

Midwest Microscopy and Microanalysis Society



Electron Microscopy for Materials Research: Recent Developments and Future Opportunities

Agenda:

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| 8:00 AM: Registration | 1:30 PM: Nigel Browning |
| 8:50 AM: Welcome and Opening Remarks | Observing the Atomic Scale Kinetics of Dynamic Processes in Liquids and Gasses by Transmission Electron Microscopy |
| 9:00 AM: Vinayak Dravid | 2:15 PM: Robert Klie |
| Making a Mountain Out of a Molehill: Electron Microscopy for Materials Research at Northwestern | In-Situ Materials Characterization at High Spatial Resolution: 2D Materials Based Liquid-Cell Microscopy |
| 9:45 AM: Jim Zuo | 3:00 PM: Coffee Break |
| The Future of 4D Materials Science Using Electron Beams | 3:20 PM: Yue Li |
| 10:30 AM: Coffee Break | Controlled Rotation Tomography for Scanning Probe Microscopy |
| 10:50 AM: Kai He | 3:40 PM: Paul Voyles |
| Probing nanoscale dynamic materials transformation and electrochemistry in lithium ion batteries by in situ electron microscopy | High Precision STEM Imaging of Cation Vacancies in Oxides and Nanocatalyst Surfaces |
| 11:35 AM: Ben Myers | 4:25 PM: Closing Remarks |
| Orientation Mapping by Electron Channeling: Stage-Rocked Electron Channeling in a Conventional SEM | |
| 12:05 PM: Lunch and Visit with Vendors | |

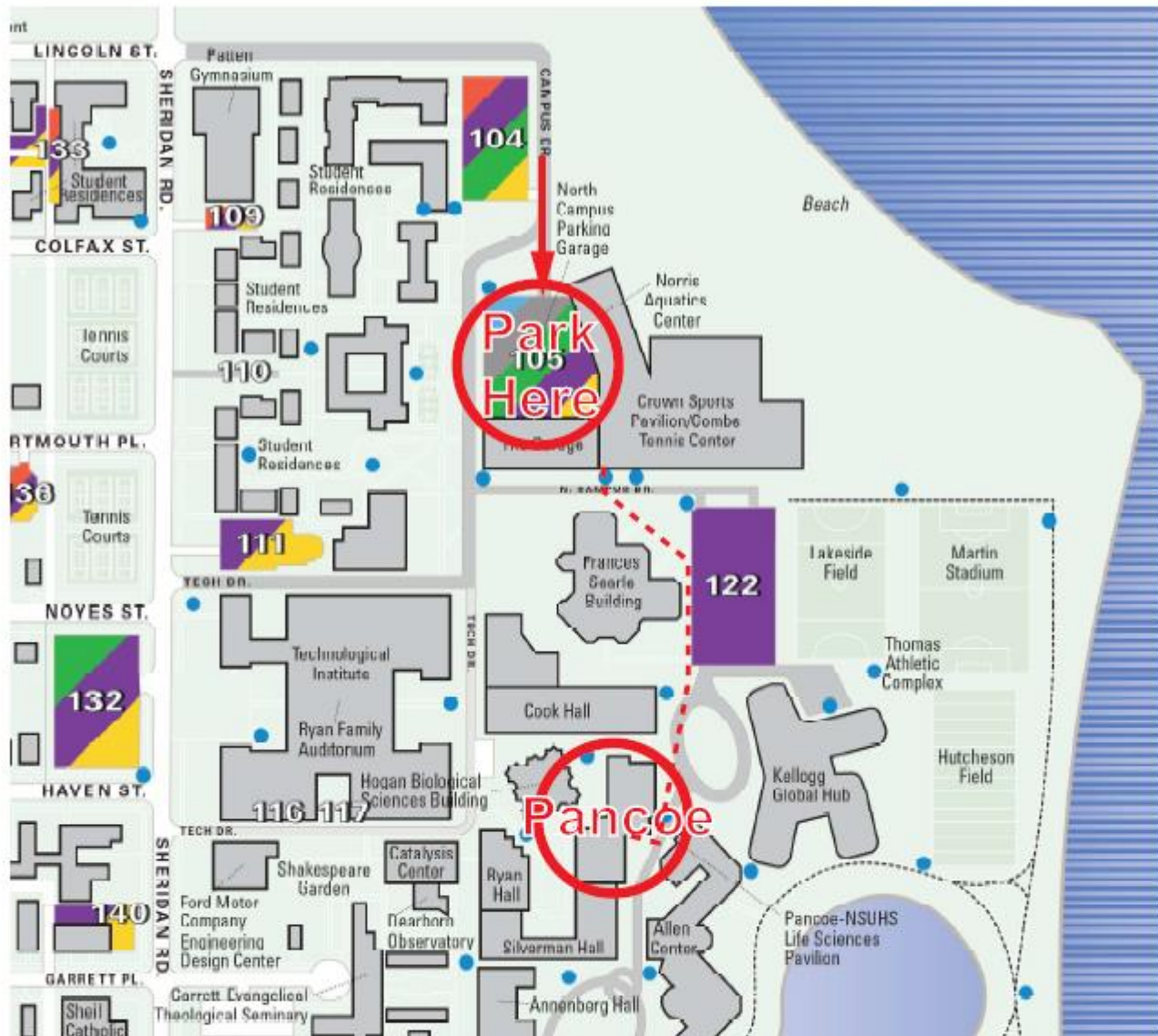
Free for M³S members
\$5 for students; \$20 for non-members
Fee includes 2018 M³S membership
RSVP by emailing:
secretary@midwestmicroscopy.org

