APS Workshop 3: Full Field Imaging at APS-U: Back to the Future

Thursday, May 8, Morning

8:30 – 8:35 Francesco De Carlo (X-ray Science, Argonne National Laboratory)
Welcome

8:35 – 8:45 Alberto Mittone (X-ray Science, Argonne National Laboratory)
Current Status and Recent Results from APS Imaging Beamlines

8:45 – 9:15 Stuart Stock (Department of Cell and Developmental Biology, Feinberg School of Medicine, Northwestern University)
2-BM: Two Decades and Beyond

9:15 – 9:45 Nuno Macarico da Costa (Allen Institute)
Towards a Scalable Pipeline for Mapping Neuronal Circuits at Centimeter Scale and Nanometer Resolution

9:45 – 10:15 Jake Socha (Department of Mechanical Engineering, Virginia Tech)
Full-field Imaging of Insects and Other Small Animals

10:15 – 10:30 Break

10:30 – 11:00 Nikhilesh Chawla (School of Materials Engineering, Purdue University)
High Resolution Imaging of Defects in Semiconductors: Detection, Reliability, and Mitigation

11:00 – 11:30 Tao Sun (Department of Mechanical Engineering, Northwestern University)
The Next Phase of Operando *Synchrotron Experiments on Additive Manufacturing*

11:30 – 12:00 Conghao Yi (Department of Civil and Environmental Engineering, Princeton University)
3D Mineral Composition Heterogeneity: LABQ3 Quantitative Analysis of XCT

12:00 – 1:00 Lunch Break

Thursday, May 8, Afternoon

1:00 – 2:00 IMG Beamline Visits

2:00 – 2:30	Douglas Lars Nelson (School of Materials Science and Engineering, Georgia Institute of Technology) *Investigating Chemo-mechanical Degradation in Solid-state Batteries with Synchrotron-enabled* Operando *X-ray Computed Microtomography*
2:30 – 3:00	Eva Allen (Applied Materials, Argonne National Laboratory) *Three-dimensional Quantification of Chemical Heterogeneity in Lithium-ion Cathodes for Synthesis and Direct Recycling*
3:00 – 3:40	Songyuan Tang (X-ray Science, Argonne National Laboratory) *How AI-based Spatiotemporal Fusion Can Benefit the High-speed Imaging User Community*
3:40 – 3:55	Break
3:55 – 4:30	Xiaoyang Liu (X-ray Science, Argonne National Laboratory) *Deep Learning Segmentation with Dragonfly*
4:30 – 4:55	Viktor Nikitin (X-ray Science, Argonne National Laboratory) *New Data Acquisition Schemes and Reconstruction Methods for the Projection X-ray Microscope*
4:55 – 5:00	Francesco De Carlo (X-ray Science, Argonne National Laboratory) *Wrap-up*
5:00	Adjourn

Current Status and Recent Results from APS Imaging Beamlines

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

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On June 27, 2024, the Advanced Photon Source (APS) began delivering its first beam for scientific use, marking a gradual return to user operations and providing the scientific community with an upgraded facility featuring outstanding brilliance and beam coherence. The commissioning of the Imaging Group beamlines began in March 2025, and preliminary results obtained with the new APS-U machine, as well as the status of the 2-BM, 7-BM, and 32-ID beamlines, will be reported.

2-BM: Two Decades and Beyond

Stuart R. Stock[1]

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The speaker has been working at 2-BM at the Advanced Photon Source (APS) since November 2001. This talk outlines personal observations of the changing microCT capabilities over the years, illustrated by the biomineralized specimens the speaker has studied. The speaker's first synchrotron microCT examples predate the opening of 2-BM and include fatigue crack closure observed in an aluminum alloy under load and the densification of SiC/SiC composite samples. The next examples are tomography at APS centered on imaging sea urchin teeth, spines, and other structures of biocalcite. The third set of examples are of bioapatite in bones and teeth and in scaffolds designed to promote bone healing. Then recent work on 3D quantification of the bioapatite-cartilage trabecular structures of the shark vertebral centra is reviewed. The final topic is the speaker's view of where tomographic imaging is headed over the next few years.

Towards a Scalable Pipeline for Mapping Neuronal Circuits at Centimeter Scale and Nanometer Resolution

Nuno Macarico da Costa[1]

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Transmission Electron Microscopy (TEM) has played a fundamental role in neuroscience, from revealing the structure of the first synapse [2] to enabling the complete neuronal wiring diagrams of *C. elegans [4]* and *Drosophila* larvae [5], and more recently, the adult *Drosophila* brain [6,1]. It has also been instrumental in the largest reconstructions of mammalian axons [3]. Recent advancements have demonstrated that serial-section TEM, when combined with large-scale imaging and reconstruction techniques, can map synaptic connectivity at nanometer resolution across millimeter-scale volumes. As the field moves toward larger and more complex nervous systems, the next logical step is the complete reconstruction of a mammalian brain, with the mouse as the primary target.

