of micrometers. With this new tool, I demonstrate how dislocations evolve in aluminum during annealing, and how cumulative radiation damage evolves. Time-resolved DFXM holds important opportunities for future studies on mesoscale dynamics, as it can inform models that have previously been refined only by indirect measurements and multi-scale models.

Towards Quantitative Modeling of Precipitate Morphology Evolution in Co-based Superalloys: W. Wu[1]; J. Warren[2]; P. Voorhees[3]; *O. Heinonen*[4]; [1]Center for Hierarchical Materials Design (CHiMaD), Northwestern University; [2]Materials Measurement Laboratory, National Institute of Science and Technology; [3]CHiMaD, Northwestern University; Department of Materials Science and Engineering, Northwestern University; [4]Materials Science Division, Argonne National Laboratory

Cobalt-based superalloys with γ/γ' microstructures offer great promise as candidates for next-generation high-temperature alloys. It is essential to understand the thermodynamic and kinetic factors that influence the microstructural evolution of these alloys in order to optimize the alloy compositions and processing steps with a goal to improve their coarsening, creep and rafting behavior. We are developing a phase field approach using the chemical free energies extracted from CALPHAD thermodynamic data developed at NIST to predict the equilibrium shapes of Co-Al-W γ' precipitates. We find that the equilibrium shape of the precipitate results from a delicate competition between chemical, interfacial and elastic energies. We are examining how modeling input parameters such as the form of the chemical free energy, anisotropy of the interfacial energy, and variant misfit and diffusivity due to temperature change affect the coarsening and rafting behavior of superalloy models and relate these parameters to experimentally available values.

Process Microstructure Properties I

Tuesday AM | June 29, 2021

Session Chair: D. Rowenhorst, Naval Research Laboratory

Grain Scale Analysis of Deformation in Ti6Al4V Anisotropically Built Using Electron Beam Melting Powder Bed Additive Manufacturing Process: *M. Mian*[1]; J. Razmi[1]; L. Ladani[1]; [1]Arizona State University

Ti6Al4V alloy, originally developed for aerospace applications, has found wide acceptance in various industries due to its high strength and low density. EB-PBF additive manufacturing is an efficient technique to fabricate complicated geometries with this hard metal. High temperature deformation behavior of Ti6Al4V is very crucial specially to the aircraft engine manufacturers. Therefore, this study focuses on the anisotropic mechanical behaviors at elevated temperatures along with potential reasons for these behaviors. Ti6Al4V alloy is observed to experience flow softening during hot deformation around 600˚C temperature. Electron back scattered diffraction (EBSD) analysis is conducted using samples before and after the tensile test to determine possible softening mechanisms and microstructural changes. Microstructural features such as crystallographic texture and grain boundary misorientation angles are investigated via different EBSD maps. Moreover, a 3D X-ray CT analysis is conducted on the samples to investigate the internal defects and their impact on the inherent anisotropic behavior.

Thermal Expansion of Ti-7Al Using High Energy X-ray Diffraction Microscopy: *R. Lim*[1]; D. Pagan[2]; J. Bernier[3]; A. Rollett[1]; [1]Carnegie Mellon University; [2]Cornell High Energy Synchrotron Source; [3]Lawrence Livermore National Laboratory

Hexagonal metals have anisotropic coefficients of thermal expansion, and there is little agreement in literature on the CTEs for these metals. High energy x-ray diffraction microscopy, a non-destructive, in situ, microstructural characterization technique, has been used to determine the anisotropic coefficients of thermal expansion (CTEs) for Ti-7Al. Two samples of polycrystalline Ti-7Al were continuously heated from room temperature to 850C while far-field HEDM scans were being taken. The lattice parameters at a given temperature were calculated based on the distribution of lattice parameters of the individual grains. The results showed that the CTEs in were different in the a- and c-directions respectively, and the change in the ratio of these CTEs explained the discrepancies found in literature. It was also found that the equivalent strain and von Mises stress were lower after the thermal cycle with the outliers in the initial state having shifted the most after the thermal cycle.

Poster

Three-dimensional Electron Backscatter Diffraction Analysis of Fatigue Crack Propagation in Steel: *M. Kikuzuki*[1]; T. Kato[1]; T. Makino[1]; Y. Kimura[1]; [1]Nippon Steel Corporation

Resistance against crack propagation is dominant effective in improving fatigue strength in steel. Identifying the behavior of non-propagating fatigue crack tip which stop growing during cyclic stress test reveals the factors inhibiting fatigue cracks. The conventional cross-sectional observations around the cracks have been performed; however, it was difficult to determine the crystal orientation of the fracture surfaces of fatigue crack tip from such 2D observation.In this study, 3D electron backscatter diffraction (3D-EBSD) analysis using a focused ion beam scanning electron microscope (FIB-SEM) visualized the distribution of crystal grains and the primary crystal orientation of the fracture surface around the non-propagating fatigue crack tip in a ferritic model steel. The result shows that the fatigue crack propagated on the three planes of equivalent {101} slip planes in the steel, and fatigue crack tip stopped not at grain boundary but inner grain.

X-ray Tomography and 3D X-ray Diffraction for Quantifying Particle Rearrangements in Granular Materials: *R. Hurley*[1]; C. Zhai[1]; E. Herbold[2]; S. Hall[3]; [1]Johns Hopkins University; [2]Lawrence Livermore National Laboratory; [3]Lund University

The behavior of granular materials undergoing macroscopic deformation is characterized by local particle rearrangements and other deformation mechanisms, including fracture. Recent advances in X-ray computed tomography (XRCT) and 3D X-ray diffraction (3DXRD) at synchrotron facilities provides a unique approach for quantifying the details of these deformation mechanisms. Previously, access to the type of particle-scale detail that XRCT and 3DXRD can provide in 3D was restricted to photoelasticity in 2D or numerical simulations. Here, we will discuss recent experiments performed on granular materials that combined XRCT and 3DXRD to examine deformation mechanisms occurring during uniaxial and triaxial compression of granular materials. We will focus on deformation mechanisms including local particle strains, non-affine motion, and rotations. We will discuss the application of machine learning to identify and predict the location of such local deformation mechanisms from our X-ray measurements.

Statistical Analysis of Crystal Plasticity on Commercially Pure Titanium by Coupling 4D Testing and Finite Element Simulations: *C. Ribart*[1]; A. Marano[1]; F. N'Guyen[1]; A. King[2]; D. Ryckelynck[1]; W. Ludwig[3]; H. Proudhon[1]; [1]MINES ParisTech, PSL Research University, Centre des Materiaux; [2]Synchrotron SOLEIL; [3]ESRF Synchrotron

This talk will present a multimodal experiment on Grade 2 Titanium for the study of polycrystal plasticity. The data set is composed of 3D and 4D in-situ Diffraction Contrast Tomography (DCT) tests conducted at SOLEIL synchrotron. In addition in-situ SEM tests (high resolution SEM mapping and EBSD scans) have been performed on specimens where the microstructure was previously measured by DCT. Digital twins of the specimens are built using the reference 3D microstructure to access the local stress fields by means of crystal plasticity finite element simulations. The volumes studied are of the order of 1 mm cube and each contains several thousands of grains to be representative of the mesoscopic scale. This allows statistical evaluation of slip system activation and lattice curvature extracted from the data set. The method is an important step towards constitutive behaviour identification at the scale of the grain.

