Midwest Microscopy and Microanalysis Spring Meeting

Friday, March 27, 2026

Topic: *Emerging Opportunities and Challenges in In-Situ Microscopy*

Host: Northwestern University - Evanston Campus
Norris Center-Louis Room, #2-232; 2nd Floor

Presentation Abstracts

Tour de Fracture: High-cycle fatigue in the TEM. New techniques, discoveries, and opportunities

Stephen House, Sandia National Laboratories

The preponderance of in-service failures of metal structural components occurs as a result of metal fatigue from cyclic loading. As such, the ability to predict fatigue cracking and/or produce metals that resist these failure processes is of utmost importance. Unfortunately, the experimental data necessary for developing an understanding of the origins of fatigue crack processes is sorely lacking, especially at the nano- to atomic- scale. Beyond structural materials, nanoscale fatigue is also critical to the performance and reliability of microelectronics, flexible circuits and implants, and much more. The nascent field of in situ high-cycle fatigue in the TEM is enabling us to finally probe these questions at the spatial and temporal scales needed. Recently, it has even led to the discovery of an entirely unknown fatigue phenomenon: the self-healing of fatigue cracks in nanocrystalline metals.

This talk will discuss the rapid advancements taking place in this field including our development of the K*-control experimental methodology, which brought unprecedent precise control over fatigue cracking at hundreds of Hz, millions of cycles, and micron distances. This level of control enabled the observation of a wide variety of fatigue damage processes; many never directly observed in TEM before, especially at the nanoscale, and deviate from the predictions of existing models. Two of particular note, which will be discussed in greater depth, are the initial nucleation of fatigue cracks from the notch and fatigue crack self-healing. Autogenous crack healing, it turns out, is neither as rare nor simple as previously believed. We will also cover ongoing and upcoming efforts at the forefront of in situ fatigue in the TEM, the challenges facing the experimentalists and modelers working on them, and the exciting opportunities on the horizon. No doubt, many more surprises await us!

Intermittent Dynamics and Local Mobility in Metallic Supercooled Liquids: Insights from Electron Correlation Microscopy via Time-Resolved 4D-STEM

Po-Cheng Kung, University of Wisconsin-Madison

To better understand and control mobility in glasses and supercooled liquids it is necessary to develop a predictive theory linking mobility to motion-enabling structures. However, such a predictive theory is challenging due to the structural disorder and cooperative dynamics in these materials. Here, we present an improved method for computing the Two-Time Correlation Function (TTCF) from 4D-STEM based Electron Correlation Microscopy (ECM) experiments, which reveals time-resolved local dynamics and enables characterization of individual mobility events. We apply this technique to Pd-based metallic supercooled liquids, mapping the spatially resolved momentary mobility domains. To investigate the origins of intermittent mobility, we simulate ECM experiments from molecular dynamics (MD) model of supercooled liquids. The spatial/temporal patterns of ECM-detected mobility events are correlated with structural features extracted from MD trajectories, identifying atomic-scale predictors of mobility. This integrative approach connects atomic-scale simulations and nanoscale experiments, offering new insights into the structural origins of dynamics in disordered materials.

Assessing the Consequences of Liquid Thickness on the Spatial Resolution of Low Flux Liquid-Phase Transmission Electron Microscopy

Nathan Rosenmann, Northwestern University

Transmission electron microscopy (TEM) has been proven to be an incredibly powerful characterization technique leading to the understanding of structure and properties at the atomic scale. Researchers have been particularly interested in expanding this technique to the liquid-phase to enable the observation of dynamic processes with similar spatial resolutions through liquid-phase TEM (LPTEM). However, uncontrolled liquid thickness found in both graphene-based liquid cells (GLCs) and commercially available silicon nitride (SiNx)-based liquid cells have made reproducibility difficult and has led to debate on the optimal liquid layer thickness for LPTEM. Due to the bulging of the ~50 nm SiNx membranes resulting in thick (300-1000+ nm) liquid layers, the spatial resolution of LPTEM when operating at lower instantaneous electron fluxes (<1 e-/Å2/s) appears to be limited to roughly 20-30 nm due to low signal-to-noise ratios (SNR) resulting in noisy or blurry images of the solvated sample. Despite the spatial resolution being significantly improved at higher fluxes (10 e-/Å2/s), these fluxes are not amenable to the electron-beam-sensitive samples that have gained traction in recent year, which rapidly degrade or react when under the irradiation of the electron beam. We chose to investigate the impact of liquid thickness on the maximum attainable spatial resolution of a heavily studied metal-organic framework (MOF), UiO-66, which possess a face-centered cubic crystal structure and has a critical fluence of ~20 e-/Å2, which necessitates the low flux conditions to avoid significant structural degradation during LPTEM. We demonstrate that by tailoring the liquid layer thickness by fabricating different liquid cell window geometries, we have confirmed drastic changes in spatial resolution and the SNR when imaging beam-sensitive samples. Furthermore, our experimental findings closely match predicted spatial resolution limitations which were evaluated by theoretical equations and simulated LPTEM micrographs. The findings presented in this work suggest the necessity to move from the traditional dual liquid cell chip setup in favor of nanochannels (Insight Chips) with controlled liquid cell thickness to enable the possibility of atomic resolution at low fluxes. In the future, we hope that these conclusions and approaches can be expanded to other electron-beam-sensitive materials, including but not limited to covalent-organic frameworks (COFs), polymers, and potentially peptides and proteins.

