Thin Films
Room 206 B W - Session TF2-MoM

Characterization of Thin Films

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

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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11:45am **TF2-MoM-15 Advanced Characterization of Inorganic and Organic Liners for Through Vias in Glass Packages*, Meghna Narayanan,*** *Mohan Kathaperumal, Mark Losego,* Georgia Institute of Technology, USA

Electronic packaging technology is evolving towards advanced substrates, including glass, to overcome limitations posed by traditional organic substrates. Glass packaging offers low dielectric loss, high modulus, low and tunable coefficient of thermal expansion (CTE), enhanced thermal stability, high I/O density, and reduced warpage. However, fully adopting glass packaging technology still faces several hurdles, including metal adhesion, stress management, and a full understanding of long-term reliability. The CTE mismatch between glass and copper (5 to 15 ppm/°C) leads to reliability issues such as glass and through-glass via (TGV) cracking, copper via protrusion, and delamination. We aim to address the glass and TGV cracking by introducing an organic or inorganic liner inside the TGV to act as a stress buffer, lowering the propensity for TGV electrode failure. In this study, we will describe TGV substrates coated with different liners – Parylene C, SiO_2N, SiO_3N, AZO (Aluminium-Zinc oxide), and Parylene C+AZO, obtained through different methods of deposition. The foremost step is to characterize if the liner is conformally coating the 100 µm diameter via. Conformal coating is essential to mechanical performance and even electroplating through the entire depth of the TGVs. A non-uniform liner coating will lead to an uneven Ti-Cu seed layer prior to electroplating of copper. Since the liner can be 0.1 to 5 µm thick, cross-sectioning is inadequate for post-plating liner inspection. Non-destructive methods for evaluating these coatings would also be of value to research and development, as well as in-line process monitoring. We have evaluated the effectiveness of several methods. In this work, two advanced techniques—two-photon imaging and micro-computed tomography (microCT)—are used to assess liner uniformity and measure thickness. Two-photon imaging is particularly effective for fluorescent materials like Parylene C and SiO2N, enabling visualization and thickness measurement within TGVs. The thickness of Parylene C is measured to be 5.6 µm (expanding to 8.7 µm near the surface), and Parylene C+AZO is measured to be 1.87 µm (expanding to 2.8 µm near the surface). Although AZO and SiO_2N are fluorescent, the nanoscale thickness (< 100 nm) is challenging to measure due to the resolution of the tools. The paper will propose additional metrology tools and show preliminary attempts to measure such nanoscale

thicknesses. We will also show preliminary results on plating the liner-coated TGVs to assess their performance in mitigating crack formation upon thermal cycling.

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