New Trends on Structural and Electronic Characterization of Materials, Interfaces, and Surfaces Using Synchrotron and FEL-Based Radiation Sources Focus Topic
Room Ballroom A - Session LS-TuP

New Trends on Structural and Electronic Characterization of Materials, Interfaces, and Surfaces Using Synchrotron and FEL-Based Radiation Sources Poster Session

LS-TuP-2 Synchrotron Hard X-Ray Scattering for Investigation of ALD Processes, ***Jeffrey Woodward***, U.S. Naval Research Laboratory; *P. Myint, B. Jiang,* Boston University; *X. Zhang,* University of Vermont; *C. Wang, K. Ludwig,* Boston University; *R. Headrick,* University of Vermont; *S. Rosenberg,* U.S. Naval Research Laboratory; *K. Evans-Lutterodt, L. Wiegart, A. Fluerasu, R. Li, M. Fukuto,* Brookhaven National Laboratory; *C. Eddy,* U.S. Naval Research Laboratory

In recent years, synchrotron hard x-ray scattering has been increasingly utilized for *in situ* studies of atomic layer deposition (ALD) processes [1-3]. In addition to being non-destructive, sensitive to nanometer or sub-nanometer scale changes in topography or crystal structure, and providing statistical information averaged over large regions of the sample, hard x-rays are capable of penetrating the harsh process environments which preclude the use of techniques such as reflection high energy electron diffraction that are commonly used to monitor thin film growth by other methods. These advantages, combined with the high brilliance of synchrotron radiation available at modern light sources, enable the investigation of ALD growth kinetics and material properties as they evolve in real-time. This is particular useful for understanding plasma-enhanced ALD (PEALD) processes, as the plasma plays a complex role in providing both energy and reactive species through various reaction pathways, which can produce significant differences in growth behavior depending on the plasma properties.

We present an overview of various applications of synchrotron hard x-ray scattering which enable new fundamental insights into ALD processes. For each case, we highlight our studies of III-nitride semiconductor growth by PEALD conducted at National Synchrotron Lightsource II. We first discuss grazing incidence small-angle x-ray scattering (GISAXS), a highly surface sensitive technique for probing in-plane topography which is especially useful for monitoring the real-time evolution of surface islands. Next, we discuss x-ray photon correlation spectroscopy (XPCS) in a GISAXS geometry to investigate time-correlated local fluctuations about the average kinetics, or dynamics, which is made possible by utilizing a highly coherent x-ray beam. We then discuss scattering for greater incident angles, where intensity oscillations at the specular spot provide real-time information on layer thickness and surface roughness. Finally, we discuss *ex situ* grazing incidence wide-angle x-ray scattering (GIWAXS), which enables the rapid mapping of large regions of reciprocal space corresponding to interatomic distances, and is thus an extremely powerful characterization method for the myriad polycrystalline, phase change, and mixed-phase materials of interest to the ALD community.

[1] J.M. Woodward *et al.*, J. Vac. Sci. Technol. A **37**, 030901 (2019)
[2] N. Nepal *et al.*, J. Vac. Sci. Technol. A **37**, 020910 (2019)
[3] J. Dendooven *et al.*, Nat. Commun. **8**, 1074 (2017)

LS-TuP-3 High Energy X-Ray Photoelectron Spectroscopy of COTS Electronics Interfacial Failure Modes, ***Samantha G. Rosenberg***, *M. Meyerson, M. Kottwitz,* Sandia National Laboratories; *R. Rajendran,* Georgia Institute of Technology; *M. Reingold, B. Young,* Sandia National Laboratories; *P. Singh, J. Kacher,* Georgia Institute of Technology; *J. Fowler,* Sandia National Laboratories

The increasing complexity and miniaturization of electronics have driven demands to lean heavily on commercial off the shelf (COTS) electronics parts. COTS part defects are typically stochastic, making root cause identification challenging when failures occur. This study aims to identify a novel characterization scheme which can uncover fundamental relationships between interfacial material defects and chemical and physical phenomena which drive ultimate failure of COTS parts in relevant field environments. We first apply accelerated stress testing in corrosive environments using salt-fog chambers and environmental chambers to identify marginal or defective parts and then characterize these parts to identify corresponding (electro)chemical phenomena. Characterization includes studying the local electric field around the parts with scanning vibrational electrode technique (SVET) and examining latent and emerging defects in the COTS parts using in-situ liquid cell transmission electron microscopy (in-situ TEM) to correlate and predict interfacial material properties with electrochemical phenomena in relevant field environments. HAXPES will then allow for identification of subsurface chemistry related to metal migration, metal precipitate, and corrosion product formation due to exposure in various accelerated aging experiments performed before beamtime exposure using the aforementioned techniques.

Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

LS-TuP-4 Insitu AFM Imaging of the Structural and Morphological Evolution of Epitaxial $LiCoO_2$ Films during Charge and Overcharge, ***Yingge Du***, Pacific Northwest National Laboratory; *W. Samarakoon,* Oregon State University; *J. Hu, L. Wang,* Pacific Northwest National Laboratory; *Z. Feng,* Oregon State University; *J. Tao,* Pacific Northwest National Laboratory

Capacity decay of layered cathodes in high voltage applications underscores the need to utilize accurate and precise techniques to understand the underlying mechanisms. Here, we use well-defined epitaxial $LiCoO_2$ (LCO) films on $SrRuO_3/SrTiO_3$ (SRO/STO) with controlled orientations and defect structures along with *in situ* electrochemical atomic force microscopy to probe the structural and morphological evolutions during the charge and overcharge processes. We quantitatively show the morphological changes in both reversible delithiation regime and irreversible over-delithiation regime, and correlate the overall electrochemical behaviors to atomic scale defect evolutions in the films. We also observe a significantly lower charging capacity for LCO/SRO/STO(111) compared to that of LCO/SRO/STO(001) films of the same thickness, which is ascribed to the different types of atomic scale defects formed during the film growth process.

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