Thin Films
Room 206 B W - Session TF2-MoM

Characterization of Thin Films

Moderators: Joseph Falson, Caltech, **John Hennessy**, Jet Propulsion Laboratory (NASA/JPL)

10:30am **TF2-MoM-10 Mapping Nanoscale Polarity Using Scanning Nanobeam Electron Diffraction Techniques*, Megan Holtz,*** Colorado School of Mines **INVITED**

Understanding local crystallography – including local lattice parameters, interatomic spacings, and polarity – is key to understanding ferroelectricity, especially in systems with small domains, significant disorder, or interfacial features. Scanning nanobeam electron diffraction (NBED) combined with new high-speed, pixelated scanning transmission electron microscopy (STEM) detectors make it possible to measure a diffraction pattern (kx, ky) at every scan position (x, y). This opens doors to investigate crystalline properties such as lattice parameters, local fields, polarization directions, and charge densities with relatively low beam dose over large fields of view. However, extracting these signals of interest from confounding signals such as thickness or crystallographic mistilt effects remains challenging. Here, we combine a cepstral approach, which is similar to a 2D pair-correlation function, with precession electron diffraction (PED) to measure local polar displacements in non-centrosymmetric materials while suppressing artifacts from dynamical or mistilt effects. We first apply these techniques to a reference sample of GaN and demonstrate that the addition of PED reduces the standard deviation of the lattice parameter measurement 0.56 pm to 0.32 pm. We also measure the length of the vector associated with the non-centrosymmetric nature of the unit cell, and see that its standard deviation is reduced from 3.5 pm to 1.9 pm. Computational work shows that adding precession also increases the robustness of these measurements to specimen mistilt. We then apply these techniques to study polar domains in a sample of PMN-PT, and observe domains on the 100 nm length scale.

11:00am **TF2-MoM-12 Data Science Tools to Disentangle Large Electron Diffraction Datasets of Thin Films*, Matthias Young,*** *Andreas Werbrouck, Andrew Meng, Dilan Gamachchige, Indeewari Herathlage, Nikhila Paranamana, Xiaoqing He,* University of Missouri

Historically, the thin film community has been largely driven by process-property understanding, and we have had limited access to deep understanding of the atomic-scale structure of vapor-deposited thin films. This arises from challenges in measuring the atomic structure of these films due to (1) their amorphous, polycrystalline, and defective structures, and (2) the ultrathin film thicknesses, often with gradients in composition and structure in the x-y and z directions. Prior work has established an understanding of how the structure of vapor-deposited thin films evolves during growth using bulk measurements such as infrared spectroscopy, synchrotron diffraction, and nuclear magnetic resonance, or surface sensitive measurements such as X-ray photoelectron spectroscopy. However, these approaches struggle to provide position- or depth-dependent atomic structure information, especially at sub-nanometer length scales. In recent years, our group has employed transmission electron microscopy (TEM) diffraction to measure the atomic structure of atomic layer deposition (ALD) films with high spatial resolution across nanoscale interfaces. However, even with TEM, the presence of multiple phases and orientations that are distributed in different amounts throughout the film volume and co-located in the beam path in each diffraction image make interpretation challenging. Here, we employ a data science algorithm known as non-negative matrix factorization (NMF) to identify the unique component diffraction signals and map their locations throughout ultrathin interfacial volumes. To facilitate this on the large volume of data present from a series of 2D diffraction patterns collected over a rastered 2D measurement area during scanning TEM (4D-STEM), we report the use of QR decomposition for randomized non-negative factorization as well as feature reduction through superpixel clustering. Together the speed-ups provided by these approaches allow for the rapid processing of high volumes of TEM data, enabling component isolation and spatial mapping for several gigabytes of electron diffraction data in minutes on a laptop compared to the hours required without these acceleration approaches. This allows us to quickly distill high volumes of data down to meaningful insights with low computational overhead, promising to enable more rapid discovery and innovation in the thin film community.

11:15am **TF2-MoM-13 A Novel *A*pproach to Study EUV and BEUV Photoresist Sensitivity through Real-time µXPS*, Peter Sun,*** *Samuel Tenney, Chang-Yong Nam, Jerzy Sadowski,* Brookhaven National Laboratory

Extreme ultraviolet (EUV) and beyond extreme ultraviolet (BEUV) lithography can achieve sub-10 nm features in semiconductor manufacturing. These nanoscale patterns require photoresists to be highly sensitive to EUV and BEUV conditions. One of the photoresist candidates are polymethyl methacrylate (PMMA) based hybrid photoresists with vapor phase infiltrated (VPI) inorganic materials.

The sensitivity of the photoresists depends on photo absorption efficiency, secondary electron generation, and material degradation. Currently, sensitivity studies mainly focus on characterizing developed films, an approach that cannot decouple photoresist sensitivity and developer sensitivity. To isolate the photoresist sensitivity, a method to study these photoresists' in situ exposure behavior before development is needed.

This report presents a novel approach to studying photoresist sensitivity through in situ real-time low-energy electron/photoemission electron microscopy (LEEM/PEEM) and micro-spot X-ray photoelectron spectroscopy (µXPS). In particular, we model the time-dependent chemical change and the charging behavior of the photoresists under X-ray exposures at 92 eV and 400 eV. We show the approach is reliable in determining the PMMA and its VPI hybrids' sensitivity to EUV and BEUV conditions. This approach will allow the study of a broader range of EUV and BEUV photoresist candidates and assist in next-generation photoresist and developer selection.

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11:30am **TF2-MoM-14 Stoichiometric Determination in Thin Films: A Study of $BaTiO_3$*, Peter Dickens,*** *Melissa Meyerson, Mark Rodriguez, Clare Davis-Wheeler, Jonathan Heile, William Wampler, Christian Harris, Brianna Klein,* Sandia National Laboratories

Thin films are essential in modern technology, providing unique properties for electronic, sensor, and optical applications. As more complex alloys and compounds are integrated onto devices, the need to effectively characterize material composition becomes increasingly important. To surmount this hurdle, many different methods are utilized throughout literature with XRF, XPS, and EDX being prime among the reported methods; however, it is common for there to be little to no discussion on the analysis, nuances, and accuracy of the technique used. These issues are further exacerbated by the common availability of black box analysis associated with each of these techniques leading to reports and conclusions based on imprecise analysis.

In this presentation we use BaTiO3 as a case study material to compare each of the common compositional methods. We report on the determination of the Ba/Ti atomic ratio in four thin films deposited by sputter deposition under conditions to produce a range of stoichiometries. We directly compare analysis produced from X-ray Fluorescence (XRF), Rutherford Backscatter Spectroscopy (RBS), Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), X-ray Photoelectron Spectroscopy (XPS), and Wavelength Dispersive Spectroscopy (WDS). Discussion is focused on accuracy of each technique and nuances related to each method and the analysis.

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