

# APS Workshop 8: Quantum Solids under a Dark-field X-ray Microscope: A Journey from Imagination to Discovery at APS-U

**Tuesday, May 7, Morning**

8:30 – 8:35 Organizing Committee  
*Introduction and Welcome*

## **Session I: State-of-the-art Instrumentation** **Chair: Jon Tischler**

8:35 – 8:40 Introduction by Chair

8:40 – 9:10 Zhan Zhang (Argonne National Laboratory)  
*Application of Dark-field X-ray Microscopy on Epitaxial Thin Film*

9:10 – 9:40 Peter Kenesei (Argonne National Laboratory)  
*Future Multiscale Microstructural Study Opportunities at the APS's High-energy X-ray Microscope (HEXM) Beamline including Dark-field Microscopy*

9:40 – 10:10 Daniel Gianola (University of California, Santa Barbara)  
*Characterizing Phenomena in Metals and Alloys at the Single Defect Level: Insights from Electron Microscopy*

10:10 – 10:25 Break

## **Session II: Quantum Solids under a Dark-field X-ray Microscope** **Chair: Jessica McChesney**

10:25 – 10:30 Introduction by Chair

10:30 – 11:00 Jayden C. Plumb (University of California, Santa Barbara)  
*Charge Order Investigation of  $CsV_3Sb_5$  with Dark-field X-ray Microscopy*

11:00 – 11:30 Kaan Alp Yay (Stanford University)  
*Observation of Strain Wave in the Nematic Phase of the Iron Pnictide  $Ba(Fe_{1-x}Cu_x)_2As_2$  Using Dark-field X-ray Microscopy*

## **Session III: Discussion** **Moderators: Paul Fenter and John Freeland**

11:30 – 12:00 Discussion  
*Potential First Experiments*

12:00 – 1:30 Lunch Break

## **Tuesday, May 7, Afternoon**

### **Session IV: Functional Devices**

**Chair: Hao Zheng**

- 1:30 – 1:35 Introduction by Chair
- 1:35 – 2:05 Elliot Kisiel (University of California, San Diego)  
*Dark-field X-ray Microscopy for Quantum Materials and its Future at APS-U*
- 2:05 – 2:35 Pavel Salev (University of Denver)  
*Inhomogeneous Strain Development in Metal-to-insulator Transition Switching Devices: Microdiffraction and Dark-field X-ray Microscopy Perspectives*
- 2:35 – 2:50 Break

### **Session V: Novel Development and Directions**

**Chair: Chris Benmore**

- 2:50 – 2:55 Introduction by Chair
- 2:55 – 3:25 Mads Carlsen (Paul Scherrer Institute, Switzerland)  
*Wave-optical Simulation of a Dark-field X-ray Microscope*
- 3:25 – 3:55 Doga Gursoy (Argonne National Laboratory)  
*From Imagination to Realization: A Tale of a Dark-field X-ray Microscope under a Coded-aperture Lens for 3D Imaging*
- 3:55 – 4:25 Takashi Kimura (Institute for Solid State Physics, University of Tokyo, Japan)  
*Development and Application of X-ray Spectromicroscopies Using Achromatic Wolter Mirror Optics*

### **Session VI: Discussion and Wrap-up**

**Moderators: Paul Fenter and John Freeland**

- 4:25 – 5:00 Discussion  
*Potential First Experiments*
- 5:00 Adjourn

## Application of Dark Field X-ray Microscopy in Epitaxial Thin Film

Z. Zhang<sup>1</sup>, Seohyoung Chang<sup>2</sup>, and Hua Zhou<sup>1</sup>

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While the atomic structure is fundamental to the understanding of the static and dynamic properties of functional materials, it was realized that some phenomena emerge because of the collective behavior of a large number of atoms. Domains are typically collections of atoms on nano- to micro-meter length scale, whose formation, interaction, and evolution under external stimuli would dictate success or failure of the material. Studying domains would require tools with good spatial resolution, as well as large enough sampling area/volume. A diffraction based, dark field x-ray microscopy method, the x-ray reflection interface microscopy (XRIM), can be very useful in studying domains at the surfaces, buried interfaces, and inside of thin film material systems, with sub-nanometer sensitivity in the surface normal direction and better than 100 nm lateral resolution.

