WORKSHOP #9

Upcoming New Soft X-ray Nanoscale Imaging and Spectromicroscopy Capabilities at NSLS-II

Organizers: Wen Hu (NSLS-II), Brandy Toner (University of Minnesota), Andrew Walter (NSLS-II), Joe Dvorak (NSLS-II), Jiemin Li (NSLS-II)

The Soft X-ray Nanoprobe (SXN) beamline, a new beamline under construction at NSLS-II, will offer researchers state-of-the-art soft X-ray nano-imaging and spectroscopy capabilities with world-leading coherent high photon flux in the energy range from 250 eV – 2500 eV. This two half-day workshop aims to bring experienced X-ray microscopy users, as well as new and early career users seeking practical guidance, together for scientific, technical, and training presentation and discussions. The first half-day workshop focuses on discussing the next generation of science questions that can be addressed with advances in instrument capabilities (Day-1). We aim also to discuss science cases suitable for the commissioning/optimization phase of the SXN beamline. The second half-day workshop focuses on practical aspects of X-ray imaging and spectromicroscopy research (Day-2) and discussion of existing approaches and requirements including sample preparation, choosing the best instrument for your research question, research workflow, data handling and multi-modal approaches.

Thursday, May 16, 2024 (1:00 - 5:30 p.m.)
Session I: Research Areas (overview of high impact science cases of soft X-ray nanoscale Imaging and Spectromicroscopy)

Start Time (ET)	Title	Speaker (Affiliation)
1:05 - 1:15	Opening Remarks	Andrew Walter (NSLS-II)
	Chair: Joe Dvorak Moderator: Jiemin Li	
1:15 – 1:45 (1:45-2:00)	Upcoming Soft X-ray Nanoprobe Beamline at NSLS-II (Q&A)	Wen Hu (NSLS-II)
2:00 - 2:30 (2:30 - 2:45)	Opportunities for STXM and Spectro-ptychography at the Soft X-ray Nanoprobe beamline (Q&A)	Adam Hitchcock (McMaster University)
2:45 - 3:00	Break and Group photo	
	Chair: Wen Hu Moderator: Joe Dvorak	
3:00 - 3:30 (3:30 - 3:45)	Beyond the Surface: STXM as a Versatile Tool for Investigating Emergent Phenomena in Spintronics (Q&A)	Kai Litzius (University of Augsburg)
3:45 - 4:15 (4:15 - 4:30)	Mineral-organic matter interactions in aquatic systems: the need for integrated, multi-modal imaging and spectroscopy (Q&A)	Brandy Toner (University of Minnesota)
4:30 - 5:30	Discussion and summary of session I Chair: Wen Hu Moderator: Andrew Walter	All speakers and attendees
5:30	Adjourn	

Friday, May 17, 2024 (1:00 - 5:30 p.m.)
Session II: Application (details from sample preparation, experimental design to data analysis and interpretation)

Start Time (ET)	Title	Speaker (Affiliation)
1:05 - 1:15	Welcome and Introduction of session II	
	Chairs: Brandy Toner Moderators: Andrew Walter	
1:15 - 1:45 (1:45 - 2:00)	Applying STXM in multi-modal environmental science investigations: from sample preparation to publication (Q&A)	Sarick Matzen (University of Minnesota)
2:00 - 2:30 (2:30 - 2:45)	Data Analysis in spectroscopic STXM (Q&A)	Matthew A. Marcus (ALS/LBNL)
2:45 - 3:00	Break and Group photo	
	Chairs: Andrew Walter Moderators: Brandy	
3:00 - 3:30 (3:30 - 3:45)	Nanoscale scanning x-ray microscopy: a versatile tool for material characterization in 2D and 3D (Q&A)	Hanfei Yan (NSLS-II)
3:45 - 4:15 (4:15 - 4:30)	Using STXM and Spectro-ptychography to Study Energy Materials for Electrochemical Applications (Q&A)	Chunyang Zhang (McMaster University)
4:30 - 5:30	Breakout discussion and Closing Remarks Chairs: Wen Hu/Brandy Toner/Joe Dvorak/Jiemin Li Moderator: Andrew	All speakers and attendees
5:30 pm	Adjourn	

ABSTRACTS (as available to date)

Upcoming New Soft X-ray Nanoscale Imaging and Spectromicroscopy Capabilities at NSLS-II

Wen Hu
NSLS-II, Brookhaven National Laboratory, Upton NY, USA

Abstract: The Soft X-ray Nanoprobe (SXN) beamline is part of the DOE-funded NEXT-II project to deliver 3 additional "best-in-class" imaging beamlines to NSLS-II. It will offer researchers state-of-the-art soft x-ray nano-imaging and spectroscopy tools with world-leading coherent high photon flux in the energy range from 250 eV – 2500 eV and full polarization control. It will provide element access from carbon (C) to sulfur (S) through K-edges and many other important elements through L- and M-edges. The primary endstation, nanoISM, will offer both a conventional scanning transmission x-ray microscopy (STXM) mode, for high throughput 2D/3D absorption imaging, and a coherent diffractive imaging (ptychography) mode, for extra high spatial resolution. In this talk, I will give an overview of the design and status of the SXN beamline and discuss the future potential of the nanoISM endstation at SXN beamline, NSLS-II.