Wednesday Plenary

Wednesday AM | June 30, 2021

Session Chair: K. Knipling, Naval Research Laboratory

10:00 AM Plenary

Acoustic Emission Measurements of Damage Accumulation and Crack Initiation in Metals at the Micron-scale: *J. El-Awady*[1]; [1]Johns Hopkins University

Recent advances in small scale experiments have significantly improved the ability to quantify the mechanisms that control deformation and failure in metals. In this talk a new methodology coupling acoustic emission measurements and high frequency in-situ scanning electron microscopy experiments to quantify the evolution of damage and crack initiation in nickel microcrystals will be presented. Mechanical properties are continuously monitored during the cyclic loading through analysis of the different instrument signals. Through the in-situ observations the nucleation and propagation speeds of persistent slip bands (PSBs) was quantified, while postmortem characterizations have led to an unprecedented understanding of the origin of these PSBs. Additionally, quantification of crack initiation and propagation was also analyzed from acoustic emission measurements, and the statistics of these events at different stages of crack propagation (i.e. short crack and long crack regimes) are quantified. Challenges and limitations of the methodology will also be discussed.

11:00 AM Break

Wednesday Invited

Wednesday AM | June 30, 2021

Session Chair: L. Zhang, Chinese Academy of Sciences

11:10 AM Invited

Gaining Insights into Complex Dynamics of Coarsening in Bicontinuous Structures: W. Andrews[1]; K. Elder[2]; M. Ziehmer[3]; E. Lilleodden,[3]; P. Voorhees[2]; *K. Thornton*[1]; [1]University of Michigan; [2]Northwestern University; [3]Helmholtz-Zentrum Geesthacht

Bicontinuous structures form frequently in nature as a result of phase separation. These structures continue to evolve due to the excess surface energy toward structures with larger feature sizes. Currently, there is no theory that predicts the morphology resulting from such evolution. In this presentation, the findings from phase field simulations of coarsening in a variety of systems, ranging from those with bulk diffusion to surface diffusion, as well as different volume fractions and mobilities, are summarized. The morphologies of the structures are characterized via interfacial shape distributions and phase/curvature auto-correlations, and their topologies are also quantified. A comparison to nanoporous gold structure is presented, and implications of particle detachment and reattachment are discussed.

11:40 AM Invited
Analysis of Grain Structure and Voids within Additive Manufactured 316L by Serial Sectioning: *D. Rowenhorst*[1]; [1]Naval Research Laboratory
One advantage of serial-sectioning is the ability to analyze large volumes while maintaining a relatively high resolutions. Additionally, there are multiple imaging modes that can be used for each section surface, allowing for more precise segmentation of features. Here we present a method wherein electron backscattered images are used to resolve pore defects in additively manufactured 316L, while Electron Backscattered Diffraction (EBSD) is used to resolve the grain structure. Due to the high-tilt imaging conditions of the EBSD, the spatial distortions were removed by comparing common features within each of the two datasets and aligning the 3D datasets. This then allowed for co-analysis of the data where we show that small grains are highly correlated with lack of fusion pores within the AM material, indicating that the pores may play a significant role as nucleation sites for new grains during AM processing.

Characterization Methods II

Wednesday PM | June 30, 2021

Session Chair: E. Lilleodden, Helmholtz-Zentrum Geesthacht

3D Maps of Geometrically Necessary Dislocation Densities in Polycrystalline IN718: *W. Witzen*[1]; A. Polonsky[1]; T. Pollock[1]; I. Beyerlein[1]; [1]University of California, Santa Barbara
Three dimensional Electron Backscatter Diffraction (3D EBSD) as a method oforientation analysis offers the ability to characterize material microstructure in 3D space, allowing the calculation of Geometrically Necessary Dislocation (GND) densities. Feature reference misorientation within each grain imaged in this dataset reveals that some grains exceed 20 degrees in misorientation to the overall orientation of the grain, leading to the partitioning of subgrains and other areas of high misorientation within single grains. This orientation imaging technique allows GND density mapping in 3D space and provides insight to these subgrain features, particularly useful for additively manufactured material characterization. In this study, GND densities ranging from roughly 1\215 10^{11} to 1.8 \215 10^{13} m^{-2} have been calculated from the high degree of misorientation within these grains. Additionally, GNDs are calculated using different nearest-neighbor environments to understand how disorientation measured over different neighborhood configurations will influence the GND densities calculated.

Poster
3D Non-destructive Characterization of Texture Evolution in Electrical Steels with Laboratory Diffraction Contrast Tomography: *J. Sun*[1]; F. Bachmann[1]; J. Oddershede[1]; E. Lauridsen[1]; [1]Xnovo Technology
Electrical steels with high silicon contents are widely used in electrical power transformers, motors and generators. Texture is the most important property for electrical steel as the orientations of grains have strong influences on the magnetization and electrical resistance of the materials. Laboratory diffraction contrast tomography (LabDCT) is a recently developed X-ray based technique that is able to map the grain morphology and crystallographic orientation non-destructively in 3D. The capability of LabDCT in analyzing the grain structure in electrical steel with significant statistics brings unprecedented insights into the research of electrical steels. In this work, we will present the results of using LabDCT to characterize both non-oriented and oriented electrical steels, with discussion on how this non-destructive 3D technique will contribute to the study of dynamic texture development in electrical steels.

Analysis of Deformation Modes of AZ31 Magnesium Alloys Using EBSD and DCT: *J. Cho*[1]; S. Han[1]; G. Lee[1]; J. Lee[1]; J. Sun[2]; [1]Korea Institute of Materials Science; [2]Xnovo Technology ApS
Various activation of deformation modes of AZ31 magnesium alloys during plane strain compression can be experimentally investigated using electron backscatter diffraction (EBSD) and XRD diffraction contrast tomography (DCT). Activity of the major deformation modes of basal and non-basal slips and twinning during deformation are examined and compared with prediction by crystal plasticity. Three-dimensional microstructural features can be obtained using diffraction contrast tomography. In particular, laboratory diffraction contrast tomography (LabDCT) makes use of high-resolution diffraction images acquired on a ZEISS Xradia Versa X-ray microscope. The capabilities of LabDCT to include full reconstruction of the 3D grain structure including both grain morphology and crystallographic orientation, makes it possible to analyse in detail of structural heterogeneities and texture present in the sample. Effect of spatial distribution and grain boundary character of grain orientations on activity of deformation modes are discussed.

Combined Strain Measurements and 3D Tomography in Inconel 718: *M. Charpagne*[1]; J. Stinville[1]; T. Francis[1]; A. Polonsky[1]; M. Echlin[1]; P. Callahan[2]; V. Valle[3]; T. Pollock[1]; [1]University of California, Santa Barbara; [2]Naval Research Laboratory; [3]Universite de Poitiers
Damage during mechanical loading of polycrystalline metallic alloys involves plastic strain localization at the scale of individual grains. The development of predictive models for monotonic and cyclic loading requires quantitative assessment of these processes at the sub-micron scale. This study aims at understanding strain localization processes in relation to the 3D microstructure in the structural alloy Inconel 718, by combining Digital Image Correlation data acquired in a Scanning Electron Microscope with 3D EBSD data. The use of discontinuity tolerant DIC codes enables to resolve the individual components at each slip band, which when coupled to crystallographic data, enables to determine the active slip system. To do so, multi-modal data merging techniques have been employed to recombine the strain localization information with the 3D grain structure and crystallographic orientations, acquired using TriBeam tomography. Quantitative correlations between microstructure features (grain boundaries, twin boundaries, triple junctions, quadruple points, etc.) will be discussed.