With the penetrating power of hard x-rays, XRIM can emphasize the features at different depths from the top surface by selecting proper scattering conditions, making it an excellent candidate to study the films *operando* in real time. Combined with the reciprocal space mapping (RSM), the spatially resolved structure evolution can be identified. A couple examples will be discussed to demonstrate the capability of XRIM method and its potential applications in a broader field.

## Future Multiscale Microstructural Study Opportunities at the APS's High-energy X-ray Microscope (HEXM) Beamline including Dark-field Microscopy

Peter Kenesei<sup>1</sup>, Jonathan Almer<sup>1</sup>, Sarvjit Shastri<sup>1</sup>, Jun-Sang Park<sup>1</sup>, Andrew Chuang<sup>1</sup>, John Okasinski<sup>1</sup>, Hemant Sharma<sup>1</sup>, and Ali Mashayekhi<sup>1</sup>

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The APS-Upgrade brings a great opportunity to review past years demands from the user community, and to address these needs with the new beam properties, and state-of-the-art instrumentation along with recently developed and emerging new measurement techniques. High-energy x-rays (above 35 keV) provide sufficient transmission through experimentally relevant size of specimens in many scientific fields, making them a suitable probe to investigate 3D microstructural properties inside the bulk. At the new high-energy x-ray microscope (HEXM) beamline at the APS, a great suite of measurement techniques will be available for detailed and sophisticated studies not possible with other conventional, surface-based interrogation approaches.

With a millimeter-scale overview of the specimen with microtomography ( $\mu$ CT) and near-field and far-field high-energy diffraction microscopy (NF and FF-HEDM) and advanced software tools, it becomes possible to capture and locate rare events in bulk materials occurring at external stimuli. Then zooming into the region of interest with a higher resolution full-field x-ray technique, like dark-field x-ray microscopy (DFXM) or transmission x-ray microscopy (TXM), will reveal more details of the microstructure in situ or operando, without compromising the integrity of the original specimen.

Complementing these with high-energy Bragg coherent diffraction imaging (HE-BCDI) and scanning diffraction techniques, further high-resolution large-scale information can be obtained. The observations and the results of quantitative analysis can be directly used to evaluate, develop, or validate material models or extend understanding of microstructure related mechanisms on performance which are crucial steps before engineering final products.

In this presentation the scientific mission of the future 20-ID HEXM beamline will be shown based on experiences and technique developments at the 1-ID beamline. A quick virtual tour will be presented of the instrument design which will serve mainly energy sciences (nuclear, battery, solar) and materials physics and engineering (aerospace, automotive, medical and civil engineering, functional materials) related research. The planned measurement techniques will be discussed with example use cases.

# Characterizing Phenomena in Metals and Alloys at the Single Defect Level: Insights from Electron Microscopy

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Defects in crystalline solids, as well as the local atomic environments in their direct vicinity, play a central role in plastic deformation and, accordingly, key mechanical and physical properties governed by their formation and motion. More recently, the existence of confined phases that emerge from, and remain anchored to, defects in crystals such as planar faults, grain boundaries and dislocations. These *defect phases* (sometimes referred to as complexions to delineate their differences with bulk phases) can exist in a local equilibrium with respect to the abutting phases and are not predicted by bulk phase diagrams. Provided that the reduced dimensionality of the defect is appropriately considered, the adherence of defect phase equilibria to similar thermodynamic and kinetic principles that govern bulk materials provides a tantalizing pathway towards a "defects by design" framework that inextricably marries phase transitions with crystalline defects aimed at the goal of achieving unexplored property space. While the characterization of these defects represents some of the pioneering early electron microscopy developments, linking the structure and dynamics of defects directly to properties across all relevant scales has been accelerated by recent advances in electron sources, aberration correction, direct electron detectors, *in-situ* instrumentation, and machine-learning based image analyses. Capturing the chemical, structural, and magnetic degrees of freedom within a single-defect micro-environment is necessary to fully identify and describe a defect phase.