Friday, March 30

8 am - 4:30 pm

Northwestern University - Evanston campus
Pancoe Hall | Abbott Auditorium

M³S Meeting Parking Directions



1) Park in the North Campus Parking Garage:
2311 Campus Drive, Evanston IL
Vehicle entrance is on the North side of garage
Parking is \$8 - kiosks are on the ground floor

2) Exit on ground level and walk to Pancoe Hall
Registration will be on the first floor

Making a Mountain out of a Molehill:
Electron Microscopy for Materials Research at Northwestern

Vinayak Dravid
The NUANCE Center, Department of Materials Science & Engineering
Northwestern University, Evanston, IL 60208

Abstract

Electron microscopy, particularly, scanning transmission and transmission electron microscopy (S/TEM) have been enjoying a *renaissance era*; thanks to the convergence of high-brightness field emission sources, aberration-corrected lenses and myriad of *in-situ* or *in-operando* specimen stages, to name major developments in past 2+ decades. Yet, the fundamental electron-specimen interactions remain the same ever since. Here too, there has been steady and improved understanding of the physics of electron scattering and associated phenomena. All of this have greatly helped refine models and mechanisms. Perhaps most importantly, electron microscopy jargon has become a lexicon in broader and diverse scientific conversation; which has greatly advanced its acceptance alongside typical x-ray scattering, optical and related “turn-key” techniques.

The NUANCE Center has always been at the vanguard of these developments in advances in electron microscopy. This presentation will cover brief historical journey of the NUANCE center from its inception to rapid growth to maturity as a national node of facility excellence under the NSF-NNCI program. Several recent examples of advancing materials research using conventional and advanced electron microscopy will be presented; ranging from *in-situ* dynamics of nanostructured materials to performance of the recently installed aberration-corrected S/TEM.

Finally, future prospects for NUANCE, especially under the setting of brand new A/B In-fill building, will be outlined. A case will be made that the best days of NUANCE are still ahead!

The Future of 4D Materials Science Using Electron Beams

Jian-Min Zuo

Dept of Materials Science and Engineering and Materials Research Laboratory
University of Illinois, Urbana-Champaign

I will highlight several exciting developments in my research group and at Materials Research Laboratory toward 4D and in-situ characterization of materials' microstructure:

- At atomic scale, with funding from NSF, an environmental TEM is installed at MRL, UIUC. The microscope is equipped with a direct detection electron camera capable of recording at 400 frames per second. Using this instrument, we have examined nanostructure transformation dynamics at milliseconds time resolution, oxidation and support interactions of nanocatalyst. The need for dose efficient high resolution electron microscopy will be highlighted.
- At nanometer-scale, we have developed a high resolution orientation mapping technique based on electron nanodiffraction for the determination of 3D nanostructure. Potential applications include correlative microscopy with APT or STEM/EDX for complete 3D composition and structure determination.
- By combining in-situ nanoindentation with observation of dislocations, we have successfully correlated dislocation multiplications, motions and interactions with nanoscopic mechanic properties that shines critical insights about collective dislocation motions and avalanches.

The needs for further development of better detectors, electron source and algorithms will be highlighted in the discussions.