Equations for Grain Boundary Motion in Finite-element Simulations: *E. Eren*[1]; J. Mason[1]; [1]University of California, Davis
Simulating microstructure evolution on a finite element mesh is challenging; some existing formulations do not allow for anisotropic grain boundary properties, have unphysical anisotropy from the underlying numerical model, or allow only a restricted set of topological events that bias the grain boundary network evolution. Our group has been developing a FEM simulation that (1) uses a volumetric mesh to allow the inclusion of arbitrary material physics, (2) significantly expands the set of topological events to allow for general grain boundary network dynamics, and (3) proposes an energy dissipation criterion to identify the physically most plausible of these events. The performance of three proposed equations of motion for grain boundaries is evaluated and compared to known analytical results. The resulting code is expected to improve our ability to use recently-available experimental observations of three-dimensional microstructure evolution to develop predictive simulations.

Large-volume 3D EBSD System and Its Application to the Investigation of Grain Boundary Corrosion in 316L Stainless Steel: *S. Tsai*[1]; P. Konijnenberg[2]; I. Gonzalez[1]; S. Hartke[1]; T. Griffiths[1]; S. Zaefferer[1]; A. Taniyama[3]; K. Kawano-Miyata[3]; [1]Max-Planck-Institut für Eisenforschung GmbH; [2]Bruker Nano GmbH; [3]Research and Development, Nippon Steel Corporation

A large-volume 3D EBSD system consisting of an SEM (Zeiss crossbeam XB 1540) with a dedicated sample docking station, an adapted mechanical polishing automaton (ATM X-Change), and a collaborative robotic arm (Universal Robots UR5) has been assembled. An in-house designed software orchestrates the whole automation process via a communication hub. To obtain accurate serial sections of constant thickness, the polishing parameters (e.g., force exerted on the polishing cloths) were fine-tuned in a large number of preliminary tests on the material of interest. All samples were featured with top-observable side markers for polishing depth measurement. These markers were produced by plasma focused ion beam (PFIB). 3D EBSD data sets with a total volume of 300 × 300 × 300 µm³ reconstructed from approx. 100 slices were acquired to study the grain boundary corrosion behavior of 316L stainless steel. Corrosion tests were applied on the remaining bulk surface.

Poster

Optimizing Laboratory X-ray Diffraction Contrast Tomography for Characterization of the Grain Structure in Pure Iron: *A. Lindkvist*[1]; H. Fang[1]; D. Juul Jensen[1]; Y. Zhang[1]; [1]Technical University of Denmark

Laboratory diffraction contrast tomography (LabDCT) is a recently developed technique for 3D non-destructive grain mapping using laboratory X-rays. The aim of the present work is to quantify the effects of the experimental parameters on the characterization of the 3D grain structure in iron. The experimental parameters studied include accelerating voltage, exposure time, as well as the number of projections. The quality of the grain reconstruction as a function of these parameters is evaluated by comparing the experimental and simulated diffraction patterns. Based on such evaluation, optimized experimental parameters are obtained for the 3D reconstruction of grains with certain sizes. The results of this optimization will be presented, and possible ways to extend the limits of the LabDCT technique will be discussed.

The Isothermal Evolution of Nanoporous Gold (npg) from the Ring Perspective: *M. Ziehmer*[1]; E. Lilleodden[1]; [1]Helmholtz-Zentrum Geesthacht

The interconnected ligament network of npg may be seen as put together by torus-like rings. Similar to the reduction of the number of structural elements in other coarsening phenomena, npg reduces its number of rings during coarsening. Two basic mechanisms appear conceivable, ring collapse and ligament pinch-offs, both being fundamentally different topological transitions. To better understand this aspect of npg microstructure evolution, isothermally annealed samples were decomposed into their relevant rings, applying results and algorithms from graph theory to skeletonized 3D reconstructions from focused ion beam tomography. This approach enabled to analyze distributions of topological classes, referring to number of ring edges, and their evolution over isothermal annealing. The results from five samples show a broadening of these distributions, suggesting an increasing relative dominance of pinch-offs over time. One consequence is a slow but steady increase of the average number of ring edges, that speaks against a self-similar evolution.

Data Processing and Machine Learning I

Wednesday PM | June 30, 2021

Session Chair: P. Voorhees, Northwestern University

Application of Machine Learning to 3D Reconstruction of SOFC Electrodes: *S. Du*[1]; S. Barnett[2]; K. Thornton[1]; [1]University of Michigan; [2]Northwestern University

Solid oxide fuel cells (SOFCs) are one of the promising energy conversion devices with pollution-free and high efficiency under elevated temperature operations. Understanding the source and mechanism of microstructural degradation as well as linking microstructure to performance is vital to optimizing the performance of SOFCs. In recent years, 3D reconstruction is the state-of-the-art approach used in microstructure investigations, within which the image segmentation is the most critical step. The 2D images of SOFC electrodes obtained from FIB-SEM or XCT are grayscale images with unavoidable noises and artifacts, which makes microstructural analyses difficult. In our work, we present an advanced image processing approach to achieve high-quality microstructure reconstruction of a SOFC electrodes enhanced by machine learning techniques. We also generated an artificial dataset with similar noises compared to the experimental data to enable quantification of errors resulting from different algorithms.

Challenges in 3D Correlative Tomography and Microscopy for Materials Science: *B. Winiarski*[1]; R. Geurts[1]; G. Pyka[2]; A. Chirazi[1]; D. Lichau[2]; [1]Thermo Fisher Scientific; [2]Université Catholique de Louvain

Multi-scale/multi-modal correlative methods, which involve the coordinated characterization of materials across a range of length scales using various apparatus, allowing solving broad range of scientific problems previously unreachable by the typical experimental operando. However, developing a repeatable/adaptable protocol for multi-scale/multi-instrument investigations is difficult to bring together. Problems arise as sub-samples are dissected from samples and remounted using various specimen holders needed for different apparatus. These generate 3D data clusters having different imaging modalities with range of voxels sizes and each in its own coordinate system. In this work we address these challenges with new, automated and multi-scale correlative workflows. These correlative methods use metrological X-Ray µCT helical scanner, multi-ion Plasma DualBeam and fs-Laser Plasma TriBeam platforms, cross-platform correlative holder kits and integrated image processing and analyses routines. As the practical example of the correlative solutions we explore microstructural features and imperfections of a glass-fiber reinforced polymer composite for automotive applications.

Characterization of 3-D Slip Fields in Deforming Polycrystals: *D. Pagan*[1]; K. Nygren[1]; M. Miller[2]; [1]Cornell High Energy Synchrotron Source; [2]Cornell University

The interactions of slip systems at grain boundaries have been posited to be a critical factor in stress localization and subsequent nucleation of damage, especially during the dwell fatigue process in titanium alloys. To test these hypotheses, quantitative methods are needed to characterize slip activity in-situ in the bulk of deforming polycrystals. Here we present a new methodology that combines measurements of grain average stresses and lattice orientation fields made using high-energy X-ray diffraction microscopy (HEDM) with crystal plasticity kinematics to reconstruct full 3-dimensional slip activity fields with micron-scale resolution. The utility of the method will be demonstrated through analysis of slip activity in Ti-7Al deformed in uniaxial tension with a focus on analyzing slip mismatch at grain boundaries.