Opportunities for STXM and Spectro-ptychography at the Soft X-ray Nanoprobe beamline

Adam P. Hitchcock
Dept. of Chemistry & Chemical Biology, Brockhouse Institute for Materials Research, McMaster University, Hamilton, ON L8S 4M1, Canada

Abstract: Soft X-ray spectromicroscopy provides excellent chemical sensitivity through near edge spectroscopy (NEXAFS), geometric and magnetic anisotropy through linear and circular dichroism, and the highest spatial resolution of all synchrotron-based imaging methods. The new Soft X-ray Nanoprobe (SXN) beamline presently under construction at NSLS-II will provide state-of-the-art capabilities. I will outline research challenges in materials science, energy, bio-geo-chemistry and biology that this new facility will help tackle. Based on the current trend of rapid development of *in situ* and *operando* techniques, I expect these methods to be an important feature of this premier new facility.

Beyond the Surface: STXM as a Versatile Tool for Investigating Emergent Phenomena in Spintronics

Kai Litzius
University of Augsburg, Augsburg, Germany

Abstract: Scanning Transmission X-ray Microscopy (STXM) harnesses the X-ray Magnetic Circular Dichroism (XMCD) effect, a phenomenon where the absorption of X-rays depends on the magnetic orientation of the material, enabling high-resolution imaging and spectroscopy with elemental and chemical specificity. In this talk, we focus on the capabilities and the applications of STXM for unraveling emergent phenomena in spintronics, focusing on current-induced skyrmion dynamics and the investigation of 2D magnets.

The investigation of current-induced skyrmion dynamics holds promise for next-generation data storage and processing applications owing to the topologically protected stability and nanoscale size of skyrmions. STXM offers unparalleled capabilities for directly visualizing skyrmions at the nanoscale while at the same time allowing for versatile excitation mechanisms, shedding light on their formation, motion, and manipulation under the influence of, e.g., electric currents.

Furthermore, the study of 2D magnets presents intriguing prospects for understanding and exploiting novel magnetic properties in atomically thin materials. STXM enables precise characterization of magnetic ordering and domain structures in these materials, clarifying their fundamental magnetic interactions and spin configurations. Through imaging the magnetic response of 2D magnets to external stimuli such as electric fields or strain, STXM facilitates the design and optimization of spintronic devices based on these materials.

This discussion delves into recent advancements in STXM techniques for investigating current-induced skyrmion dynamics and the unique magnetic properties of 2D magnets. Through a combination of experimental observations and theoretical insights, we aim to unveil the underlying mechanisms governing these emergent phenomena and explore their potential applications in future spintronic devices.

Further Reading:

1. G. Schütz *et al.* Magnetism studied with circularly polarized X-rays. *Int. J. Mater. Res.* **102**, 773–783 (2011).
2. H. Stoll *et al.* Imaging spin dynamics on the nanoscale using X-Ray microscopy. *Front. Phys.* **3**, 1–18 (2015).
3. S. Finizio *et al.* In situ membrane bending setup for strain-dependent scanning transmission x-ray microscopy investigations. *Rev. Sci. Instrum.* **87**, 123703 (2016).
4. K. Litzius *et al.* Skyrmion Hall effect revealed by direct time-resolved X-ray microscopy. *Nat. Phys. 13*, 170–175 (2017).
5. M. T. Birch *et al.* History-dependent domain and skyrmion formation in 2D van der Waals magnet Fe3GeTe2. *Nat. Commun.* **13**, 3035 (2022).
6. L. Powalla *et al.* Single Skyrmion Generation via a Vertical Nanocontact in a 2D Magnet-Based Heterostructure. *Nano Lett.* **22**, 9236–9243 (2022).