Dose Fractionated EELS: Pushing the limits of Low-Dose Experiments

Stephen Mick, Gatan Inc.

Direct-detection EELS with the GIF Continuum enables quantitative spectroscopy of challenging beam-sensitive materials. Several innovations are fundamental to enabling this capability including ultrafast camera readout, real-time electron counting, high-speed hardware synchronization, continuous drift correction, and multi-pass data collection. Potassium sodium niobate (KNN) was mapped over a 3000 eV energy range using the Stela detector at 9000 spectra/s. Low-loss and core-loss edges were captured via DualEELS in a multi-pass, high-speed acquisition. The data was drift-corrected, accumulated, and displayed as a chemical map in real-time (Figure 1). This demonstrates that high-quality, high-energy-resolution EELS can be executed while maintaining dose e iciency suitable for beam-sensitive oxides. SrRuO (SRO) / DyScO (DSO) heterostructures is a combination of the highly beam-sensitive SRO complex oxide grown on a DSO radiation insensitive substrate. In fact, SRO is so beam sensitive that atomic resolution EELS is not possible at room temperature. To study the chemistry of this sample and the interface between the epitaxial film and substrate, highspeed, multi-pass, direct detection EELS data must be collected at liquid nitrogen temperatures, but when this is done, Figure 2 shows that atomic resolution EELS is indeed possible. These two examples along with several other experiments will be discussed to demonstrate the impact and need for modern

Figure 1: KNN with 45pA beam current and 9000 spectra/sec

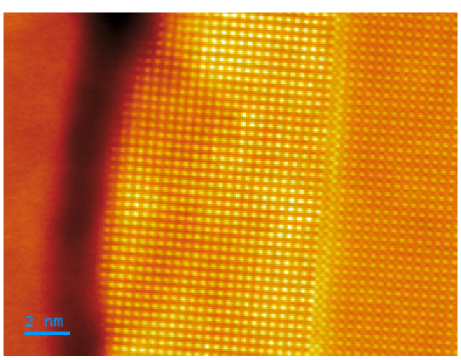
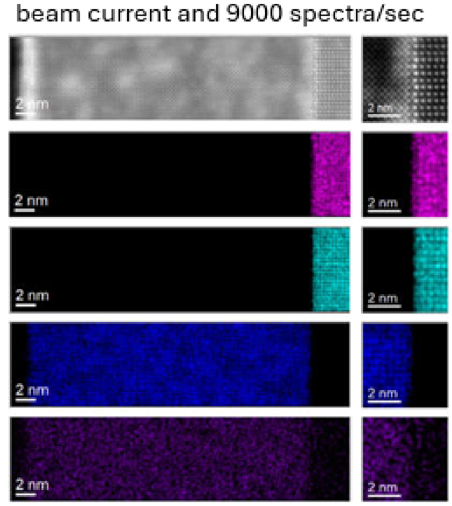


Figure 2: SrRuO3 / DyScO3 with 20pA beam current and 9000 spectra/sec



Pushing the Limits of In-Situ Electron Microscopy: Enabling Atomic-Resolution Imaging and Chemistry in 2D-Material Liquid Nanoreactors

Shoaib Masood, University of Illinois Chicago

In-situ electron microscopy provides a unique platform to directly visualize dynamic processes in liquid environments. In this talk, I will present recent advances that enable atomic-resolution imaging using 2D-material based liquid nanoreactors. These atomically thin platforms open new opportunities to observe early-stage nucleation and single-atom dynamics in liquids with unprecedented spatial resolution. I will further discuss how achieving such resolution requires a quantitative understanding of electron-liquid interactions, including radiolysis chemistry and scattering effects arising from liquid thickness and confinement. By combining high-resolution imaging with spectroscopic analysis, we establish a pathway toward controlled, atomic-scale observation of chemical processes in liquid-phase electron microscopy.

