APS Workshop 6: Elucidating 3D Microstructures through Diffraction-based Imaging and Simulations

Wednesday, May 8, Morning

8:30 – 9:00 Jun-Sang Park (Argonne National Laboratory)
Opening Remarks
An Overview of Microstructure Characterization Techniques Using High-energy X-rays after the Advanced Photon Source-Upgrade (APS-U)

9:00 – 9:30 Hemant Sharma (Argonne National Laboratory)
MIDAS: A Tool for HEDM Data Analysis

9:30 – 10:00 Weijian Zheng (Argonne National Laboratory)
Rapid Detection of Rare Events from In-situ *X-ray Diffraction Data Using Machine Learning*

10:00 – 10:20 Break

10:20 – 10:50 Stephan Hruszkewycz (Argonne National Laboratory)
Pushing the High Energy Frontier of Bragg Coherent Diffraction Imaging Experiments of Materials

10:50 – 11:20 Laura Vietz (University of Utah)
Characterizing Open-cell Metal Foams Using High-fidelity Models and In-situ *High-energy X-ray Experiments*

11:20 – 11:50 Wenxi Li (University of Michigan)
The Current and Future Status of Point-focused High-energy Diffraction Microscopy

11:50 – 1:00 Lunch Break

Wednesday, May 8, Afternoon

1:00 – 1:30 Darren Pagan (Pennsylvania State University)
Opening Remarks
Introduction to Diffraction Microstructure Imaging Data Post-processing

1:30 – 2:00 Marm Dixit (Oak Ridge National Laboratory)
Grain-level Chemo-mechanics and Polymorphism in Garnet Solid Electrolytes

2:00 – 3:00 Matthew Kasemer, Ezra Mengiste, and Amit Singh (University of Alabama)
Neper and FEPX: Application of Open-source Polycrystal Generation, Deformation Simulation, and Post-processing Software to HEDM Experiments

3:00 – 3:20 Break

3:20 – 4:20 Michael Sangid, Krzysztof Stopka, and Kyle Jung (Purdue University)
Scientific Examples and Demonstration of Fast Fourier Transform-based Simulations with X-ray Experiments to Explore Deformation and Fatigue in Structural Alloys

4:20 – 4:50 Ryan Hurley (Johns Hopkins University)
In-situ *X-ray Tomography and High-energy Diffraction Microscopy for Sand, Rocks, and Concrete: Examples and Challenges*

4:50 – 5:00 Break

5:00 – 5:30 Darren Pagan (Pennsylvania State University)
Discussion and Closeout

5:30 Adjourn

An Overview of Microstructure Characterization Techniques Using High-energy X-rays after the Advanced Photon Source-Upgrade (APS-U)

Jun-Sang Park[1], Jonathan Almer[1], Peter Kenesei[1], Chihpin Andrew Chuang[1], Leighanne Gallington[1], John Okasinski[1], Hemant Sharma[1], and Sarvjit Shastri[1]

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The Advanced Photon Source has been a world leader in developing novel microstructure characterization techniques that use high-energy (> 50 keV) x-rays to investigate materials in their bulk form. In the past decade, several tomographic techniques using absorbed or scattered beam have been realized to obtain a 3D view of a sample. These advanced characterization techniques have been combined with unique thermo-mechanical sample environments to provide an unprecedented view into how the microstructure evolves with external stimulus. For instance, a suite of grain-resolving techniques such as high-energy diffraction microscopy can non-destructively provide 3D information – individual grain orientation, location, morphology, and grain-averaged strain – about a polycrystalline sample with thousands of grains in the illuminated volume. Scattering tomography technique has been an important tool for characterizing the local microstructure variation in nanocrystalline materials. The 3D information extracted from these techniques have been instrumental in material model development and validation at the mesoscale.

