APS Workshop 4: The Future of Full-field 3D Imaging at APS-U: A Multiscale and Multimodal Approach

Friday, May 10, Morning

8:30 – 8:50	Alberto Mittone (Argonne National Laboratory) *Hard X-ray Full-field Imaging at APS-U*
8:50 – 9:10	Viktor Nikitin (Argonne National Laboratory) *Imaging Gas Hydrate Formation Processes Using Synchrotron Techniques: Current Results and Plans*
9:10 – 9:35	Aaron Kuan (Yale University) *Neural Cartography: Mapping the Brain with X-ray and Electron Microscopy*
9:35 – 10:00	Nathan Bechle (USDA Forest Service, Forest Products Laboratory) *3D Imaging for Wood Science Applications*
10:00 – 10:20	Break
10:20 – 10:45	Jake Socha (Virginia Polytechnic Institute & State University) *It's Both a Bug and a Feature: 3D Imaging of Insects and Other Small Animals*
10:45 – 11:10	Tim Fister (Argonne National Laboratory) *Multiscale Imaging of Aqueous Batteries during Formation and Cycling*
11:10 – 11:35	Nikhilesh Chawla (Purdue University) *Multimodal Characterization of Sn-Bi Alloy Solidification Using Synchrotron X-ray Microtomography and Energy Dispersive Diffraction*
11:35 – 12:00	Christopher Powell (Argonne National Laboratory) *X-ray Imaging of Fuel Injection in 2D and 3D*
12:00	Adjourn

Hard X-ray Full-field imaging at APS-U

A. Mittone[1], V. Nikitin[1], S. Clark[1], A. Deriy[1], K. Fezzaa[1], A. Kastengren[1], P. Shevchenko[1], and F. De Carlo[1]

[1]Advanced Photon Source, Argonne National Laboratory, Lemont, IL 60439

The Imaging Group at APS manages three specialized beamlines – 2-BM, 7-BM, and 32-ID – dedicated to full-field imaging in the micro- and nano-micrometer scale and to ultra-high-speed applications. This group of instruments enables the group to span three orders of magnitude in spatial and temporal resolution.

Within the ongoing APS-U project, the Imaging Group is aims to enhance its capabilities, with a primary focus on improving speed and expected image quality. This collective enhancement in instrumentation creates novel opportunities for researchers engaged in multiscale, multimodal, and time-resolved experiments. A noteworthy addition to the facility is the forthcoming installation of a projection microscope (PM) at 32-ID, effectively bridging the current gap between nano-tomography (represented by a Transmission X-ray Microscope at 32-ID) and micro-tomography (at 2-BM and 7-BM).

Subsequent to the APS-U project, the Imaging Group at APS will offer a diverse array of instruments and techniques to the user community, facilitating versatile approaches for multiscale and multimodal research. An overview of the expected capabilities and instrumentation will be presented, ensuring a comprehensive understanding of the opportunities available to researchers.

Imaging Gas Hydrate Formation Processes Using Synchrotron Techniques: Current Results and Plans

V. Nikitin[1]

[1]Advanced Photon Source, Argonne National Laboratory, Lemont, IL 60439

Gas hydrates, formed by water and gas molecules under specific pressure and temperature conditions, are crucial in energy production, climate change, and environmental processes. Understanding their dynamic formation within porous media is essential for optimizing extraction methods and assessing risks. In this presentation, we share results from computed tomography (CT) experiments on methane hydrate formation and dissociation within an environmental cell filled with sand and coal. Synchrotron micro-CT imaging provided high spatial and temporal resolution scans, offering insights into water movement and diverse gas hydrate formation types. Additional nano-CT measurements delved deeper into water extraction mechanisms via micro-channels within grains. The environmental cell for the hydrate growth was also equipped with acoustic sensors, which facilitated correlating CT with acoustic data for further analysis and using the results during real seismic surveys. In the presentation, we will also discuss the potential of integrating new modalities into our experiment. For instance, x-ray diffraction techniques may provide new insights into gas hydrate crystallography, aiding in characterizing hydrate phases, interfacial interactions, and the total amount of formed hydrate.