About Prof. Jian-Min Zuo

Jian-Min Zuo received his Ph.D. in Physics from Arizona State University in 1989. Prior to joining the faculty at the University of Illinois, he was a research scientist in Physics at ASU and a visiting scientist to a number of universities and institutes in Germany, Japan and Norway. He currently holds Ivan Racheff Professorship at University of Illinois. Prof. Zuo has developed research programs focused on electron diffraction, high resolution electron microscopy and structure and property relationship studies in a broad range of materials. He is a fellow of American Physical Society and the author of a recent book on advanced transmission electron microscopy.

Probing nanoscale dynamic materials transformation and electrochemistry in lithium ion batteries by in situ electron microscopy

Kai He

Northwestern University

Understanding the physical and chemical processes occurring at nanoscale environments is essential to a variety of scientific disciplines. Recent advancement of aberration correction has enabled routine atomic resolution imaging and spectroscopy using transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) methods. Further, combined with modern in-situ approaches, they have been proven to be powerful for probing dynamic structural, chemical and functional evolutions in real time. Here, I will present our recent research effort on development of in-situ analytical S/TEM techniques and their implementations in energy storage materials, in particular, to unveil the phase transformation during electrochemical reactions upon battery charge/discharge and their correlation to realistic battery performance. We have investigated electrode materials for lithium ion batteries with different crystal structures, from simple cubic structure with no interstitial space, to spinel and layered structures with vacant openings, and we found multiple lithiation modalities with distinct reaction pathways, which illustrated that the kinetic effect plays a critical role in realistic electrochemical conditions where thermodynamic nonequilibrium is generally applied. The knowledge gained from the in-situ experiments has addressed key questions to leverage the structure-pathway-property relationship and provides mechanistic insight into future battery design and manufacture.

Benjamin D. Myers

SHyNE Resource and Department of Materials Science and Engineering
Northwestern University

Orientation Mapping by Electron Channeling (OMEC): Stage-Rocked Electron Channeling in a Conventional SEM

Abstract: Orientation mapping of bulk, polycrystalline samples has become a common technique due to the rapid development and dissemination of electron backscatter diffraction (EBSD). Electron channeling-based techniques, such as those based on selected area channeling patterns (SACP) actually predate EBSD, but never gained popularity as they require specialized electron optics and have other limitations. In this presentation, I will describe a new technique for the generation of electron channeling patterns (ECP) by "rocking" the microscope stage. This method, called orientation mapping by electron channeling (OMEC), relies on automated image acquisition and computational image alignment to produce high-resolution channeling patterns that can be used to generate orientation maps. Unlike the aforementioned techniques, OMEC can be performed on a range of conventional scanning electron microscopes (SEM) without the need for additional hardware as required for EBSD and SACP. In addition, the technique may offer new possibilities for application of intelligent sampling routines, data-mining for electron channeling contrast imaging of dislocations and strain-mapping by HOLZ line analysis.

Bio: Ben Myers is the Director of Operations for SHyNE Resource, the joint Northwestern/U Chicago NSF-NNCI site. He received a BS in Materials Science and Engineering from the University of Illinois at Urbana-Champaign in 2000 and a MS in Materials Science and Engineering from Northwestern University in 2006 (PhD expected in 2018). Ben began his career in the semiconductor industry at Motorola SPS as a metals process engineer. Ben joined Northwestern in 2002 in a joint position with the NUANCE Center and the Department of Materials Science and Engineering. In this role, he managed the SEM and FIB instruments in the NUANCE Center and coordinated lab courses on SEM. In his current role, Ben leads the SHyNE external user program and coordinates activities across several core facilities. His research interests span from materials characterization to nanofabrication and nanoscale self-assembly.

Observing the Atomic Scale Kinetics of Dynamic Processes in Liquids and Gases by Transmission Electron Microscopy