Here, we present our progress in developing critical technologies to overcome the challenges of scaling up to the whole mouse brain while linking structural connectivity to cell types. We describe advances in staining and serial sectioning of whole mouse hemispheres and leverage the speed, cost-effectiveness, high resolution, and re-imaging capabilities of TEM to acquire whole-hemisphere sections at ~4 nm resolution.

As part of our imaging pipeline, TEM-acquired data is registered with whole-hemisphere imaging using micro-CT at the Argonne Advanced Photon Source and the European Synchrotron Radiation Facility. This approach provides essential quality assessment of the sample, establishes a coordinate framework for each dataset, aligns each mouse sample to the *Mouse Common Coordinate Framework*, and captures high-resolution details of somata and blood vessel networks.

Finally, we are working toward integrating these high-resolution TEM datasets with light microscopy reconstructions to link connectivity findings to gene expression. Our ultimate goal is to create an integrated atlas of cell types and connectivity, providing a comprehensive framework for understanding the mammalian brain at unprecedented resolution.

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Full-field Imaging of Insects and Other Small Animals

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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 how insects function biomechanically and provide suggestions for future improvements, focusing on some recent efforts at the APS that include TXM imaging.

High Resolution Imaging of Defects in Semiconductors: Detection, Reliability, and Mitigation

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Heterogeneous integration in packaging (HIP) offers a power-efficient approach to achieving high-density three-dimensional system integration along with improved bandwidth density and energy efficiency. HIP of Interconnected Circuits (ICs) in dense 2D and 3D packages can provide significantly enhanced functionality; however, as packages become more densely packed with ICs, newer, smaller defects that affect reliability are emerging. Solder micro bumps, through silicon vias (TSVs), hybrid copper-copper bonding, and silicon interposers are important, technologically relevant structures where defects can form and affect device performance and reliability. Conventional defects, such as cracking of interconnects, surface delamination, void formation, and solder joint failures, are further exacerbated in HI packages (HIPs), leading to losses in performance, reliability, and overall lifespan.

In this talk, I will describe advanced x-ray imaging metrologies and predictive multiscale modeling tools for solving complex problems and accelerating manufacturing and integration. We propose a transformational approach for rapid detection of defection in next generation HIPs and the quantification of the impact of these defects on reliability of packages. The systematic approach aims at making detection efficient and autonomous, along with a framework that encompasses probabilistic and uncertainty analysis to assess the probability of failure of a particular component. The rigorous fusion of materials science, mechanics, and machine learning, coupled with numerical and analytical modeling across different length scales, will be discussed.

The Next Phase of *Operando* Synchrotron Experiments on Additive Manufacturing

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In the past decade, synchrotron x-ray techniques have become powerful tools for studying additive manufacturing (AM) processes, particularly those used for printing metallic materials. The superior penetration power of high-energy x-rays and the extremely high beam brightness provided by the Advanced Photon Source and other third-generation synchrotron facilities enabled the quantitative characterization of dynamic structural evolution in bulk metallic materials with unprecedented spatial and temporal resolution. Many highly transient phenomena occurring during AM processes have been investigated. Specifically, *operando* synchrotron x-ray experiments contribute to this field by: (i) quantitatively measuring key process and material parameters (e.g., melt pool and keyhole morphologies, spattering, cooling rate, etc.) to calibrate and validate models; (ii) supporting mechanistic studies of defect generation mechanisms through direct visualization of sub-surface structure dynamics; and (iii) benchmarking process sensing techniques by providing high-fidelity ground truth.

The major upgrades at synchrotron facilities and the impact of the pandemic have occasionally caused pauses in *operando* AM experiments over the past few years. However, the community's enthusiasm for developing more sophisticated AM simulators and conducting more complex synchrotron experiments has remained strong. An increasing number of research teams are joining this field, committed to advancing the fundamental understanding of AM processes. In this presentation, I will start by highlighting the insights gained from our past synchrotron experiments and then discuss the remaining fundamental questions that could potentially be addressed in the next phase of synchrotron studies. In particular, I will present the new opportunities and challenges associated with experiments at fourth-generation synchrotron facilities.

The presenter acknowledges the contributions from his past and current team members at Argonne National Laboratory, University of Virginia, and Northwestern University. This presented research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science user facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.