Comprehensive 3D Microstructural Characterization of Nuclear Fuel: *C. McKinney*[1]; A. Aitkaliyeva[1]; [1]University of Florida

In this work, data sets gathered from focused ion beam (FIB) tomography with complementary electron backscatter diffraction (EBSD) and energy dispersive X-ray spectroscopy (EDS) are used to reconstruct the 3D microstructure of irradiated oxide fuels. The incorporation of EBSD and EDS data sets allows for both microstructural and microchemical characterization, which is particularly important for nuclear fuels where harsh reactor environments (temperature, irradiation, pressure) cause microstructural changes. The large thermal gradients produce a non-uniform microstructure across the pellet radius where regions separated by a few millimeters can have grain sizes differing by a factor of 100. When an atom fissions, it splits into different atoms that agglomerate into new phases and precipitates. These fission products can have different properties compared to the fuel, which can alter fuel performance. Comprehensive 3D characterization provides insight into the evolution of these microstructural aspects that could ultimately compromise the safety of the reactor.

Poster

Direct Estimation of Structure Parameters from 3D Image Data without Segmentation: *E. Brenne*[1]; V. Dahl[1]; P. Jørgensen[1]; [1]Denmark Technical University

Materials science based on 3D imaging and quantitative analysis is often impeded by the analysis of the image data rather than their acquisition. Human bias and labor-intensive processing of large datasets are of concern. We present a new model for the distribution of intensity and gradient magnitude values in volumetric datasets such as from X-ray computed tomography. In contrast to conventional statistical models such as the Gaussian mixture model, our model accounts for mixed-material interface voxels stemming from blurring inherent to the imaging technique. This results in an improved model fit based on physical parameters the sample and the imaging system. We demonstrate how the model allows for direct extraction of physical sample parameters, like volume fractions and interface areas, without segmenting the data first. The model has potential to improve segmentation accuracy and reproducibility, and with automated maximum-likelihood model parameter estimation, bias from manual segmentation parameter tuning is avoided.

Poster

Meaningful Learning in Materials Engineering: Learning through Virtual Reality Learning Environments: *L. Davila*[1]; D. Vergara[2]; J. Extremera[3]; M. Rubio[3]; [1]University of California, Merced; [2]Catholic University of Ávila; [3]University of Salamanca

The increasing dissemination of virtual reality learning environments (VRLEs) compels the elucidation of how these didactic tools can improve their effectiveness at the formative level. The motivation generated in students by a VRLE is revealed as a key factor in achieving meaningful learning, but such a motivation alone does not guarantee the long-term retention of knowledge. To identify the necessary characteristics of a VRLE to achieve an appropriate level of meaningful learning, this work compares a set of VRLEs created in previous years with a group of recently developed VRLEs, after being used by students. A description of the design process of the both VRLEs groups is included. Analysis of the response of students in a survey reveals how a protocol system helped improve students' knowledge and retention after a year of using a VRLE. This study demonstrates the importance of using modern engines to achieve long-term retention of knowledge.

Using Distance Metrics to Evaluate 3D RVE Size for Micromechanics and Texture: *R. Lim*[1]; J. Pauza[1]; M. Wilkin[1]; A. Rollett[1]; [1]Carnegie Mellon University

A representative volume element (RVE) is the smallest possible volume element of a material which is statistically representative of the macroscopic properties. The extended time required to capture 3D microstructural data and to run full-field micromechanical simulations drive a need for determination and implementation of RVEs. Far-field high energy x-ray diffraction experiments were carried out to study RVE size vs micromechanical response. These results were supplemented by simulation work on the effect of varying texture on volume element size. Distance metrics (e.g. Hellinger distance) were applied to resolved shear stress distributions throughout the work to evaluate RVE size. The work intends to help improve efficiency in both 3D data acquisition as well as efficiency in performing micromechanical simulations.

Practical Applications

Wednesday PM | June 30, 2021

Session Chair: To Be Announced

3D Characterization of Grain Structure and In-situ Deformation of Open-cell Metal Foam Using Micro-computed Tomography and High-energy X-ray Diffraction Microscopy: Q. Johnson[1]; J. Plumb[2]; P. Kenesei[3]; H. Sharma[3]; J. Park[3]; *A. Spear*[1]; [1]University of Utah; [2]University of California, Santa Barbara; [3]Advanced Photon Source, Argonne National Laboratory

Ultra-low-density foams have complex hierarchical structures that give rise to desirable properties like high strength-to-weight ratio and excellent energy absorption. The sparse, fragile network of struts in open-cell foams makes characterizing sub-strut-scale material structure especially challenging. In this work, 3D grain and precipitate structures are characterized for open-cell aluminum foam using synchrotron characterization techniques. X-ray computed tomography and high-energy X-ray diffraction microscopy data were collected in-situ at interrupted loading intervals during compression. A novel scanning strategy developed at the APS 1-ID beamline enabled complete characterization of a 6%-dense foam sample that was four times larger than the X-ray beam width. A data-analysis procedure was developed to track grains through large strut displacement and deformation. The 3D precipitate maps were used to correlate ligament failure to precipitate distributions. The methods and procedures developed here can be applied to other low-density structures (e.g., AM lattices) and enable new possibilities for high-fidelity modeling.

Poster

In-situ 3D Imaging of Mechanical Failure in TRISO Particles: *S. Kelly*[1]; H. Bale[1]; P. Hosemann[2]; [1]Carl Zeiss Microscopy; [2]University of California

Tri-structural isotropic (TRISO) particles are leading candidates for use as fuel in advanced, high temperature nuclear reactors. They are small (~1 mm diameter) composite spheres containing a nuclear fuel core surrounded by concentric layers of carbon and SiC. Their composite construction allows them to operate at high temperatures with excellent resistance to corrosion, oxidation, and neutron irradiation. The robust containment of the fuel core and fission products depends critically on the integrity of the SiC layer, and the mechanical properties of this layer must be understood for proper fuel particle design and integration into larger scale reactor systems. We present here a 3D in situ investigation into the fracture properties of the SiC layer in a surrogate (non-radioactive core) TRISO particle. We apply a compressive load while simultaneously imaging the 3D microstructure using sub-micrometer x-ray microscopy to visualize crack propagation in the SiC layer.

Possibilities and Limitations of Tomography Methods for the Detailed Analysis of Lithium-ion Battery Cathode Microstructures: J. Joos[1]; *S. Kelly*[2]; T. Volkenandt[2]; S. Freitag[2]; A. Weber[1]; E. Ivers-Tiffée[1]; [1]Institute for Applied Materials (IAM-WET), Karlsruhe Institute of Technology (KIT); [2]Carl Zeiss Microscopy

Deep knowledge of the 3D microstructure is essential for optimizing performance in Lithium ion batteries electrodes. Tomography methods like focused ion beam-secondary electron microscopy (FIB-SEM) and X-ray tomography are the most frequently used 3D characterization techniques for battery electrodes. While each method has advantages and disadvantages, understanding the complex morphology of electrodes requires a complementary, multi-scale 3D approach. In this work, the possibilities and limitations for both 3D techniques, X-ray and FIB-SEM tomography, are studied using the example of a $LiNiCoAlO_2$-$LiCoO_2$ blend cathode from a commercial cell. The same cathode sample was analyzed with both techniques and moreover with different devices to enable a comparison of the obtained data quality. The material fractions, porosity, surface area and tortuosity are calculated from the different data sets and the results are compared with special emphasis on the differentiation of the different active materials and the carbon-binder phase.