The APS-U project presents an exciting opportunity to significantly improve the spatial and temporal resolution of these mapping techniques with enhanced x-ray beam coherence and brilliance. For instance, the newly constructed High Energy X-ray Microscope (HEXM) beamline will push the spatial resolution of HEDM into sub-micrometer range. Real-time zoom-in and zoom-out capabilities to obtain a more detailed map of the local microstructure will be realized through novel hardware design exploiting state-of-the-art fabrication technology and advanced controls and software and workflows leveraging high-performance computing and machine learning.

MIDAS: A Tool for HEDM Data Analysis

Hemant Sharma[1]

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I will cover recent advancements to MIDAS, a software package for HEDM data analysis. This will include the registration of multimodal data, including Far-Field HEDM, Near-Field HEDM, and micro-computed tomography. Further, I will show improvements in data storage and retrieval for high-throughput analysis workflows. I/O and compression benchmarks will be shown to demonstrate APS-U advances.

Rapid Detection of Rare Events from *In-situ* X-ray Diffraction Data Using Machine Learning

Weijian Zheng[1], Jun-Sang Park[2], Peter Kenesei[2], Ahsan Ali[1], Zhengchun Liu[1], Ian Foster[1], Nicholas Schwarz[2], Rajkumar Kettimuthu[1], Antonino Miceli[2], and Hemant Sharma[2]

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High-energy x-ray diffraction methods can non-destructively map the 3D microstructure and associated attributes of metallic polycrystalline materials without causing destruction. These techniques, often enhanced by external stimuli such as thermo-mechanical loading, allow for the monitoring of material attribute evolution over time. However, the large volume of data and the high costs associated with standard data gathering and processing techniques have been major obstacles in obtaining fast, actionable insights and in increasing the temporal resolution of these observations. To overcome these challenges, we present a novel, fully automated method that significantly speeds up the detection of plasticity onset in high-energy x-ray microscopy data. Our approach offers processing speeds up to 50 times faster. Utilizing self-supervised image representation learning alongside clustering algorithms, this method condenses extensive data into compact, semantically-rich summaries that spotlight essential features, such as peak shapes. These features quickly identify critical changes, like alterations in diffraction peak shapes, indicative of plasticity onset. Our method can facilitate the generation of timely, actionable information for the design of more intelligent experimental setups. It promises to optimize the use of multimodal x-ray diffraction techniques, potentially transforming experimental strategies in materials science. This presentation will explore the methodology behind this groundbreaking method, its application potential, and our plans for future work in experiment steering utilizing our method.

Pushing the High Energy Frontier of Bragg Coherent Diffraction Imaging Experiments of Materials

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Recent progress in 3D coherent x-ray diffraction imaging methods can enable high resolution structural imaging of nano-structured crystalline materials. By performing experiments at a crystalline Bragg peak, the resulting images are exquisitely sensitive to crystalline defects (i.e., strain, dislocations) that often play critical roles in materials performance. Furthermore, the use of hard x-rays allows for the design of imaging experiments of nanoscale crystalline volumes within difficult-to-access environments such as synthesis chambers or fully encapsulated grains within a polycrystal. Experiments of this nature fall into two categories: 1) Bragg coherent diffraction imaging (BCDI) for imaging sub-beam-sized crystallites, and 2) Bragg ptychography (BP) for imaging specific sub-volumes of an otherwise continuous crystal. Both approaches will be discussed in the context of the vast improvements in coherent flux at high x-ray energies coming with the APS-U and in terms of integration with high energy diffraction microscopy, which is now poised to leverage x-ray beam coherence to provide intragrain strain information.

Characterizing Open-cell Metal Foams Using High-fidelity Models and *In-situ* High-energy X-ray Experiments