3D Mineral Composition Heterogeneity: LABQ3 Quantitative Analysis of XCT

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Mineral compositions have significant implications for environmental and geological applications such as carbon sequestration, resource recovery, enhanced geothermal system, and water remediation. Due to variations in precipitation kinetics, diffusion, and transport, mineral compositions are often spatially varied (e.g., compositional zonation). Despite this known spatial heterogeneity, there is a lack of quantitative analysis methods that can spatially resolve elemental mixtures at the nanoscale. Leveraging our newly developed method, LABQ3 (Linear Attenuation Bayesian Quantitative 3D-mapper), we can quantify chemicals in nano-3D using synchrotron x-ray computed tomography (XCT) and Bayesian methods of data analysis. In this presentation, we present LABQ3 and findings on solid phase heterogeneity in ternary and binary solid solutions of calcium, zinc, cadmium carbonates synthesized via coprecipitation. XCT scans of the particles (~10 μm in diameter) were collected at beamline 32-ID-C of the Advanced Photon Source (Argonne National Laboratory). A bimodal composition distribution is observed in the ternary $(Ca,Zn,Cd)CO_3$ mineral mixture. However, the average composition falls at the valley of the two modes, indicating the average composition of a solid mixture is not necessarily representative. Additionally, it is known that the binary $(Ca,Zn)CO_3$ solid solution has a miscibility gap. Interestingly, we did not observe such a gap along the Ca-Zn axis in the $(Ca,Zn,Cd)CO_3$ ternary composition diagram. It is also known that the binary $(Ca,Cd)CO_3$ solid solution does not have a miscibility gap, and our findings were consistent with that. These insights are made possible by LABQ3, which enables first-of-a-kind chemical analysis along a composition continuum at the nanoscale, a feature that existing XCT analysis methods (e.g., segmentation) cannot achieve.

Investigating Chemo-mechanical Degradation in Solid-state Batteries with Synchrotron-enabled *Operando* X-ray Computed Microtomography

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Solid-state batteries could improve upon the energy density and safety of current lithium-ion batteries. However, the chemo-mechanical degradation of lithium-ion electrodes in contact with solid-state electrolytes is not well understood. Many investigations into this behavior employ *ex-situ* characterization methods that require disassembly of the battery, leading to artificial degradation that can skew results. Furthermore, these techniques are limited to observing degradation after cycling the battery and cannot provide information on the dynamic changes that occur during cycling. Realizing commercial-scale solid-state batteries will require *operando* and *in-situ* characterization methods to better understand and mitigate the dynamic chemo-mechanical degradation that the materials in these systems experience. To this end, we use synchrotron-enabled x-ray computed microtomography on 2-BM at APS to observe the morphological evolution and degradation of solid-state battery electrodes in *operando* and *in-situ* conditions that are otherwise inaccessible with laboratory-scale CT devices. Our experiments have revealed previously unknown degradation mechanisms in solid-state batteries with metal foils and particulate alloy anodes as well as in anode-less configurations. These results have guided investigations into new materials systems for solid-state batteries that could enable safe, energy-dense solid-state batteries in the future.

Three-dimensional Quantification of Chemical Heterogeneity in Lithium-ion Cathodes for Synthesis and Direct Recycling

Eva Allen[1], Jessica Macholz[1], Matthew Nisbet[2], Nighat Chowdhury[1], Feng Wang[1], Mansi Porwal[4], Denis T. Keane[5,6], William Guise Jr.[5,6], Zachary Dundek[1], Michael Caple[1], Viktor Nikitin[5], Jordi Cabana[3,4], Qiang Dai[1], and Albert Lipson[1]

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The evolution of chemical phenomena induced by high-temperature cathode synthesis significantly impacts cathode performance and stability. Reliable methods to measure and quantify heterogeneous elemental and oxidation states within battery cathodes are essential to ensure that process conditions yield the desired architectures. In this study, we present two investigations where nondestructive three-dimensional transmission x-ray microscopy (TXM) was employed to examine cathodes synthesized with full compositional gradients (FCG) for surface and structural stabilization, as well as direct recycling processes involving relithiation and upcycling. By integrating TXM with differential x-ray absorption spectroscopy (XAS) and Ni X-ray Absorption Near Edge Spectroscopy (XANES), we fully quantified the relationship between particle location and elemental content, demonstrating high statistical significance.

In the first study, FCG materials were designed with gradients of increasing manganese content and decreasing nickel content from the secondary particle core to the surface. After high temperature lithiation, elemental compositions evened out due to elemental diffusion, but gradients remain. In the direct recycling study, cathode materials from manufacturing scrap and end-of-life batteries were recovered intact. Relithiation processes were employed to resolve lithium deficiencies and rejuvenate the material for reuse. Cathode upcycling through Ni addition was also performed to produce cathodes with higher energy density. The desired end products for both relithiation and upcycling are cathodes with homogeneous elemental content; however, limitations in elemental diffusion can cause inefficient conversion. The methodology presented should be used to guide synthesis while ensuring that electrochemical performance is linked to precise elemental distributions at the nanoscale.