Precipitation and Strengthening in AlCoCrFeNi High Entropy Alloys as Studied by Atom Probe Tomography: *K. Knipling*[1]; P. Callahan[1]; D. Beaudry[2]; [1]Naval Research Laboratory; [2]University of Florida

High entropy alloys (HEAs) typically contain five or more principal elements in nearly equiatomic proportions, significantly expanding the composition space and achievable properties of novel metallic materials. In this study we present the complex three-dimensional nanoscale microstructures formed in $Al_{0.5}CoCrFeNi$ (atomic fraction) HEAs in the as-cast state and after thermal aging at 700 and 1000 °C. The alloy solidifies into dendritic regions that have a face-centered cubic (FCC) crystal structure enriched in Co, Cr, and Fe, and interdendritic regions that are comprised of a disordered body-centered cubic (BCC, A2) phase and an ordered BCC phase (B2) formed by spinodal decomposition. During aging these regions form a variety of strengthening precipitates, including NiAl (B2 structure), Ni_3Al ($L1_2$), and a-Cr (BCC). Atomic-scale clustering and ordering is assessed in three dimensions using atom-probe tomography and these experimental results are compared with the equilibrium phases predicted by thermodynamic modeling.

Properties Relied on Internal Structure of Ceramics from 3D View of X-ray Tomography: *L. Zhang*[1]; S. Wang[1]; Z. Wu[1]; L. Cao[1]; [1]Chinese Academy of Sciences

Highly porous ceramic was quantitatively characterized for the morphological parameters within the 3D volume. The size, shape of the pores as well as their distribution and connectivity, can be evaluated for the property of thermal insulation with the supplementary of modeling and analysis. As a bio-inspirited material, sea urchin spine is composed of bio ceramic and organic substance with a hierarchical structure. The complex 3D volume of a sampled structure was digitalized with the XRT techniques. The meshed 3D volume can be input Finite Element Analyses (FEA) for mechanical test with the 3D global scale. Compression of the real structure model revealed that the stress concentrates along the dense growth rings and dissipates through the strut structures. It implied that the rationally constructed hierarchical structure in nature play an important role in high strength-to-weight property. Its mechanical property might be optimized for potential applications of bone defect repair.

Revealing Complex Materials Structure in Dealloying and Energy Storage by Synchrotron X-ray Nano-tomography: *Y. Chen-Wiegart*[1]; [1]Stony Brook University

Synchrotron X-ray nano-tomography with operando analysis and chemical sensitivity is powerful to study complex structures. We will discuss two types of such systems – porous metals fabricated by dealloying and energy storage materials in batteries. Dealloying, a selective etching process, can fabricate a variety of nanoporous metals with a characteristic bi-continuous structure with promising applications in catalysis, energy storage and bio-sensing. Our work includes addressing the processing-structure correlation in different dealloying systems, from solid-state interfacial dealloying, chemical dealloying, molten salt corrosion, and vapor phase dealloying. We utilized both full-field and scanning techniques. In addition, by combining X-ray imaging with other modalities, we applied a multimodal approach to address the complex chemical, morphological and structural evolution in energy storage materials. Novel systems with nanoporous electrodes, beyond-lithium batteries and aqueous batteries, will be discussed. These works highlight how X-ray nano-tomography methods can advance our understanding in complex materials for future materials design.

Three-dimensional Characterization and Modelling of Cyclic Deformation in Magnesium Alloys by High-energy X-ray Diffraction Microscopy: *D. Greeley*[1]; M. Yaghoobi[1]; D. Pagan[2]; V. Sundararaghavan[1]; J. Allison[1]; [1]University of Michigan; [2]Cornell High Energy Synchrotron Source

Fatigue behavior is a primary concern for lightweight structural components. To accurately predict the effect of microstructure and composition on low-cycle fatigue in rare-earth magnesium alloys, it is necessary to understand the effect of texture and grain size on micromechanical evolution during fatigue cycling. High-energy X-ray diffraction microscopy (HEDM) was utilized to analyze the three-dimensional character of deformation during a fully reversed tension-compression cycle in coarse and fine-grain size Mg-2.4wt.%Nd. Grain average strains in each microstructure were tracked in-situ with far-field HEDM. Slip activity and localization of strain were modelled with the PRISMS-Plasticity crystal plasticity finite element software using unloaded 3D grain morphologies and grain orientations characterized using near-field HEDM. The results of these crystal plasticity predictions are compared to far-field measurements. The dataset from this study is published publicly in the Center for Predictive Integrated Materials Science (PRISMS) Materials Commons data repository.

Thursday Plenary

Thursday PM | July 1, 2021

Session Chair: M. Miller, Cornell University

12:00 PM Plenary
Operando and *Insitu* Synchrotron Experiments Following Microstructural Evolutions: *H. Van Swygenhoven*[1]; [1]Paul Scherrer Institute & École Polytechnique Fédérale de Lausanne
Thanks to the high brilliance and the fast detectors available at synchrotrons, operando and insitu experiments have become possible, adding the timescale to microstructural characterization techniques. Such experiments allow identification of operating mechanisms that are responsible for microstructural evolutions. This talk will demonstrate the potential of such experiments. Addressing microstructures of superelastic NiTi alloys, insitu xray diffraction experiments provide understanding why some microstructures can accommodate for changing strain paths, and why degradation during fatigue loading depends strongly on the strain path (Acta Materialia 144(2018)68 and 167(2019)149) . Addressing 3D printing, operando selected laser melting of Ti6Al4V during xray diffraction, allows to track with a time resolution of 50µs the dynamics of the á and â phases during fast heating and solidification, providing the cooling rates of each phase and the duration the â phase exists during processing. Such an experiment reveals for instance the link between the cooling rate, the scan vector length and the resulting microstructure (Materials Today, November 2019).This research is performed within the ERC Advanced Grant MULTIAX (339245), PREAMPA and the CCMX-AM3 challenge projects financed by the ETH board and the Swiss watch and precious metal industry.

1:00 PM Break

Thursday Invited

Thursday PM | July 1, 2021

Session Chair: E. Lilleodden, Helmholtz-Zentrum Geesthacht

1:10 PM Invited
New Opportunities in Scanning Intragranular Deformation at the Materials Science Beamline at the ESRF: P. Reischig[1]; W. Ludwig[2]; *J. Wright*[3]; [1]InnoCryst Ltd, European Synchrotron Radiation Facility; [2]Université de Lyon, European Synchrotron Radiation Facility; [3]European Synchrotron Radiation Facility
The current ESRF source upgrade is delivering an order of magnitude higher photon flux for the Materials Science beamline, ID11, and, together with newly installed state-of-the-art detectors and control software, this will greatly enhance polycrystal diffraction imaging performance. The "nanoscope" instrument provides a 100nm to 1µm size monochromatic point or line focus beams for single crystal, powder and PDF measurements. Grain boundary and intragranular strain scanning using an adaptive tomographic reconstruction have been demonstrated at submicron spatial and ~10-4 strain resolution. The "3D-XRD microscope" in the same hutch offers point, line or box beam illumination for 3D-XRD to obtain grain average properties in the far-field, and grain boundaries and sub-grain properties in the near-field (via Diffraction Contrast Tomography and Topo-Tomography), as well as phase- and absorption contrast imaging. Novel hardware calibration and reconstruction methods are increasing the sensitivity to intragranular misorientation and strain in the near-field for in-situ studies.