In this talk, we highlight recent developments in dislocation characterization across a suite of electron microscopy modalities, including scanning transmission electron microscopy in both TEM and SEM platforms, the latter of which offers versatility for multimodal and *in-situ* testing. We further demonstrate how the richness of information encoded in electron backscattered diffraction (EBSD) patterns is amplified by a new generation of direct electron detectors that enable high speed mapping and acquisition of high-fidelity patterns that can be used for statistically meaningful defect analyses. We will show how these techniques can be employed for *in-situ* experiments to study the nature of dislocations dynamics in several structural metals and alloys, including Heusler alloys hosting distinct spin exchange interactions at defect cores, interstitial-engineered refractory BCC multi-principal element alloys, and FCC alloys hosting local chemical and structural ordering in the vicinity of dislocation cores.

## Charge Order Investigation of CsV<sub>3</sub>Sb<sub>5</sub> with Dark-field X-ray Microscopy

Jayden Plumb<sup>1</sup>, Elliot Kisiel<sup>2</sup>, Felliipe Carneiro<sup>3</sup>, Ganesh Pokharel<sup>4</sup>, Sam Daly<sup>1</sup>, Zahir Islam<sup>5</sup>, and Stephen Wilson<sup>4</sup>

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A relatively new class of superconducting kagomes (alkali metal + V<sub>3</sub>Sb<sub>5</sub>) host a series of novel electronic ground states that are being currently explored. CsV<sub>3</sub>Sb<sub>5</sub> is one such material, whose superconducting state is preempted by a transition to charge density wave (CDW) ordering below 94 K. X-ray diffraction data for this kagome in particular shows evidence of CDW splitting, associated with 2 x 2 x 2 and 2 x 2 x 4 supercells respectively, but the relationship between these two orderings has yet to be fully explained. A picture of the material's microstructure and its contributions to the unique electronic structure is also incomplete, but previous work suggests that the CDW transition coincides with a transition from hexagonal to a twinned orthorhombic system. The work presented here details a spatially resolved investigation of charge density phases in CsV<sub>3</sub>Sb<sub>5</sub> using dark-field x-ray microscopy (DFXM). Through the use of an x-ray compound refractive lens (CRL), real-space diffraction contrast images were collected for structural and charge density wave Bragg peaks below the CDW transition temperature. A first of its kind study, the resulting DFXM mappings of the CDWs show a clear spatial separation between the two states, supporting working theories of phase coexistence. Estimates of the CDW domain sizes from this data correspond well to previous work, showing correlations spanning 100-250 microns or more. However, high-resolution DFXM images of the (2 2 0) structural peak, indicate that the microstructure of CsV<sub>3</sub>Sb<sub>5</sub> is characterized by much smaller sub-domains, averaging 0.5 to 1.5 microns in length. Coupled with observations of 120-degree domain boundaries, the structural images also give weight to the hypothesis of tri-variant twin formation in CsV<sub>3</sub>Sb<sub>5</sub> at low temperature. The results of this investigation help form a more complete understanding of quantum materials and low temperature phase transitions while also demonstrating a new modality in the application of DFXM to study charge order.

# Observation of Strain Wave in the Nematic Phase of the Iron Pnictide $\text{Ba}(\text{Fe}_{1-x}\text{Cu}_x)_2\text{As}_2$ Using Dark-field X-ray Microscopy

Kaan Alp Yay<sup>1,2</sup>, Elliot Kisiel<sup>4,5</sup>, Matthew J. Krogstad<sup>4</sup>, Doga Gursoy<sup>4</sup>, Stephan O. Hruszkewycz<sup>4</sup>, Zahir Islam<sup>4</sup>, and Ian R. Fisher<sup>1,3</sup>

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To gain a better understanding of the nematic phase in iron pnictides, we study underdoped  $\text{Ba}(\text{Fe}_{1-x}\text{Cu}_x)_2\text{As}_2$  (Cu-Ba122) using dark-field x-ray microscopy (DFXM). DFXM is a novel technique which enables one to image the real-space distribution of a selected diffraction peak emanating from a domain within the bulk of a sample. Cu-Ba122 undergoes a tetragonal-to-orthorhombic structural phase transition at low temperature due to electronic nematicity, at which point orthorhombic twin domains form along the *ab*-plane. By imaging a single domain, we observed (1) micron-scale periodic spatial modulations of diffraction intensity and strain within a domain, and (2) an increase in the amplitude and period of the spatial modulations as temperature is lowered.

These observations demonstrate the existence of a mesoscopic strain wave within the orthorhombic phase of iron pnictides that has so far been undetected in traditional x-ray diffraction due to its long wavelength. Our results also establish DFXM as a powerful new tool to probe the mesoscopic behavior of strongly correlated quantum materials.