Neural Cartography: Mapping the Brain with X-ray and Electron Microscopy

Aaron Kuan[1]

[1]Yale University, New Haven, CT 06520

One of the grand quests in neuroscience is to build complete maps of the brain, charting all of its cells and the connections between them. Such wiring diagrams, called connectomes, promise to shed light on how networks of neurons can give rise to thoughts, memories, and actions, and to help reveal the underlying causes of neurological diseases. Today, complete connectomes are only available for very small organisms such as worms and fruit flies. To map the immensely more complex brains of mice, primates, and ultimately humans, new imaging technologies will be needed.

In this talk, I will present recent innovations that are expanding the scope and detail at which we can image the brain. I will share how we have combined 3D electron microscopy with *in-vivo* experiments to investigate how neural circuits in the mouse brain implement decision-making. I will then demonstrate how synchrotron-based x-ray nano-holography can resolve neuronal wiring, and discuss the potential of scaling this non-destructive, high-throughput imaging approach to brain-wide circuits. In conclusion, I will propose that the complementary techniques of *in-vivo* imaging, x-ray nano-holography, and electron microscopy together form a multiscale approach that can tackle the massive scaling challenge of mapping mammalian brains.

3D Imaging for Wood Science Applications

Nathan J. Bechle[1] and Joseph E. Jakes[1]

[1]Forest Biopolymers Science and Engineering, USDA Forest Service, Forest Products Laboratory, Madison, WI 53726

Lignocellulosic biomass, like wood, is primed to play a major role in our future bioeconomy. Wood is an anisotropic cellular material with a complex hierarchical structure and hygroscopic cell walls. The development of technologies to fully utilize lignocellulosic biomass resources is hindered by the lack of fundamental processing-structure-property-performance relationships, especially at small length scales in the cellular structure. Synchrotron x-ray computed tomography (XCT) can readily be used to study wood at these small length scales with sub-micrometer spatial resolution. The highly penetrating nature of x-rays and open layout of beamlines opens the possibility to also employ *in-situ* environmental chambers. To study the effects of moisture, we built an *in-situ* relative humidity (RH) chamber for Advanced Photon Source (APS) XCT beamline 2-BM. The environment inside of the RH chamber is continuously controlled from 0 to 100% RH using an external RH generator. Examples of wood science research conducted at APS 2-BM will be presented, including studies of wood-adhesive bondlines, moisture swelling, thermal decomposition, and real-time imaging of liquid water transport through the wood cellular structure. Future opportunities utilizing the APS-U will also be discussed, such as real-time imaging of wood pyrolysis, multiphysics interactions within the wood cellular structure, and multimodal studies combining XCT, XFM, and XANES to better understand wood decay mechanisms in wood cell walls.

It's Both a Bug and a Feature: 3D Imaging of Insects and Other Small Animals

Jake Socha[1]

[1]Department of Biomedical Engineering and Mechanics, Virginia Polytechnic Institute & State University, Blacksburg, VA 24061

Insects can be viewed as highly effective microfluidic systems, capable of producing multiple types of fluid flows within a small body. They are able to pump viscous liquids to ingest meals, to pump air through a highly reticulated respiratory system, and to pump hemolymph (insect blood) through a non-vessel dominated, porous body. Understanding how these systems function has been greatly aided by synchrotron full-field imaging: 2D projection imaging to visualize internal movements and 3D tomographic imaging to determine complex internal anatomy, which can be used to lend insight into movement mechanisms and serve as a basis for computational models. In this talk, I will discuss some of our efforts to understand 3D insect anatomy and provide desires for future improvements, focusing on some recent efforts at the APS that include TXM imaging.