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iPE4Pilot: integrated Process Engineering for Pilot-ready Cathode Production Enabled by In Situ Synchrotron X-ray and Electron Microscopy

Feng Wang, Argonne National Laboratory

Batteries underpin energy storage across consumer electronics, electrified transportation, grid storage, and rapidly growing data-center infrastructure. Achieving safer, higher-performance batteries hinges not only on discovering new electrode materials but also on understanding how material structure and chemistry evolve during synthesis, processing, and operation across scales. Such insight is key to bridging laboratory breakthroughs and pilot-scale manufacturing. Advanced in situ and correlative characterization is making this bridge possible by revealing, in real time, how materials form and function—and by translating those observations into actionable guidance for process engineering.

In this talk, I will overview recent advances in in situ and correlative synchrotron X-ray and electron microscopy/spectroscopy that address critical research needs in battery materials development and engineering. I will illustrate how these approaches generate quantitative, multiscale datasets that connect reaction pathways and microstructural evolution to process engineering protocols. Examples will be drawn from our latest work — particularly from iPE4Pilot, our integrated Process Engineering platform designed to enable Pilot-ready production of coated and doped cathode powders for industry-relevant cell prototyping.

By combining in situ diagnostics, automated image/spectrum analysis, and predictive modeling, iPE4Pilot establishes a data-centric pathway from mechanistic discovery to scalable manufacturing. The resulting process–structure–performance knowledgebase aims to shorten the path from mechanistic discovery to scalable manufacturing, driving battery innovation from research bench to the production line.

Liquid Phase Electron Microscopy for High-Temperature Electrochemical Studies

Thilini Umesha Dissanayake, University of Illinois Chicago

Understanding electrochemical processes at elevated temperatures is critical for advancing next-generation energy storage systems, particularly magnesium-ion batteries, which requires high-temperature cycling of cathode material to enhance reversible Mg^{2} intercalation and long-term stability. While ex-situ studies have demonstrated improved performance in cathode materials such as α-V_2O_5 and MgV_2O_5, the mechanisms driving these transformations at high temperatures remain unclear due to the absence of in-situ characterization tools operating under realistic conditions. Therefore, a modified liquid cell holder was developed by integrating a heating circuit in addition to the biasing circuit to enable simultaneous electrochemistry and heating, The benchmarking experiments using the standard Fe^{2} /Fe^{3} redox system demonstrated stable electrochemistry data at high temperatures with cyclic voltammetry. Further studies with the Cu^{2} plating/stripping system visualized temperature-dependent deposition and stripping processes, with increased Cu deposition at higher temperatures due to increased reaction rates and mass transportation. Overall, this dual-function LC holder provides a powerful platform for direct visualization of temperature-dependent electrochemical processes and advancing the study of high-performance battery systems.

RISE Microscopy: Correlative Raman–SEM Integration for Multimodal Micro- and Nanoscale Characterization

Miguel Blanco, CIQTEK America

Confocal Raman imaging and scanning electron (RISE) microscopy, when combined in a microscope, complement each other and provide the emerging opportunities to clarify morphological, structural, and chemical information of materials at the micron and even nanoscale. Specifically, such advanced RISE microscopy enables the elucidations of one region of interest from a perspective of multiple characterization methods rather than the routine sample-based analysis, representing a major leap in comprehensively characterizing samples. As an emerging advanced spectral analysis method, RISE microscopy has been widely used in energy storage, catalysis, and environmental science as well as in other fields due to its powerful ability of comprehensive analysis in the same region of a sample.

The technique combines SEM and Raman microscopy, a mutually beneficial pairing, allowing not only the high-resolution morphological information obtained by SEM but also the chemical and structural analyses of specified areas by Raman, with the distribution of each phase can also be visually described in a 2D image. More importantly, the confocal function also allows for rapid 3D imaging of Raman spectra by scanning layer by layer in three dimensions with an optical objective lens. A promising future of RISE microscopy is highly expected in physical science, particularly in characterizing advanced functional materials.