Nigel D. Browning

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Many processes in materials engineering, physics, chemistry and biology take place in a liquid or gas environment. In these cases, the final desired outcome of the process is a result of a series of complicated transients, where a change in the order, magnitude or location in each of the steps in the process can lead to a radically different result. Understanding and subsequently controlling the final outcome, for example in the design of an efficient catalytic reaction, therefore requires the ability to directly control and observe the kinetics of these transients as they happen. The transmission electron microscope (TEM) has the potential to observe precisely these kinetic processes on the atomic and molecular scale. To accomplish this, in-situ gas and liquid stages are incorporated into the microscope and images are acquired under a compressive sensing/machine learning optimization of the dose/data content. In this presentation, the unique in-situ methodologies employed to study dynamic chemical reactions in the aberration corrected scanning transmission electron microscope (STEM) at the University of Liverpool will be described. New results showing the use of in-situ liquid stages to study nucleation and growth using inpainting will be presented and the potential insights that can be gained by increasing the image acquisition speed and/or decreasing the electron dose will be described. Importantly for all in-situ observations, the kinetic control of the nucleation and growth process using sub-sampling highlights the role of interfaces in controlling the process. Sub-sampling and inpainting is not limited to STEM (or even to electron microscopy) as similar methods can be used in TEM mode to increase the speed of any camera. The potential to apply these methods together to extract quantitative image information from a wide range of images used for engineering/medical applications will also be discussed.

Nigel Browning is currently chair of Electron Microscopy at the University of Liverpool. He received his undergraduate degree in Physics from the University of Reading, U. K. and his Ph. D. in Physics from the University of Cambridge, U. K. After completing his Ph. D. in 1992, he joined the Solid State Division at Oak Ridge National Laboratory (ORNL) as a postdoctoral research associate before taking a faculty position in the Department of Physics at the University of Illinois at Chicago (UIC) in 1995. In 2002, he moved to the Department of Chemical Engineering and Materials Science at the University of California-Davis (UCD) and also held a joint appointment in the National Center for Electron Microscopy (NCEM) at Lawrence Berkeley National Laboratory (LBNL). In 2005 he moved the joint appointment from LBNL to Lawrence Livermore National Laboratory (LLNL) to become project leader for the Dynamic Transmission Electron Microscope (DTEM). In 2009, he also joined the Department of Molecular and Cellular Biology at UCD to focus on the development of the DTEM to study live biological structures. From 2011-2017 he was a Laboratory Fellow and Lead of the Chemical Imaging Initiative at the Pacific Northwest National Laboratory (PNNL).

He has over 25 years of experience in the development of new methods in electron microscopy for high spatial, temporal and spectroscopic resolution analysis of engineering and biological structures. His research has been supported by DOE, NSF, NIH, DOD and by industry, leading to research projects for over 30 graduate students and 35 postdoctoral research fellows. He is a Fellow of the American Association for the Advancement of Science (AAAS) and the Microscopy Society of America (MSA). He received the Burton Award from the Microscopy Society of America in 2002 and the Coble Award from the American Ceramic Society in 2003 for the development of atomic resolution methods in scanning transmission electron microscopy (STEM). With his collaborators at LLNL he also received R&D 100 and Nano 50 Awards in 2008, and a Microscopy Today Innovation Award in 2010 for the development of the dynamic transmission electron microscope (DTEM). He has over 350 publications (h-index=69) and has given over 300 invited presentations on the development and application of advanced TEM methods.

R.F. Klie
The University of Illinois at Chicago

In-Situ Materials Characterization at High Spatial Resolution: 2D Materials Based Liquid-Cell Microscopy

Xuan Hu, A. Mukherjee, J. Jokisaari, C. Wang and R.F. Klie¹

¹ University of Illinois at Chicago, Dept of Physics, Chicago, IL 60607

The last few years have seen a paradigm change in scanning transmission electron microscopy (STEM) with unprecedented improvements in both spatial and spectroscopic resolution being realized by aberration correctors, cold-field emission guns and monochromators. Aberration correction also allows increased flexibility in choosing the appropriate electron energy to minimize beam-induced damage while maintaining atomic-resolution (e.g. 60 keV electrons for studying graphene with 1.3 Å resolution).[1] These developments have enabled a revolution in resolution, which now allows us to perform *in-situ* or *operando* measurements at, or close to, the limits dictated by sample properties rather than the instrumentation resolution.

We have recently developed a novel approaches towards in-situ electron microscopy, including EELS-based thermometry allowing the thermal properties to be mapped with nm-resolution and novel liquid-cell configurations that allow the effects of radiolysis to be examined on the atomic-level scale. [2, 3] In this talk, I will demonstrate how 2D materials, such as graphene and transition metal dichalcogenides (TMD), can be used to encapsulate a small volume of liquid,[4] measure the local thermal expansion coefficient and enable phonon spectroscopy of liquids with unprecedented spatial resolution.

