Technical Aspects of Synchrotron X-ray and Neutron Measurements for Diffraction Microstructure Imaging

Organizing Committee:
Thien Phan: Chair, NIST (thien.phan@nist.gov)
Darren Pagan, CHESS (dcp99@cornell.edu)
Ulrich Lienert, DESY (ulrich.lienert@desy.de)
Adam Creuziger, NIST (adam.creuziger@nist.gov)

Description:
The workshop will be focused on the communication and discussion of technical aspects of synchrotron X-ray- and neutron-based microstructural characterization techniques associated with diffraction microstructure imaging in an interactive setting. We will cover the latest developments on data acquisition, management, and reconstruction; sample environments; and a dedicated session focused on support of advanced manufacturing. Discussion sections will focus on the development of physical and software standards and the building of an IUCr commission for this research community.

Day 1: Wednesday, July 15 (start 10 AM EST)
Workshop Introduction (10 min.): Darren Pagan / Adam Creuziger / Ulrich Lienert
Speaker Session (30 min. each, 60 min. total): In-situ and Ex-situ quantification of advanced manufacturing process/materials

- Fan Zhang, NIST – "From Storage Ring to Manufacturing Prosperity: Applications of Synchrotron and Neutron Research in Promoting Advanced Manufacturing"
- Tao Sun, UVA – "In situ/operando measurement of critical structure parameters in metal additive manufacturing using high-speed synchrotron x-ray imaging"

Update Session (10 min. each, 75 min. max): Beamline updates

- Jon Wright, ESRF – "News from ID11 at the ESRF"
- Ulrich Lienert, DESY – "Grain resolved diffraction at the PETRA III P07 and P21.2 beamlines"
- Peter Kenesei, APS – "High-energy X-ray Microscopy Beamlines Instrumentation Developments at the APS"
- Jon Tischler, APS – "Status of using micro-diffraction at < 500 nm at the APS"
- Thomas Wroblewski, HZG – "Energy dispersive diffraction imaging"
- Carsten Detlefs, ESRF – "ID06, the ESRF Dark-Field X-ray microscopy beamline is now open for general user proposals"

Break (15 min.)
Discussion (60 min.): Physical Standards– led by Jun-Sang Park, APS

Day 2: Thursday, July 16 (start 10 AM EST)
Daily Introduction (5 min.): Darren Pagan / Adam Creuziger / Ulrich Lienert
Speaker Session (30 min. each, 90 min. total): New Data Reconstruction Methods

- Johan Hektor, Lund University – "Novel methods for reconstructing strain fields from scanning 3DXRD data"
- Kelly Nygren, CHESS – "Leveraging far-field and near-field HEDM data to efficiently reconstruct
- intragranular orientation fields"
- Peter Reischig, InnoCryst – "Three-dimensional sub-grain mapping of lattice strains and orientations in polycrystals"

Update Session (10 min. each, 60 min. max): Codebase updates

- Bob Suter, CMU – “New Dimensions in HEDM”
- Hemant Sharma, APS – “Data driven microstructure reconstruction”
- Joel Bernier, LLNL – “Updates to the HEXRD Software Library”
- Hamid Abdolvand, Western Ontario – “A tool box for post-processing 3D-XRD data and reconstructing finite element input files”
- Henri Proudhon, PSL University - MINES ParisTech – “Towards a data platform for the mechanics of microstructures combining experiments and simulations”

Break (15 min.)
Discussion (60 min.): Code Standards – led by Jette Oddershede, XNOVO

Day 3: Friday, July 17 (start 10 AM EST)
Daily Introduction (5 min.): Darren Pagan / Adam Creuziger / Ulrich Lienert
Speaker Session (30 min. each, 60 min. total): New Instrumentation and Sample Environments

- Paul Shade, AFRL – “Advanced In Situ Loading Environments for Synchrotron X-ray Diffraction Experiments”
- Ashley Bucsek, U. Michigan – “Studying Phase Transformations and Twinning in Metals with Dark-Field X-ray Microscopy”