This work was performed through the ReCell Center, which gratefully acknowledges support from the U.S. Department of Energy (DOE), Office of Energy Efficiency and Renewable Energy, and the Vehicle Technologies Office. This work was predominantly conducted at the Materials Engineering Research Facility, Argonne National Laboratory, a U.S. Department of Energy Office of Science laboratory operated by UChicago Argonne, LLC under contract DE-AC02-06CH11357. This research used beamlines 32-ID-C and 5-BMC DuPont-Northwestern-Dow Collaborative Access Team (DND-CAT) located at Sector 5 of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357. DND-

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How AI-based Spatiotemporal Fusion Can Benefit the High-speed Imaging User Community

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Full-field ultra-high-speed (UHS) x-ray imaging experiments have been well established to characterize various processes and phenomena. However, the potential of UHS experiments through the joint acquisition of x-ray videos with distinct configurations has not been fully exploited. In this talk, I report a deep learning-based spatiotemporal fusion (STF) framework to fuse two complementary sequences of x-ray images and reconstruct the target image sequence with high spatial resolution, high frame rate, and high fidelity. A transfer learning strategy was applied to train the model and the peak signal-to-noise ratio (PSNR), average absolute difference (AAD), and structural similarity (SSIM) of the proposed framework, evaluated on two independent x-ray data sets were compared with those obtained from a baseline deep learning model, a Bayesian fusion framework, and the bicubic interpolation method. The proposed framework outperformed the other methods with various configurations of the input frame separations and image noise levels. With three subsequent images from the low-resolution (LR) sequence of a four times lower spatial resolution and another two images from the high-resolution (HR) sequence of a 20 times lower frame rate, the proposed approach achieved average PSNRs of 37.57 dB and 35.15 dB, respectively. When coupled with the appropriate combination of high-speed cameras, the proposed approach will enhance the performance and therefore the scientific value of UHS x-ray imaging experiments. Selected cases from this talk will be presented using Python programs in the training session.

Deep Learning Segmentation with Dragonfly

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This talk will give a tutorial on how to use the deep learning segmentation workflow in Dragonfly [1] software on x-ray computed tomography (CT) images. Semantic segmentation is important for x-ray tomography images quantification. Conventionally, thresholding segmentation and classic machine learning methods have been used widely. However, it struggles to segment distinct phases with similar range of pixel values in complex samples. Recently, with the rapid development of deep learning – leveraging artificial neural networks with multiple layers – machine learning-based segmentation in x-ray CT images has been explored and successfully demonstrated as a useful tool across various applications [2,3].

In the tutorial, I will talk about the deep learning semantic segmentation workflow in Dragonfly including labeling image, training deep learning model, applying model, and batch process using the x-ray CT images of a plant sample collected at micro-CT beamline 2-BM, Advanced Photon Source (APS). The user-friendly software interface, along with ready-to-use tools or functions, will accelerate the accumulation of training data and simplify the workflow. By exploring image segmentation tools, we aim to incorporate them into the tomographic experiments and support on post data analysis.

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New Data Acquisition Schemes and Reconstruction Methods for the Projection X-ray Microscope

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The Projection X-ray Microscope (PXM) instrument under development by the Imaging Group at sector 32-ID will implement phase-contrast 3D nanoimaging techniques called near-field ptychography [1] and holotomography [2]. Both techniques can be used to recover the sample refractive index consisting of the absorption and phase components. The phase component is typically 10-100 higher than the absorption component and therefore is more optimal for obtaining better quality images than the ones obtained with regular absorption contrast from full-field imaging instruments like micro-CT beamline 2-BM or nano-CT TXM at 32-ID.

While the two-phase contrast techniques are very optimal, especially with diffraction limited sources like APS-U, they require more complex data acquisition and reconstruction methods than in regular 3D tomography. The techniques also require using high-performance computing resources to process data quick enough to control dynamic in-situ experiments more efficiently.

In this talk, I will introduce the near-field ptychography and holotomography acquisition techniques and demonstrate how acquired data can be efficiently reconstructed using our recently developed methods [3-5]. New software package called HolotomocuPy (a follower of TomocuPy) will be used at 32-ID beamline as soon as the instrument is ready. For the current demonstration, I will use experimental data acquired at a similar PXM instrument from the European Synchrotron Radiation Facility.

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