1:40 PM Invited
Next Generation TriBeam Systems and 3D Data Workflows: *M. Echlin*[1]; A. Polonsky[1]; T. Francis[1]; W. Lenthe[2]; M. Charpagne[1]; J. Stinville[1]; S. Randolph[3]; J. Filevich[3]; A. Botman[3]; R. Geurts[3]; M. Straw[4]; B. Manjunath[1]; M. De Graef[2]; T. Pollock[1]; [1]University of California, Santa Barbara; [2]Carnegie Mellon University; [3]Thermo Fisher Scientific; [4]Applied Physics Technologies
TriBeam experiments have proven to be a path forward for cubic-mm scaled, micron resolution, 3D microanalytical data acquisition. In this talk we review new hardware developments that have enabled low damage laser ablation over larger (>1.5mm) fields of view, with high current (µA) Xenon plasma FIB for surface cleanup if necessary, and EBSD detectors than can capture 3000-5000 fps with greater electron sensitivity than existing systems. Sub-micron layers of material are laser ablated using the standard microscope stages and pre-tilt specimen holders. Data acquisition times have decreased with this new hardware, and the balance between reasonable acquisition times, imaging modalities, and data volumes are presented. Raw EBSD patterns are processed systematically, in parallel, using the spherical indexing method (EMSphInx) running on a co-located high performance computing center. Discussion of the advancements in the 3D data acquisition workflow, reconstruction and analysis software, and the collaborative data infrastructure BisQue will be made.

Data Processing and Machine Learning II

Thursday PM | July 1, 2021

Session Chair: M. Echlin, University of California, Santa Barbara

A Surface-mesh Gradation Tool for Generating Optimized Tetrahedral Meshes of Microstructures with Defects: *B. Phung*[1]; J. He[1]; A. Spear[1]; [1]University of Utah
A meshing tool for generating tetrahedral meshes of polycrystalline microstructures with an optimal element-size distribution, determined by underlying defects (e.g., cracks and voids), is presented. The meshing tool provides users the capability to tune the element-size distribution by specifying parameters such as desired edge lengths and refinement zone radii around defects. Surface meshes can be obtained from 3D image data generated from experiments or tools such as DREAM.3D. Remeshing is performed directly on the original surface mesh via edge splitting and collapsing operations. The modified surface mesh is used in conjunction with a volume mesher to create a gradated volume mesh optimized for the defect(s). Relative to an unmodified uniform mesh, the framework is shown to significantly reduce element count and improve computational efficiency while ensuring sufficient mesh refinement in regions with high gradients. The meshing capabilities are demonstrated with examples involving crack propagation within polycrystalline microstructures.

Advanced Acquisition Strategies for Laboratory Diffraction Contrast Tomography (LabDCT): *J. Oddershede*[1]; J. Sun[1]; F. Bachmann[1]; E. Lauridsen[1]; [1]Xnovo Technology
Imaging the three-dimensional grain microstructure of polycrystalline material nondestructively is key to better understanding of the material performance. Recent advances of Laboratory Diffraction Contrast Tomography (LabDCT) allow to record and reconstruct larger representative volumes seamlessly. We will present and discuss different acquisition strategies with emphasis on how to approach a given acquisition problem inherent to the sample. Combining the 3D grain microstructure, i.e. grain morphology and crystallographic orientation, together with traditional absorption contrast tomography gives unprecedented insights into materials structure. Time resolved studies of the response of the material under investigation to external stimuli in- or ex-situ can be conducted.

Automatic Segmentation of 3D Microstructures of Steel Using Machine Learning Methods: *H. Kim*[1]; Y. Arisato[1]; J. Inoue[1]; [1]The University of Tokyo

Microstructure greatly influences mechanical and chemical properties, so many endeavors have been made to characterize it. Especially, 3D microstructural characterization of steel is very challenging due to the existence of various phases as well as the high cost for delicate 3D observations. Recently, researches using machine learning methods have shown good performances in classifying 3D microstructures. However, those approaches are generally based on supervised learning algorithms that require preparation of labeled datasets, which is not only time-consuming but also difficult even for experts. In this study, we propose an unsupervised algorithm that performs 3D microstructure segmentation without the need for labeled datasets. The new method, which is a combination of feature extraction with filters for texture analysis and a clustering algorithm, is applied to a 3D image obtained with a serial sectioning technique and optical microscope. The segmentation result shows that 3D microstructures are well divided into constituent phases.

Formation and Annihilation of Thin Twins in Hexagonal Close Packed Materials: *H. Abdolvand*[1]; K. Louca[1]; C. Mareau[2]; M. Majkut[3]; J. Wright[3]; [1]The University of Western Ontario; [2]Arts et Metiers ParisTech; [3]European Synchrotron Radiation Facility

Slip and twinning contribute significantly to plastic deformation in hexagonal close packed (HCP) materials. In this study, zirconium and magnesium specimens with HCP crystals are deformed in-situ to study formation and annihilation of twins using three-dimensional synchrotron X-ray diffraction. Methods are developed for accurate determination of grain properties, results of which are compared to those from electron backscatter diffraction measurements. New methods are developed to find the twins that form with loading and determine their corresponding parents. The measured initial microstructures are mapped into crystal plasticity models to study the evolution of grain resolved stresses. It is shown that twins are stressed when they are thin but relax with further loading. While a sign reversal is observed for the resolved shear stress (RSS) acting on the twin habit plane in the parent, the sign of RSS within the majority of twins stays unchanged until twin annihilation during the load reversal.

Predicting Microstructure-dependent Mechanical Properties in Additively Manufactured Metals Using Machine- and Deep-learning Methods: C. Herriott[1]; *A. Spear*[1]; [1]University of Utah

The efficacy of machine-learning (ML) and deep-learning (DL) models to predict microstructure-sensitive mechanical properties in metal additive manufacturing (AM) is assessed using results from high-fidelity, multi-physics simulations as training data. Build domains exhibiting vastly different microstructures of AM SS316L were generated using the physics-based framework. Microstructural subvolumes and corresponding homogenized yield-strength values (~7700 data points) were then used to train two types of ML models (Ridge regression and XGBoost) and one type of DL model (convolutional neural network, CNN). The ML models require substantial pre-processing to extract volume-averaged microstructural descriptors; whereas, 3D image data comprising basic microstructural information are input to the CNN models. Among all models tested, the CNN models that use crystal orientation as input provide the best predictions, require little pre-processing, and predict spatial-property maps in a matter of seconds. Results demonstrate that suitably trained data-driven models can complement physics-driven modeling by massively expediting structure-property predictions.