References

- [1] Zhou, W. et al. Nature Nanotechnology, 7(3),161-165 (2012)
- [2] Wang, C., Q. et al., Advanced Materials, 26,3410–3414 (2014)
- [3] Wang, C., T. Shokuhfar, and R.F. Klie, Advanced Materials, 28(35), 7716 (2016)

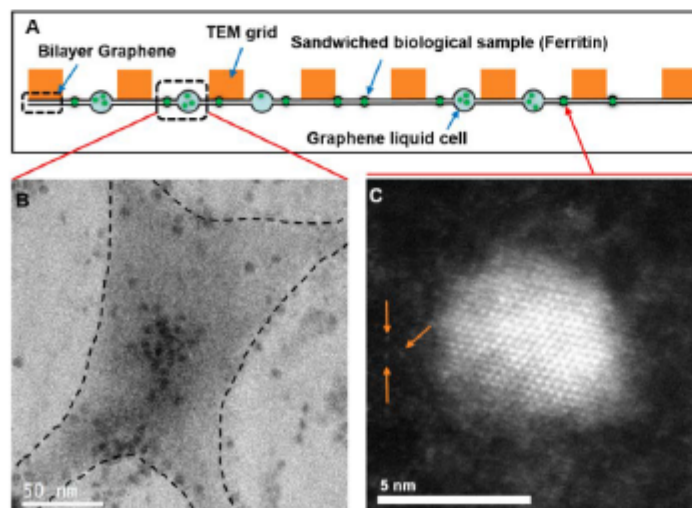


Figure 3: Schematic diagram (A), as well as STEM images (B) and (C) of ferritin molecules in GLCs and graphene sandwiches. (B) is an annular bright field (ABF) image showing ferritin molecules encapsulated in both a GLC and graphene layers. The edges of the GLC is indicated by dash lines. (C) is a high angle annular dark field (HAADF) image showing atomic-resolution image of a sandwiched ferritin molecule, with the 12nm in diameter protein shell and individual Fe

Yue Li
Northwestern University

Ph.D. student presentation:

Controlled Rotation Tomography (CORT) for Scanning Probe Microscopy

Abstract: A novel sampling strategy, controlled rotation tomography (CORT), was developed for tomography tilt series collection for scanning probe microscopy. Traditionally, the sample is parked at different orientation for each projection image collection. In CORT, the sample rotates at preset speeds continuously during the probe scanning. With both synthetic data and metadata generated from real experiments, we showed that CORT has a more even coverage in the sinogram. Combined with inpainting algorithms in the sinogram domain, and a penalized maximum likelihood (PLM) tomography reconstruction algorithm, CORT showed superior reconstruction quality with significantly reduced electron dose.

Bio: Yue Li is a Ph.D candidate in the Applied Physics Program. She is co-advised by Prof. Vinayak Dravid in Materials Science and Prof. Vadim Backman in Biomedical Engineering. Her research focuses on the visualization of nanoscale chromatin architecture by low-dose tomography reconstruction, and the quantification of fractal dimension alterations of chromatin organization in field carcinogenesis.

Paul M. Voyles

University of Wisconsin, Madison

High precision STEM Imaging of Cation Vacancies in Oxides and Nanocatalyst Surfaces

Jie Feng, Chenyu Zhang, Dane Morgan, **Paul M. Voyles**

High precision scanning transmission electron microscopy (STEM) uses advanced non-rigid registration algorithms to produce extremely high signal-to-noise ratio, extremely low distortion STEM images of radiation-robust specimens. Using high precision STEM, we can locate a column of atoms in an image to <1 pm uncertainty and count the number of atoms in the column with less than one atom random uncertainty. We have used high precision STEM to image La vacancies in LaMnO_3 and study the displacements of surface atoms on a Pt nanocatalyst. We identify single La vacancies by imaging both the reduced intensity of the atomic column caused by the missing atom and the shifts of the neighboring columns in toward the vacancy. By combining these distinct measurements, we can distinguish surface vacancies left over from TEM sample preparation from bulk vacancies intrinsic to the material and localize bulk vacancies to within one or two potential atomic sites in three dimensions. Pt nanocatalysts on a SiO_2 support show substantial contraction of the corner between two flat $\{111\}$ facets toward the center of the particle, consistent with previous reports, and expansion of the $\{111\}$ facets, consistent with DFT calculations. A web app for high precision STEM data processing is available at www.nanohub.org/resources/nrr.