Update Session (10 min. each, 75 min. max): In-Situ Measurements

- Matthew Miller, CHESS – “The Materials Solution Network at CHESS (MSN-C) - a new paradigm for supporting military and industrial research”
- Mustafacan Kutsal, DTU – “Update on high resolution 3DXRD at ESRF”
- Lianyi Chen, Wisconsin – “Quantitatively revealing the dynamics and mechanisms of metal additive manufacturing processes by high-speed synchrotron x-ray imaging and diffraction”
- Sven Vogel, LANL – “TBD”
- Yunhui Chen, University College London – “Understanding Directed Energy Deposition Additive Manufacturing Process using In situ and Operando X-ray Imaging and Diffraction”
- Sébastien Merkel, Université de Lille, France – “TIMEleSS tools: a toolbox for high pressure multigrain diffraction experiments”

Break (15 min.)
Discussion (60 min.): IUCr Commission Update – led by Darren Pagan, CHESS
Close-out (10 min.)

From Storage Ring to Manufacturing Prosperity: Applications of Synchrotron and Neutron Research in Promoting Advanced Manufacturing

Fan Zhang, NIST

Manufacturing is an engine of U.S. economy and a pillar of national security. Advanced manufacturing, encompassing innovative manufacturing methods and products made possible by these methods, enables continuous technology development, increase productivity, and serve as fundamental driving force behind creation of new industrial sectors. The high value added, often associated with advanced manufacturing, ensures a strong U.S. manufacturing industry and its leadership in the face of intense global competition.

Synchrotron and neutron based techniques represent a frontier of materials characterization. Because of their penetration ability, they are well suited to probe the structure-property relationship of almost all materials and have an eminent role in modern materials science and engineering research. They have greatly impacted a wide range of advanced manufacturing technologies, ranging from nanotechnology, sustainable and green technology, to additive manufacturing (AM) technologies.

AM technologies represent a fast-growing manufacturing domain and are adopted into increasingly demanding industry-driven applications. AM is unique in that the material is built at the same time as the component. The nonequilibrium nature of the AM processes introduces highly heterogeneous microstructure across multiple length scales. A proper understanding of the properties and behaviors of AM materials, including the build process, presents a major characterization challenge to the construction of the processing-structure-performance relationship of the final product and to the certification of materials with predictable materials properties. Using examples, we will demonstrate the importance of synchrotron and neutron based characterization techniques in understanding AM materials and their processing at a fundamental level, as well as their roles in validating and benchmarking computer modeling and simulations. We will also highlight their contribution to the development of AM technology and industrial innovation.

<u>In situ/operando measurement of critical structure parameters in metal additive manufacturing using high-speed synchrotron x-ray imaging</u>

Tao Sun, University of Virginia

Additive manufacturing (AM) of metals have found many applications in a variety of fields, owing to its unique advantages, including but not limited to, the capability of building complex 3D parts, short supply chain, on-site and on-demand manufacturing, and reduction of material and energy consumption. Despite years of development and its promise to revolutionize the manufacturing industry, metal AM is facing a few critical challenges which prevent it from reaching its full potential, which include existence of substantial defects, lack of high-fidelity models, limited materials, and poor reliability. Collectively, these issues cause tremendous problems for qualifying and certifying AM materials and parts. One of the major reasons why we have not fully addressed these issues is that we had not been able to characterize the metal AM processes in situ and in real time. The deficiency of direct observation and quantitative measurement of the energy-matter interaction process limited our understanding of the metal AM technologies.

Metal AM processes are conceptually simple. Taking laser powder bed fusion (LPBF), the most extensively used metal AM technique, as an example, the layer-by-layer printing process involves scanning a high-power-density laser beam across the repeatedly coated thin powder bed. The laser beam melts the powder locally and fuse it to the previous layer. The high power and scan speed of the laser creates extreme thermal conditions which leads to complex multi-phase dynamics. At the Advanced Photon Source, we recently developed an experimental platform for in situ/operando study of the LPBF process using high-speed x-ray imaging. We've demonstrated that many important dynamic structure parameters can be measured with unprecedented spatial and temporal resolutions, such as the morphologies and fluctuation of melt pool and keyhole, melt flow, solidification velocity, etc. Also, the generation of different types of defects can be observed directly, thanks to the superior penetration of high-energy x-rays. In addition, we've applied the high-speed x-ray imaging technique for studying other metal AM processes, such as blown-powder directed energy deposition and binder jetting.