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## Dark-field X-ray Microscopy for Quantum Materials and its Future at APS-U

Elliot Kisiel<sup>1,2</sup>, Ishwor Poudyal<sup>2,3</sup>, Pavel Salev<sup>4</sup>, Zahir Islam<sup>2</sup>, Stephan Hruszkewycz<sup>3</sup>, Zhan Zhang<sup>2</sup>, Ivan K. Schuller<sup>1</sup>, and Alex Frano<sup>1</sup>

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While full-field DFXM has demonstrated to be a potent imaging modality, it is the anticipated increase in coherent flux at APS-U that will enable unprecedented direct visualization of the intricate mesoscale network of competing phases in 3D through novel contrast mechanisms recently predicted with true bulk sensitivity. Our studies of VO<sub>2</sub> laid the groundwork for understanding phase separation in quantum materials which will be key in moving towards other systems that exhibit quantum phenomena. Establishing coherence-enhanced dark-field x-ray microscopy (cDFXM) at APS-U as a versatile, and in many cases superior, imaging tool for an incisive understanding of structural, electronic, and magnetic properties in real-time on a “mesoscale” with a spatial resolution reaching tens of nanometers which goes far beyond an “average” picture afforded by routine diffraction measurements. We explore these novel contrast mechanisms and their unique applications, for imaging boundaries between electronic and magnetic phases with a greater resolving power but also to distinguish those that separate phases differing in their mutual chirality, which is challenging, if not impossible, by other means that rely solely on a diffraction-intensity contrast. Simulation work suggests the use of cDFXM will not only enhance phase boundaries but can be instrumental in studying nucleations in mixed-phase systems, such as VO<sub>2</sub>, enhancing boundaries, and studying magnetic structures.



# Inhomogeneous Strain Development in Metal-to-insulator Transition Switching Devices: Microdiffraction and Dark-field X-ray Microscopy Perspectives

Pavel Salev<sup>1</sup>, Elliot Kisiel<sup>2,3</sup>, Dayne Sasaki<sup>4</sup>, Brandon Gunn<sup>2</sup>, Wei He<sup>2</sup>, Mingzhen Feng<sup>4</sup>, Junjie Li<sup>2</sup>, Nobumichi Tamura<sup>5</sup>, Ishwor Poudyal<sup>3</sup>, Zahir Islam<sup>3</sup>, Yayoi Takamura<sup>4</sup>, Alex Frano<sup>2</sup>, and Ivan K. Schuller<sup>2</sup>

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Electrically actuated phase-change materials are actively explored as promising candidates for improving the scalability and energy efficiency of conventional, neuromorphic, and reservoir computing electronics. While the significance of structure-function relationships is universally acknowledged, studies of electrically driven structural evolution in phase-change materials are highly challenging, because they require performing local, *operando* measurements. Here, we explore structural signatures of the electrically triggered metal-to-insulator transition (MIT) in (La,Sr)MnO<sub>3</sub> (LSMO) devices. MIT triggering results in the formation of a characteristic spatial pattern: an insulating phase barrier splitting a metallic phase matrix. Employing a combination of x-ray microdiffraction and dark-field x-ray microscopy, we show that electrically induced insulating barrier formation leads to the development of an inhomogeneous strain profile across the entire switching device, even though LSMO does not undergo a discontinuous structural transition coinciding with the MIT in equilibrium. Diffraction microscopy measurements revealed unique features of the electrical MIT triggering including lattice distortions, tilting, and twinning, which indicate structural nonuniformity of the metal and insulator regions inside the switching device. Such lattice deformations do not occur under equilibrium, zero-voltage conditions, highlighting the qualitative difference between the structural states achieved by increasing temperature and by applying voltage in nonlinear electrothermal materials. Electrically induced strain, lattice distortions, and twinning could have an important contribution in the MIT triggering process and drive the material into non-equilibrium states, providing an unconventional pathway to explore the phase space in strongly correlated electronic systems.

