Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Pacific D - Session H1-1-MoM

Spatially-resolved and In-Situ Characterization of Thin Films and Engineered Surfaces I

Moderators: Grégory Abadias, Institut Pprime - CNRS - ENSMA - Université de Poitiers, France, **Xavier Maeder**, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland, **Michael Tkadletz**, Montanuniversität Leoben, Austria

10:00am **H1-1-MoM-1 In Situ Observations and Measurements of Plastic Deformation, Phase Transformations and Fracture With 4D-STEM*,* *Andrew Minor (aminor@berkeley.edu)*, UC Berkeley and LBNL, USA **INVITED**

In situ TEM experiments are typically recorded either in real space or diffraction space. However, it would be ideal to have information from both for when transient events occur that cannot be repeated exactly (ie-defect generation or irreversible phase transformations). 4D-STEM can come close to providing simultaneous real-space imaging and diffraction analysis during *in situ* testing, making it possible to perform orientation and strain mapping and defect and phase identification via diffraction pattern analysis during in-situ deformation in a TEM. This talk will highlight recent *in situ* 4DSTEM nanomechanical deformation experiments that explore transient events where both information from diffraction space and real space are used. The diffraction patterns are used to identify different phases, defects, orientations and relative strain, while the images formed by using virtual apertures provide microstructural context for the analysis. Example experiments include defect generation and fracture in multi-principal element alloys and in situ heating and cooling of materials going through phase transformations.

10:40am **H1-1-MoM-3 Real-Time N_2-Mediated Growth Manipulation of Ultrathin Ag Layers*,* *Gregory Abadias (gregory.abadias@univ-poitiers.fr)*,** Institut PPrime - CNRS - ENSMA - Université de Poitiers, France*; A. Jamnig, D. Babonneau, A. Michel, Y. Robin,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; A. Resta, A. Vlad, A. Coati,* Synchrotron SOLEIL, France*; K. Sarakinos,* University of Helsinki, Finland*; B. Krause,* Karlsruhe Institute of Technology (KIT), Germany

Noble-metal ultrathin films, with nominal thickness smaller than ~15 nm, are ubiquitous in a wide range of plasmonic devices and other optoelectronic applications. Silver (Ag) layers have recently gained interest as alternative transparent conductive electrode (TCE) candidates for flexible photovoltaics to currently used indium tin oxide, which is inherently brittle and suffers from high cost and poor sustainability. However, Ag films obtained by conventional physical vapor deposition have the natural tendency to self-assemble into 3D agglomerates on weakly interacting substrates, resulting in the formation of rough surface profiles. Therefore, strategies to produce fully continuous, ultrathin and ultrasmooth Ag layers without compromising their electrical conductivity are needed. Among them, the use of gaseous additives, such as N_2 or O_2, appears as an efficient route to shift the continuous film formation thickness to lower values [1,2]. However, to understand the full evolutionary growth regime requires the implementation of *in situ* and real-time diagnostics.

In the present work, the impact of N_2 addition on the morphological and structural evolutions of ultrathin Ag layers is investigated by coupling complementary *in situ* and real-time diagnostics. Lab-scale studies include wafer curvature, surface differential reflectance spectroscopy and electrical resistivity to determine the morphological transition thicknesses (percolation and continuous formation thickness) [3] as a function of N_2 partial pressure. These are augmented by real-time X-ray synchrotron studies (SIXS beamline at SOLEIL) in which the diffraction and reflectivity signals are simultaneously recorded, together with stress evolution. This enable us to explore the influence of N_2 on island shape, texture and stress development, as well as relaxation mechanisms during growth interruptions.

1. Yun, J. *et al.* An unexpected surfactant role of immiscible nitrogen in the structural development of silver nanoparticles: An experimental and numerical investigation. *Nanoscale***12,** 1749–1758 (2020).