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Open-cell metal foams are hierarchical structural-material systems that have applications as light-weight impact absorbers, noise insulators, biomedical implants, and heat sinks, to name a few. This talk will highlight two recent studies in which we investigate the effect of grain structure on compressive mechanical response of open-cell metal foam using both numerical modeling and *in-situ* grain-scale characterization at the Advanced Photon Source (APS). In the numerical study, multiple polycrystalline instantiations (overlaid on a foam volume derived from x-ray tomography) are simulated using crystal-plasticity finite-element modeling to quantify grain-size effect on the global compressive response of investment-cast aluminum foam. The high-fidelity numerical framework captures the deformation mechanisms across multiple length scales and is able to predict the inhomogeneous grain-to-continuum compressive response in the foams. Also, by incorporating grain-boundary strengthening and free-surface softening mechanisms, the framework accounts simultaneously for the Hall-Petch effect in polycrystalline alloys and the effect of unconstrained slip-based deformation at the strut free surfaces. Results from the numerical simulations provide new insights into the mechanical behavior of open-cell metal foams and are used to enhance the Gibson-Ashby model for predicting plastic collapse strength. In a parallel effort, 3D grain and precipitate structures are characterized for an open-cell aluminum foam using synchrotron characterization techniques. X-ray tomography and high-energy x-ray diffraction microscopy (HEDM) 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 for both studies can be applied to other low-density structures (e.g., AM lattices) and enable new possibilities for investigating the micromechanical failure mechanisms of open-cell metal foams and lattices.

The Current and Future Status of Point-focused High-energy Diffraction Microscopy

Wenxi Li[1,2], Hemant Sharma[3], Peter Kenesei[3], Jonathan Wright[4], Sidharth Ravi[5], Huseyin Sehitoglu[5], and Ashley Bucsek[1,2]

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Point-focused high-energy diffraction microscopy (pf-HEDM) offers the ability to map orientation and elastic strain across polycrystalline grain networks with spatial resolutions that are 100× greater than those of conventional 3D multigrain characterization techniques (from ~10 μm to ~100 nm). The result is the ability to study intragranular stresses and orientation changes across mm-sized 3D grain networks. Here, we will present *in-situ* pf-HEDM experiments at the APS (1-ID-E) and the ESRF (ID11) on intragranular deformation in coarse-grained titanium, and on grain growth in fine-grained alumina. In the former, the results show the evolution of heterogenous stress distributions inside individual grains and across grain boundaries. In the latter, the results show the motion of grain boundaries with 250 nm spatial resolution. Finally, we will also briefly discuss the current state of the art of pf-HEDM data analysis procedures and considerations at the APS, the ESRF, and SPring-8.

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[1] Li, W., Sharma, H., Kenesei, P., Ravi, S., Sehitoglu, H. and Bucsek, A., 2023. Resolving intragranular stress fields in plastically deformed titanium using point-focused high-energy diffraction microscopy. Journal of Materials Research, 38(1), pp.165-178.

Introduction to Diffraction Microstructure Imaging Data Post-processing

Darren C. Pagan[1]

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Once the experiment has been completed and the microstructural features have been reconstructed, the real work begins. Here we introduce the essentials of diffraction microstructure imaging (DMI) data analysis techniques and strategies. The presentation will begin with a discussion of coordinate systems and transformation of bases. Using these fundamentals, we will then discuss multimodal data registration. This will be followed by a discussion of constitutive laws along with interpretation and analysis of DMI data through this lens. Lastly, we will discuss packaging of data for insertion into micromechanical modeling workflows.

Grain-level Chemo-mechanics and Polymorphism in Garnet Solid Electrolytes

Marm Dixit[1]

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Understanding the behavior of polycrystalline solid electrolytes at the grain level is crucial for comprehending filament formation and short-circuit phenomena in these materials. In this discussion, I will outline a method that combines far-field high-energy diffraction microscopy and tomography to assess the chemo-mechanical behavior of dense, polycrystalline garnet (Li7La3Zr2O12) solid electrolytes. By mapping the mechanical state of all grains within bulk solid electrolytes during cycling of a symmetric Li | LLZO | Li cell, we found that failure in garnet solid electrolytes typically begins locally and appears to follow a stochastic process. Additionally, the presence of a minor cubic polymorphic phase can create local gradients in both transport and mechanical properties within the solid electrolyte. We have observed a notable correlation between areas of high microstructural variation and the presence of this secondary cubic polymorph, with stress concentrations aligning with these regions. Through a combination of experiments that capture both real- and reciprocal-space imaging, we have gathered evidence of a significant coupling between field-driven ion transport and the mechanical response of individual grains.