Reconstruction of Microstructure and Defects in an Alpha+Beta Processed Ti-6Al-4V Plate Product Using High-energy X-ray Diffraction Microscopy and DREAM.3D: *K. Stopka*[1]; J. Park[2]; H. Sharma[2]; A. Chuang[2]; P. Kenesei[2]; Y. Gao[3]; T. Broderick[4]; W. Musinski[4]; P. Shade[4]; S. Donegan[4]; M. Jackson[5]; J. Almer[2]; D. McDowell[1]; [1]Georgia Institute of Technology; [2]Argonne National Laboratory; [3]GE Global Research Center; [4]U.S. Air Force Research Laboratory; [5]BlueQuartz Software LLC

In situ near-field (NF) and far-field (FF) high-energy x-ray diffraction microscopy (HEDM) and micro-computed tomography (μCT) were conducted on an alpha+beta processed Ti-6Al-4V specimen subject to cyclic loading to non-destructively characterize the evolution of microstructure associated with short fatigue crack growth (SFCG) and micromechanical response. A novel reconstruction and data fusion approach was developed to obtain a detailed 3D microstructure using DREAM.3D; the NF-HEDM was used to reconstruct the alpha grains while μCT distinguished the alpha and beta regions in the volume of interest. Combined with appropriate boundary conditions and specification of initial micromechanical state, the ensuing virtual microstructure will be used in micromechanical simulations. These simulation results can be compared to the FF-HEDM data to understand the SFCG process in Ti-6Al-4V.

Poster

Supervised Texture-based Classification for 3DEM: *A. Hall*[1]; R. Blanc[1]; J. Caplan[2]; M. Muniswamy[3]; [1]Thermo Fisher Scientific; [2]University of Delaware; [3]University of Texas Health, San Antonio

Dense 3DEM data recovered by Serial Block Face Imaging (SBFI), Serial Section Transmission Electron Microscopy (ssTEM), and Focused Ion Beam Scanning Electron Microscopy (FIB-SEM) present challenging image segmentation needs. Traditional image processing may struggle to faithfully follow features. The variance in electron density between materials provides ample signal for machine learning methods, though. Here, we demonstrate supervised image classification based on image texture as implemented in Amira-Avizo Software. One provides training data from manual segmentation or automated methods. The program then trains a classifier using two statistical categories: features based on co-occurrence matrices and features based on intensity statistics. We tested our implementation on a 10 Gb SBFI mouse heart tissue block collected using an Apreo VolumeScope SEM. When followed by a small amount of image processing, the texture-supervised classification was able to fully label this data set with less than 10 minutes of user effort.

Process Microstructure Properties II

Thursday PM | July 1, 2021

Session Chair: Y. Zhang, Technical University of Denmark

Investigation of Grain Boundary Behavior Using Crystal Based Finite Element Method on a 3D Characterized Oligocrystal Aluminum: *E. Demir*[1]; [1]Sabanci University
Grain boundary behavior has been studied up to date due to its complex behavior; acts as a sink or a source, low-angle grain boundaries allowing dislocation transmission, etc. In this study a columnar-like aluminum sample with large grain sizes reaching over millimeters was fully characterized by using EBSD technique from the two opposite side surfaces. The sample was tensile tested and strains were measured using DIC method. To better understand grain boundary effects, crystal-based finite element models with three different constitutive laws were developed including a phenomenological model, dislocation density based model and a non-local flux based dislocation density model. The amount of grain boundary strengthening was found to be a function of the amount of slip inside grains, misorientation between the grains, and grain boundary normal. Non-local dislocation flux based model revealed significantly better strain distributions near the grain boundary, even though the sample scale was relatively large.

Microstructures and Properties of As-Cast $Al_{2.7}CrFeMnV$, $Al_{2.7}CrFeTiV$, and $Al_{2.7}CrMnTiV$ High Entropy Alloys: *K. Knipling*[1]; P. Callahan[1]; R. Michi[2]; D. Beaudry[3]; [1]Naval Research Laboratory; [2]Northwestern University; [3]University of Florida
High entropy alloys (HEAs) typically contain five or more principal elements in nearly equiatomic proportions, significantly expanding the composition space and achievable properties of novel metallic materials. Here we present the complex microstructures of as-cast $Al_{2.7}CrFeMnV$, $Al_{2.7}CrFeTiV$, and $Al_{2.7}CrMnTiV$ HEAs. All alloys are predominantly body-centered cubic (BCC). $Al_{2.7}CrFeMnV$ is single-phase BCC with lattice parameter (a) of 0.2968 nm. $Al_{2.7}CrFeTiV$ is multiphase, with a high volume fraction of Al-rich G-phase precipitates and Laves phases in a BCC matrix (a = 0.3006 nm). $Al_{2.7}CrMnTiV$ contains micron-scale AlTi ($L1_0$) precipitates as well as coherent, nanoscale cuboid B2 precipitates in a BCC matrix (a = 0.3946 nm). In each of the alloys, atom-probe tomography is used to visualize the nanoscale microstructures in three dimensions and assess atomic-scale clustering and ordering. These experimental results are compared with the equilibrium phases predicted by thermodynamic modeling.

Investigation of Microstructural Evolution during Compression of Ni Foam Using Micro-CT and FEM: *J. Cho*[1]; J. Lee[1]; G. Lee[1]; J. Rha[1]; [1]Korea Institute of Materials Science
Metal foams have been widely used in many applications ranging from weight-saving structures and heat exchange to functional applications, due to low density, high ratio of weight to strength, and large surface area. In this study, mechanical responses and microstructural evolution of porous Ni with approximately 92% porosity are investigated using micro-CT and finite element method. During uni-axial compression, flow curves consist of linear elastic, quasi-plateau and densification stages. Using micro-CT, three-dimensional model was reconstructed and was used for FE modeling of compression. Stress relaxation observed near transition between linear elastic and quasi-plateau stages was associated with buckling of narrow ligament structures. Thick ligaments revealed high stress distribution and few buckling. Similar pore distribution but thicker ligament resulted in decrease in porosity and little stress relaxation. With increase in porosity, energy absorption decreased but ideal efficiency of energy absorption increased. The stress relaxation somewhat improved ideal efficiency of energy absorption.

OOF: Modular, Extensible Finite Element Analysis for Materials Science: *A. Reid*[1]; [1]National Institute of Standards and Technology
The OOF object-oriented finite element software, developed at the National Institute of Standards and Technology, provides an interactive finite-element modeling tool which packages sophisticated mathematical capabilities with a user-interface that speaks the language of materials science. Users can construct finite-element meshes directly from material images in either 2D or 3D, and then perform virtual experiments to explore structure-property relationships within the microstructure, including effective properties. The 3D version of the software has recently been extended to include crystal plasticity, demonstrating the flexibility of the software architecture. The development team is also interested in integrating this software into emerging data-driven analytical and modeling workflows, ingesting constitutive data from on-line materials data repositories, with the ultimate aim of particpating in a closed materials optimization loop.

Simulation and Modelling

Thursday PM | July 1, 2021

Session Chair: J. El-Awady, Johns Hopkins University

Application of ICME towards Thin Wall Casting Technology Development: *J. Shah*[1]; [1]PDA LLC
Casting process modeling of filling and solidification has been in use by OEMs and casting producers for over two decades and the ICME models have matured over the recent past for the most of common alloys used. For the design and development of thin wall casting configurations, it is critical to evaluate the impact of higher cooling rates due to thinner walls on the micro-structure and mechanical properties in the entire component configuration in 3D. The presentation will share the ICME work conducted on new alloys from projects funded by LIFT (Lightweight Innovations for Tomorrow, a Manufacturing USA public-private partnership) towards the development of thin wall super vacuum high pressure die casting aluminum and sand cast iron casting technologies. The key aspects of ICME model integration for 3D visualization, calibration, verification and validations will be presented with case studies.