Mineral-organic matter interactions in aquatic systems: the need for integrated, multi-modal imaging and spectroscopy

Brandy M. Toner
University of Minnesota

Abstract: The Earth and environmental science (EES) community needs increased access to scanning transmission X-ray microscopy (STXM) instruments in North America. The tender X-ray elements (e.g. P, S) are important for many EES research questions but these energies are underserved in the North American synchrotron facilities. Samples from natural systems are complex and heterogeneous. STXM applications for EES require fast, efficient, and sensitive mapping of elements. Fluorescence detection and reliable coarse and fine stages are needed. Photon damage to natural samples, especially those rich in

organic C, require special handling and sample cooling may provide some scientific benefits. Automatic or robotic sample transfer, along with remote operation, could provide increased accessibility to the instrument for disabled researchers and researchers from non-R1 institutions, as well as reduce greenhouse gas emissions typically emitted due to travel for research teams. Multi-modal research will be enhanced with sample compatibility with transmission electron microscopes. Ptychography would be valuable for some studies to provide the highest spatial resolution for X-ray microscopy. Overall, reliable sample exploration, reliable spectromicroscopy from C 1s to S 1s, and remote access will increase scientific discoveries, productivity, and accessibility. The new SXN beamline has the potential to fulfill these research needs for the EES community.

Applying STXM in multi-modal environmental science investigations: from sample preparation to publication

Sarick Matzen
University of Minnesota

Abstract: Synchrotron spectromicroscopy techniques, including Scanning Transmission X-ray Microscopy and ptychography, are powerful tools to inform earth and environmental science investigations, yet such communities make up a small portion of users at relevant beamlines. Our goal here is to help users new to these methods learn to apply them effectively. Using our current work on hydrothermal plume particles as examples, this practical workshop starts with examples of scientific questions that can be answered using these methods, helps users understand sample selection criteria and sample preparation, and reviews data collection and analysis pipelines. Participants will leave with a clear idea of how to embark on exciting new spectromicroscopy work in collaboration with beamline scientists.

Data analysis in spectroscopic STXM

Matthew A. Marcus
Lawrence Berkeley National Laboratory (LBNL), Berkeley, CA (United States)

Abstract: The typical output of a STXM (Scanning Transmission X-ray (spectro)Microscopy) measurement is a data cube consisting of a set of images (measurements of X-ray transmission at a grid of pixels) taken at a sequence of incident energies. As with any experimental measurement, this raw data must be reduced to some standard form and interpreted. In this talk, I will review the basics of how to go from raw data to information about the sample. I will discuss the fundamentals of X-ray spectromicroscopy, data reduction, descriptive and model-based analysis, and available software, with examples taken from the literature and my work.

Nanoscale scanning x-ray microscopy: a versatile tool for material characterization in 2D and 3D

Hanfei Yan
NSLS-II, Brookhaven National Laboratory, Upton NY, USA

Abstract: Hard x-ray nanoprobe beamline of National Synchrotron Light Source II is a scanning microscopy beamline with world-leading spatial resolution. With a beam size of approximately 10-nm, it enables the characterization of elemental, chemical, and structural variations within complex and heterogeneous material systems in both 2D and 3D. Furthermore, it provides sub-10 nm resolution with ptychographic imaging. In this presentation, I will discuss a variety of X-ray imaging techniques available at the beamline for scientific exploration. These include fluorescence mapping, nano-XANES, nano-diffraction, tomography, and ptychography. I will present a few scientific applications such as the study of defects in 3D superlattices, strain mapping in next-generation microelectronics, and tomography for energy storage materials to demonstrate its capabilities. Lastly, I will briefly touch on new developments at the beamline. These include machine-learning assisted tomography with limited-angle, sparse spectro-tomography for 3D chemical imaging, and fast data acquisition.

Using STXM and Spectro-ptychography to Study Energy Materials for Electrochemical Applications

Chunyang Zhang
Dept. of Chemical Engineering, McMaster University, Hamilton, ON L8S 4M1, Canada

Abstract: Scanning transmission X-ray microscopy (STXM) and X-ray spectro-ptychography (SP) are synchrotron-based spectro-microscopic techniques, which provide microscopic imaging and spatially resolved quantitative chemical mapping of chemical states based on near-edge X-ray absorption fine structure (NEXAFS) spectroscopy. Spatial resolution is routinely ~20-40 nm with STXM and ~6-15 nm with SP. In recent years, STXM and SP have been used widely to study materials and processes for electrochemical energy conversion and storage. When compared with characterization approaches relying on only bulk average spectroscopy or spatially localized imaging without spectroscopy, STXM and SP spectromicroscopies are very powerful tools for investigating energy materials which have high structural heterogeneity and complicated chemical composition. I will show some of our recent STXM and SP studies of single-atom Ni electrocatalysts, Cu nanoparticle electrocatalysts and MnO_2 supercapacitor materials. The talk will focus on my practical experiences, including (1) sample selection and preparation (such as drop-cast powders; microtoming; electrodeposition); (2) experimental design (studying active sites, observing chemical/morphological changes associated with electrochemical reactions, and in situ/operando experiments); (3) STXM and SP data analysis.