Multiscale Imaging of Aqueous Batteries during Formation and Cycling

Tim Fister[1], Seongjun Kim[1], Mark Wolfman[1], Alan Kastengren[1], and Viktor Nikitin[1]

[1]Advanced Photon Source, Argonne National Laboratory, Lemont, IL 60439

While the overall cost of Li-ion has dramatically decreased in the last 30 years, its plateauing economics is unlikely to reach DOE's long duration earth shot goal of 90% cost reduction within a decade. In stark contrast, aqueous batteries involving low-cost materials like iron, zinc, or lead, are intrinsically low voltage, but have substantial capacity since they rely on electrochemically-driven crystal growth and dissolution. This intrinsically large capacity is often only used for single-use batteries (i.e., alkaline cells), or deliberately limited to reach higher cycle life (i.e., lead acid batteries). For aqueous batteries to reach their theoretical capacity over hundreds to thousands of cycles requires control over the nanoscale morphology of the individual crystallites, as well as the evolving microstructure of the porous electrode. In the case of lead acid batteries, repeated cycling can lead to ripening of PbSO4 crystallites that form on discharge; these particle-scale changes lead to larger scale pore-clogging and reduced electroactive surface area, both factors that impede rechargeability. To better understand this hierarchical problem, we have used both transmission x-ray microscopy (TXM) and high energy white beam tomography on aqueous battery electrodes during operation. In this talk, I will outline the dramatic evolution of lead acid battery electrodes during formation, where volume change and parallel gassing reactions lead to pronounced changes in overall porosity. Similar data was also measured on iron-air "rust" batteries, where rapid cycling leads to irreversible growth of discharge species within the metal anode. In parallel, I will show TXM reconstructions on lead acid electrodes at the start of cycling and at the end-of-life where charge and discharge species can be easily distinguished. To better understand factors limiting charge acceptance, we will compare dissolution behavior between acid and water solutions, which have starkly different solubilities. Building from this work, I will finally discuss opportunities to dynamically switch between these regimes using new capabilities available in the APS Upgrade.

Multimodal Characterization of Sn-Bi Alloy Solidification Using Synchrotron X-ray Microtomography and Energy Dispersive Diffraction

N. Chawla[1], A. Luktuke[1], J. Wu[1], and A. Kastengren[2]

[1]School of Materials Engineering, Purdue University, West Lafayette, IN 47907
[2]Advanced Photon Source, Argonne National Laboratory, Lemont, IL 60439

Sn-Bi alloys have gained considerable interest in semiconductors due to their potential applications in heterogeneous integration in packaging (HIP). The formation of primary phases such as faceted Bi particles and Sn dendrites during solidification of solder alloys play pivotal role in determining the mechanical and electrical behavior of a solder joint. It is important to understand the complex relationship between the evolution of microstructure and composition during solidification. In this study, for the first time, we have studied the Sn-70Bi alloy solidification using simultaneous 4D X-ray Tomography (XRT) and Energy Dispersive Diffraction (EDD) at beamline 7-BM of the Advanced Photon Source (APS). The microtomography analysis revealed the morphological changes and growth kinetics, while the EDD investigation provided the information regarding phase composition and lattice strain. In particular, we will report on the formation of Bi pyramid structures during solidification. The combined analysis provided unique quantitative correlations between these techniques which will be discussed.

X-ray Imaging of Fuel Injection in 2D and 3D

Christopher F. Powell[1]

[1]Advanced Photon Source, Argonne National Laboratory, Lemont, IL 60439

Fuel injection is an important technology for modern combustion engines since the mixing of fuel and air determines the pollutant formation and the combustion efficiency. This is crucial for both piston engines aviation engines, since, as sustainable fuels are introduced, the engines must be optimized or even re-engineered. X-ray imaging, both 2D and 3D, can improve our understanding of this process in several ways. First, the flow internal to the fuel injector can be diagnosed non-destructively, with imaging of cavitation inside the injector, measurements of micron-scale geometric features that influence the fuel flow, and quantification of fuel deposits that may form over time.

X-ray diagnostics are also a powerful tool for measurements of atomization as the fuel exits the injector, since they can penetrate high number density fields of droplets that are optically opaque. This allows x-ray imaging to capture the morphology of spray breakup, determine near-nozzle velocity, and measure the density distribution in regions that are too dense for quantification with visible light.

These data are used to provide quantitative measurements of fluid flows that are otherwise unavailable. The presentation will discuss the diagnostics as well as past and future applications.