Neper and FEPX: Application of Open-source Polycrystal Generation, Deformation Simulation, and Post-processing Software to HEDM Experiments

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Over the past 5 years, two software packages – Neper and FEPX – have undergone significant transformation to create a software ensemble for the study of polycrystal plasticity. Namely, the packages have both received extensive upgrades and extensions in an attempt to create extensive, harmonious interoperability. Specifically, the two packages were upgraded to common standards, both in terms of usage (e.g., common input/output) and resources (e.g., documentation), allowing for fully self-contained and automated workflow "from data to results." Here, we will demonstrate the application and usage of Neper and FEPX in conjunction with HEDM experiments. In particular, we will discuss how polycrystal data from both near-field and far-field HEDM experiments may be utilized in the Neper/FEPX workflow to generate predictions of the intragranular micromechanical response of the material. We will provide demonstrative examples on actual HEDM datasets, discussing various polycrystal generation methodologies, deformation predictions, and post-processing. We will further discuss how these advances facilitate rapid predictions which may be used to inform experiments on-the-fly.

Scientific Examples and Demonstration of Fast Fourier Transform-based Simulations with X-ray Experiments to Explore Deformation and Fatigue in Structural Alloys

Michael D. Sangid[1], Krzysztof Stopka[1], and Kyle Jung[1]

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During mechanical loading, polycrystalline alloys experience crystallographic slip in a heterogenous fashion, leading to strain localization and stress concentrations. Upon cyclic loading, the evolutions of these fields result in crack initiation and propagation, known as fatigue. Fatigue represents a primary failure mechanism in structural materials. It is essential to understand the physics of fatigue, in order to create a cause-and-effect relationship in an effort to reduce the probability of such failures. The close synergy between *in-situ* high-energy x-ray diffraction microscopy (HEDM) experiments and crystal plasticity modeling provides new opportunities for model calibration, verification, and validation, by providing direct means of comparison, thus removing aspects of epistemic uncertainty in the approach. Further, data fusion between *in-situ* experimental and model-based data, along with data driven approaches, provides a paradigm shift for determining the emergent behavior of deformation and failure, which is the foundation that underpins the mechanical behavior of polycrystalline materials. In this talk, we provide an overview of the scientific examples and use of a full-field fast Fourier transform (FFT) framework to simulate HEDM results. We will present demonstrations of leveraging the output of HEDM data for processing within an open-source toolset, Dream.3D, for input into FFT simulations. The theory of FFT, along with the input/output file structure, and visualization of such data will be shown and discussed.

In-situ X-ray Tomography and High-energy Diffraction Microscopy for Sand, Rocks, and Concrete: Examples and Challenges

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Sands, rocks, and concrete play critical roles in our natural and built environments. The mechanical properties of these materials control the progression of geophysical events and the resilience of civil infrastructure. Engineers and scientists have used uniaxial and triaxial tests to evaluate mechanical properties of sands, rocks, and concrete for decades. Constitutive models used to predict their behaviors employ the outcomes of these tests and rely on assumptions regarding stress and strain behavior at the sub-sample scale.

Advances in x-ray tomography (XRT) and high-energy diffraction microscopy (HEDM) capabilities during *in-situ* mechanical testing offer tremendous opportunities to develop a deeper understanding of the behavior of sands, rocks, and concrete, with applications to continuum and mesoscale constitutive modeling. Here, we describe three recent examples of combining *in-situ* XRT and HEDM with micromechanical testing to study these materials under load. These examples highlight unique *in-situ* mechanical testing capabilities, common challenges associated with making and registering multimodal measurements, and applications to furthering our fundamental understanding of material behavior and improving constitutive modeling at both very small scales (with scanning far-field HEDM) and larger scales (with far-field HEDM). We then discuss major open challenges, including development of loading devices for *in-situ* conditions involving high pressures and application of HEDM to natural materials with non-ideal crystals. We conclude with a discussion of future measurement needs and the capabilities offered by the latest synchrotron upgrades.