2. Jamnig, A. *et al.* 3D-to-2D Morphology Manipulation of Sputter-Deposited Nanoscale Silver Films on Weakly Interacting Substrates via Selective Nitrogen Deployment for Multifunctional Metal Contacts. *ACS Appl. Nano Mater.***3,** 4728–4738 (2020)

3. Colin, J. *et al.* In situ and real-time nanoscale monitoring of ultra-thin metal film growth using optical and electrical diagnostic tools. *Nanomaterials***10,** 2225 (2020)

11:00am **H1-1-MoM-4 Phase Transformation and Solid-State Dewetting of Precious Metal High Entropy Alloy Thin Films on a Sapphire Substrate*,* *Xavier Maeder (xavier.maeder@empa.ch)*,** *A. Sharma, P. Schweizer, J. Michler,* Empa - Swiss Federal Laboratories for Materials Science and Technology, Switzerland

The transition of thin films into isolated particles at a temperature below the melting point of the bulk material is known as solid-state dewetting. So far, most of the literature is limited to dewetting studies on pure metal and binary alloys thin films. The experimental data on dewetting of compositionally complex alloys is rather missing.This work studied the solid-state dewetting behavior of precious metal high entropy alloy thin film deposited on a (0001) single-crystalline sapphire substrate and annealed in a range of temperatures. The combination of dedicated imaging, composition mapping, and diffraction techniques is used to investigate the interplay of grain growth, phase transformations, and dewetting kinetics with the process of in-situ annealing in the TEM.Both X-ray diffraction and the transmission electron microscopy observations revealed the FCC (single-phase) →FCC1+FCC2 (double-phase)→FCC3 (single-phase) phase transformation sequence during annealing. In addition, ex-situ annealing experiments have been performed in the same temperature range to assess the film's dewetting and phase transformation kinetics quantitatively.

11:20am **H1-1-MoM-5 Investigation of Silicon Samples by the Emerging Picosecond Ultrasonics***, F. Faese,* ***Julien Michelon (jmichelon@neta-tech.com)****, X. Tridon,* Neta, France

With the constantly increasing needs in microelectronics, photovoltaic cells, and other high-tech components in specialized industries, characterizing the growth of epitaxial (epi) silicon on silicon or evaluating the quality of Si/Si bonding becomes more and more critical. For instance, managing the epi Si layer paved the way to new generations of devices such as the Metal Oxide Semiconductor devices (MOS, CMOS and MOSFET) [1] as well as high performance photovoltaic cells [2]. Therefore, in order to address these increasing needs, existing characterization tools provide constantly improved performances, and new characterization tools regularly emerge to provide a technological breakthrough that offers more challenging features.

Some of the existing characterization tools that meet the needs are destructive, such as Scanning and Transmission Electron Microscopy, or semi-destructive such as Secondary Ion Mass Spectrometry. Only a few characterization tools can measure the thickness of epi Si on Si and evaluate the quality of Si/Si bonding after the fabrication process and non-destructively. Among these techniques is the emerging Picosecond Ultrasonics (PU) [3]. This communication will present the principle of operation of this technique and describe some of the potentialities regarding the characterization of Si-based samples. First, we will see how PU can measure the thickness of a layer of epi Si on a Si substrate and describe the related advantages of this technique. Second, we will focus on Si/Si bonding applications with an illustration of PU possibilities to evaluate the quality of the silicon direct bonding.

[1] Skibitzki, Oliver. "Material Science for high performance SiGe HBTs: Solid-Phase Epitaxy and III-V/SiGe hybrid approaches." (2013).

[2] Hamon, Gwenaëlle, et al. "Plasma-enhanced chemical vapor deposition epitaxy of Si on GaAs for tunnel junction applications in tandem solar cells." Journal of Photonics for Energy 7.2 (2017): 022504.

[3] Thomsen, C., et al. "Coherent phonon generation and detection by picosecond light pulses." Physical review letters 53.10 (1984): 989.

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