From 4D Images to Grain Boundary Properties: *J. Zhang*[1]; W. Ludwig[2]; Y. Zhang[3]; H. Sørensen[3]; D. Rowenhorst[4]; A. Yamana[5]; P. Voorhees[1]; H. Poulsen[3]; [1]Northwestern University; [2]European Synchrotron Radiation Facility; [3]Technical University of Denmark; [4]The US Naval Research Laboratory; [5]Tokyo University of Agriculture and Technology
We developed a rapid throughput method to measure grain boundary properties by comparing a time-resolved x-ray experiment of grain growth to phase-field simulations. Grain evolution in pure iron is determined in three-dimensions and as a function of time with diffraction contrast tomography. Using a time step from the experiment as initial condition, the simulated microstructure is compared with the experimental one at a later time step. An optimization algorithm is used to find the reduced grain boundary mobilities that yield the best match between the experiment and the simulation. Using the proposed method, 10,307 reduced mobilities of 1,619 boundaries are determined from a bulk polycrystalline material without measuring the local boundary curvatures and velocities. We find that in general there is no correlation of grain boundary mobility with boundary inclination or misorientation.

Micromechanical Finite Element (FE) Modeling in Conjunction with Alloy Development Integrated Computational Materials Engineering (ICME) Framework: *S. Schwarm*[1]; P. Lambert[1]; E. Antillon[2]; C. Stewart[3]; D. Bechetti[1]; M. Draper[1]; C. Fisher[1]; [1]Naval Surface Warfare Center Carderock Division; [2]Naval Research Laboratory; [3]National Research Council Postdoctoral Associate at the U.S. Naval Research Laboratory

Modern computational tools across a range of applications use Integrated Computational Materials Engineering (ICME) techniques to provide a functionally robust framework for the creation of new structural metals where physical iteration can be reduced by predictive modeling. Finite element (FE) methods can be applied to realistic microstructures on different length scales to predict the influence of microstructural parameters on mechanical performance. In combination with thermodynamic and kinetic modeling tools, micromechanical FE modeling enable early and frequent vetting of composition and process variables on properties such as yield strength. Building a robust FE model depends on multiple variables, including use of simulated or experimentally observed 3D microstructure geometries, identification of valid single-phase material properties, utilization of functional homogenization schemes, and application of boundary conditions. FE modeling methodologies utilized within a novel high-strength austenitic steel project are discussed here along with their integration into a greater ICME alloy development framework.

PRISMS-Plasticity: An Open-source Crystal Plasticity Finite Element: *M. Yaghoobi*[1]; S. Ganesan[1]; S. Sundar[1]; A. Lakshmanan[1]; A. Murphy-Leonard[2]; D. Greeley[1]; S. Rudraraju[3]; J. Allison[1]; V. Sundararaghavan[1]; [1]University of Michigan; [2]University of Michigan; Naval Research Laboratory; [3]University of Michigan; University of Wisconsin, Madison

An open-source parallel 3-D crystal plasticity finite element (CPFE) software package PRISMS-Plasticity is presented here as a part of an overarching PRISMS Center integrated framework. Highly efficient rate-independent and rate-dependent crystal plasticity algorithms are implemented. Additionally, a new twinning-detwinning mechanism is incorporated into the framework based on an integration point sensitive scheme. The integration of the software as a part of the PRISMS Center framework is demonstrated. This integration includes well-defined pipelines for use of PRISMS-Plasticity software with experimental characterization techniques such as electron backscatter diffraction (EBSD), Digital Image Analysis (DIC) and high-energy synchrotron X-ray diffraction (HEDM), where appropriate these pipelines use popular open source software packages DREAM.3D and Neper. In addition, integration of the PRISMS-Plasticity results with the PRISMS Center information repository, the Materials Commons, will be presented. The parallel performance of the software demonstrates that it scales exceptionally well for large problems running on hundreds of processors.

The AFRL AM Modeling Challenge: Predicting Micromechanical Fields in AM IN625 Using an FFT-based Method with Direct Input from a 3D Microstructural Image: *C. Cocke*[1]; A. Rollett[2]; R. Lebensohn[3]; A. Spear[1]; [1]University of Utah; [2]Carnegie Mellon University; [3]Los Alamos National Laboratory

The efficacy of an elasto-viscoplastic fast Fourier transform (EVPFFT) code was assessed based on blind predictions of micromechanical fields in an experimentally characterized sample of IN625 produced with additive manufacturing (AM). Blind predictions were made in the context of the AFRL AM Modeling Challenge Series. Challenge 4 in the Series required predictions of grain-averaged elastic strain tensors for target grains at six specific loading states given a 3D microstructural image, initial elastic strains of target grains, and macroscopic stress-strain response. Among all participants, our EVPFFT-based submission achieved the lowest total error in comparison to experimental results. Post-challenge investigation revealed that predictions were improved by initializing the elastic strain field through eigenstrains calculated by an Eshelby approximation based on ellipsoidal grain shape rather than assuming fully spherical grains. Lessons learned for predicting full-field micromechanical response using the EVPFFT modeling method will be discussed.

The PRISMS Framework: An Integrated Open-source Multi-scale 3D Capability for Accelerated Predictive Materials Science: *J. Allison*[1]; [1]University of Michigan

The Center for PRedictive Integrated Structural Materials Science (PRISMS) is creating a unique framework for accelerated predictive materials science. 3D simulations and experimental information play an important role in this framework. There are three key elements of the PRISMS framework. This first is a suite of high performance, open-source integrated multi-scale computational tools for predicting 3D microstructural evolution and mechanical behavior of structural metals. Specific modules include real-space DFT, statistical mechanics, phase field and crystal plasticity simulation codes. The second is The Materials Commons, a knowledge repository and virtual collaboration space for archiving and disseminating 3D information. The third element is a set of integrated scientific "Use Cases" in which these computational methods are tightly linked with advanced 3D experimental methods to demonstrate the ability of the PRISMS framework for improving our predictive understanding of magnesium alloys, in particular the influence of microstructure on monotonic and cyclic mechanical behavior.

The PRISMS-PF Open-source High-performance Framework for Phase-field Modeling and Its Application to Simulating 3D Microstructure Dynamics: *D. Montiel*[1]; S. DeWitt[1]; K. Thornton[1]; [1]University of Michigan

Over the past few decades, the phase-field model has become a widely-applied method for simulating microstructure dynamics during materials processing and operation. Given the need for simulating a wide variety of increasingly complex structures in three dimensions, general-purpose frameworks with high performance and scalability have received increasing attention compared to traditional single-purpose codes. We present an overview of PRISMS-PF, an open-source high-performance phase-field modeling framework developed within the PRISMS Center at the University of Michigan. The use of a matrix-free finite element method with high-order elements, adaptive meshing, and multilevel parallelism in PRISMS-PF allows for strong computational performance in large 3D phase-field simulations. We showcase the capability of PRISMS-PF to simulate different microstructure evolution phenomena in 3D. The PRISMS-PF framework is part of a larger platform within the PRISMS Center and designed to be integrated with other computational codes, experiments, and the Materials Commons repository for data sharing and collaboration.

A

B

C

D

E

F

G

H

I

J

K

L

M