In this presentation, I will introduce the current state of research on metal AM using the high-speed synchrotron x-ray imaging, and highlight the new insights gained from these in situ/operando experiments. Furthermore, I will elucidate how the x-ray imaging study has been helping us mitigate the structure defects, develop process models, and improve the build reliability.

<u>Novel methods for reconstructing strain fields from scanning 3DXRD data</u>

Johan Hektor, Lund University

Scanning 3DXRD allows for reconstruction of intragranular variations of properties such as strain. The standard method for reconstructing the strain field, developed in Ref. [1], assumes that the strain tensor in each voxel can be reconstructed independently of their neighbours. It is demonstrated that this approach will introduce bias in the reconstruction which will lead to e.g. dampening of strain gradients. To overcome these issues we have developed two new algorithms for reconstructing strain. The methods have been used to reconstruct the 3D strain field in a tin grain in the vicinity of a tin whisker, as well as on quartz grains subject to compressive loads. In both cases the data are from ID11. Improvements in reconstruction quality achieved by the two proposed methods are further supported by reconstructions using synthetic diffraction data.

[1] Hayashi, Setoyama & Seno (2017). Mater. Sci. Forum, 905, 157–164

<u>Leveraging far-field and near-field HEDM data to efficiently reconstruct intragranular orientation fields</u>

Kelly Nygren, CHESS

A data reduction algorithm coupling far-field and near-field high-energy X-ray microscopy has been created to extract intragranular orientation descriptors and resolve intragranular orientation fields in polycrystalline alloys. The algorithm is integrated into the HEXRD data reduction framework, which previously only extracted grain-averaged strains, grain-averaged orientations, and centroid positions of each grain in a polycrystal. Extracting intragranular orientation fields provides the ability to explore microstructure evolution due to plastic deformation and develop new constitutive models for metallic alloys. A demonstration of this algorithm is presented for the titanium alloy, Ti-7Al.

<u>Three-dimensional sub-grain mapping of lattice strains and orientations in polycrystals</u>

Péter Reischig, InnoCryst Ltd

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, although it poses a limitation on grain mosaicity. The ESRF source upgrade will bring scanning times of the order of tens 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 with a potential sensitivity in the range 10-4 to 10-3. Furthermore, the single-crystal elastic moduli of the material 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.

<u>Advanced In Situ Loading Environments for Synchrotron X-ray Diffraction Experiments</u>

Paul Shade, AFRL

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.

<u>Studying Phase Transformations and Twinning in Metals with Dark-Field X-ray Microscopy</u>

Ashley Bucsek, University of Michigan

Twinning and martensitic phase transformations are the enabling deformation mechanisms behind a diversity of mechanical and functional properties and behaviors in metals including ferroelasticity, damping, superelasticity, shape memory, toughening, ductility, and hardening. Both martensitic phase transformation and twinning are extremely sensitive to local stresses, resulting in a pronounced heterogeneous response that leads to unpredictability and non-optimal local deformation processes. Understanding these relationships is critical to be able to model twinning and phase transforming behaviors and design superior twinning and phase transforming metals. In situ dark-field X-ray microscopy (DFXM) is uniquely suited for studying twinning and martensitic phase transformations, because it offers spatial resolutions (~100 nm) sufficient for measuring the local strains surrounding the nucleation and growth of these important mechanisms under the surface of bulk samples. In this talk, I present results on using DFXM to study martensitic phase transformation in nickel-titanium shape memory alloys during in situ thermal cycling, and I discuss future opportunities for studying twinning and martensitic phase transformations with DFXM.