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# Wave-optical Simulation of a Dark-field X-ray Microscope

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The usual description of dark field x-ray microscopy (DFXM) [1] relies on geometric optics to describe both the state of the x-ray beam and its propagation through the image forming optics as well as its interaction with the sample crystallite. This treatment succeeds in describing the quantitative dependence of the microscope contrast to the sample strain state, which are the fundamental equations used to interpret DFXM in most settings. For its successes however, this treatment relies on approximations that are not always appropriate and fails to describe effects such as multiple scattering in the sample (dynamical diffraction) and coherent diffraction effects from the image forming optics.

We present a different theoretical treatment of the experiment as well as a set of simulations that rely on wave optics. In this setting dynamical diffraction can be handled within the framework of the Takagi-Taupin Equations (TTE) and coherent diffraction effects using Fourier optics. We try to use this model to illuminate the effect of (partial-) coherence and multiple scattering and to outline when these effects can, and cannot, be ignored [2].

Full forward simulation using the TTEs require a high-resolution model of the sample which is typically not available for anything but certain ideal defects such as point-defects, straight dislocations, and planar defects such as twin domain boundaries and stacking faults. We present comparisons of the simulation with one such isolated stacking fault found in an otherwise perfect region of a high temperature high pressure diamond that show a good quantitative match and reproduce artifacts with the same qualitative features [3].

[1] Poulsen, H. F., Jakobsen, A. C., Simons, H., Ahl, S. R., Cook, P. K. & Detlefs, C. (2017). *J. Appl. Cryst.* 50, 1441-1456.

[2] Borgi, S., Ræder, T. M., Carlsen, M. A., Detlefs, C., Winther, G., Poulsen, H. F, "Simulations of dislocation contrast in dark-field x-ray microscopy," *J. Appl. Cryst* (in press).

[3] Carlsen, M., Detlefs, C., Yildirim, C., Raeder, T. & Simons, H. (2022). *Acta Cryst.* A78, 482-490.

## From Imagination to Realization: A Tale of a Dark-field X-ray Microscope under a Coded-aperture Lens for 3D Imaging

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In recent years, considerable interest has emerged in utilizing coded apertures to customize beams, with the goal of enhancing spatial or temporal resolution, all while ensuring improved mechanical stability. This development is particularly timely as imaging undergoes a transformative phase with the introduction of new x-ray sources capable of higher resolution, although posing challenges to mechanical stability at such resolution levels. Initially directed towards expediting data acquisition in Laue diffraction imaging, this approach has now expanded to encompass dark-field imaging and the precise determination of depth from SAXS/WAXS signals, all without the need for complex sample scanning setups. In this presentation, I will narrate the journey of this progress, with a particular focus on dark-field imaging, while detailing both our successes and setbacks encountered along the way, offering projections for the future. These projections will primarily be informed by the ongoing global development of new x-ray sources with enhanced brightness and coherence, which hold the potential to significantly advance these efforts and unlock new scientific frontiers through this imaging approach.

# Development and Application of X-ray Spectromicroscopies Using Achromatic Wolter Mirror Optics

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X-ray spectromicroscopy provides unique insight into the internal structures of materials, combining high spatial resolution with critical chemical state information. Total-reflection mirror optics have ideal properties of achromaticity and high efficiency for x-ray spectromicroscopy, which requires wavelength scanning. In recent decades, advances in ultra-precise fabrication and measurement technologies have enabled the development of x-ray mirror optics that can achieve near diffraction-limited focusing and imaging.

We have developed several x-ray spectromicroscopies with total reflection Wolter mirrors fabricated by a precise electroforming process at the soft x-ray beamlines of SPring-8/SACLA [1-3]. Taking advantage of the achromatic optics, we have demonstrated them in spectro-ptychography and magnification imaging experiments, such as live or chemical state imaging of mammalian cells [4,5] and single-frame spectromicroscopy [6]. In this talk, we will present the development of these x-ray spectromicroscopy systems and their applications.

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[1] T. Kimura et al., *Opt. Express* 30(15), 26220–26228 (2022).

[2] Y. Takeo et al., *Appl. Phys. Lett.* 117(15), (2020).

[3] S. Egawa et al., *Opt. Express* 27(23), 33889–33897 (2019).

[4] Y. Takeo et al., *J. Electron Spectrosc. Relat. Phenom.* 267, 147380 (2023).

[5] K. Sakurai et al., submitted.

[6] Y. Takeo et al., *AIP Conf. Proc.* 2990(1), (2023).