Friday Morning, November 10, 2023

Advanced Focused Ion Beams Focus Topic
Room B110-112 - Session IB2-FrM

Advances in TEM and APT Specimen Preparation

Moderators: Tanvi Ajantiwalay, Pacific Northwest National Laboratory, **Gregor Hlawacek**, Helmholtz-Zentrum Dresden - Rossendorf

9:00am **IB2-FrM-3 Correlative and In Situ TEM/APT Technique Reveals Insights into Early Oxide Film Formation in a High Entropy Alloy*, Bharat Gwalani,*** Engineering Bldg I 911 Partners Way **INVITED**

Oxide film formation is a critical process that occurs on the surface of materials when they come into contact with oxygen. In some cases, oxide films can help protect the material from further corrosion, but in other cases, they can lead to degradation of the material over time. Understanding the early stages of oxide film formation is therefore important for designing materials that are resistant to corrosion. This study employed a combination of correlative and in situ techniques, specifically transmission electron microscopy (TEM) and atom probe tomography (APT), to investigate the initial stages of oxide film formation in a high entropy alloy (HEA). The research aimed to gain insight into the mechanisms of oxide film formation and the chemical changes that occur during the process. By analyzing the nano-scale chemical changes of the oxide film and the underlying HEA as a function of time, we observed the phase transformation pathway resulting from a single-layer to a multi-layer oxide film, which provided important insights into the process of oxide film formation. The study demonstrates the potential of these techniques in providing a deeper understanding of the processes involved in oxide film formation, enabling the design of materials with improved corrosion resistance and durability.

9:40am **IB2-FrM-5 Applications of Advanced Focused Ion Beam System to Energy Storage Materials*, Yaobin Xu,*** *X. Cao, W. Xu, J. Zhang, C. Wang,* Pacific Northwest National Laboratory

Battery technology has received considerable attention in recent decades due to the rapid growth of the electric vehicle and consumer electronics markets. An in-depth understanding of the electrode, interfacial structure, and chemical distribution of battery materials is essential to further advance battery technology. Focused ion beam-scanning electron microscope (FIB-SEM) is an analytical method combines ion beam for materials processing and electron beam for imaging. It enables both two-dimensional (2D) and three-dimensional (3D) imaging capability, combined with electron backscatter diffraction (EBSD) and energy dispersive X-ray spectrometry (EDS) detector, could also provide multimodal information collection, which is an effective and powerful analytical approach for battery structural analysis.

Lately, the plasma FIB-SEM (PFIB-SEM) technology has been developed with different ion source and higher ablation efficiency. It promises great potential for battery materials characterization, due to accessing representative 2D area and 3D volume via much faster (compared with traditional Ga^+ system) milling rate as well as enabling Ga^+ free sample preparation on advanced battery system through non-reactive ion source (Xe^+ and Ar^+ ion). Besides, for air and beam sensitive battery materials, like alkali metal electrode, sulfide based solid state electrolyte, combing cryogenic stage and inert gas transfer system, we could image and process them without air contamination during sample transfer and reduce ion/beam damage under cryogenic condition. In this presentation, cryogenic PFIB-SEM has been used to perform 2D microstructure and chemical analysis and 3D imaging/chemical analysis on different battery systems including Li metal anode, lithium nickel manganese cobalt oxide (NMC) cathode and solid-state electrolyte.

The successful demonstration of the cryogenic PFIB-SEM provides exciting possibilities for the investigation of both current and future generations of advanced battery technologies, and will be an important analytical approach for battery research and development.

10:00am **IB2-FrM-6 The Fabrication of Ruthenium single crystal specimen with Focused Ion Beam and Field Ion Microscopy for Atom Probe Tomography*, Mark G Wirth,*** *D. Perea, S. Lambeets,* Pacific Northwest National Laboratory

Metallic surfaces may undergo a series of surface and subsurface structural and chemical transformations while exposed to reactive gases that inevitably change the surface properties. Understanding such surface dynamics from a fundamental science point of view is an important requirement to build rational links between chemical/structural surface properties and design new catalysts with desired performance or new materials with enhanced resistance to corrosion. This is the case for Ruthenium (Ru), a relatively scarce precious material found in various large-scale applications such as chlorine, nitrates, and acetic acid production. Additionally, Ru shows promising properties for low temperature Haber-Bosch process to produce ammonium.

The imaging techniques of Field Ion Microscopy (FIM) and Atom Probe Tomography (APT), when combined, provide unique atomically resolved surface structure and compositional maps used to understand metal oxidation dynamics. FIM enables correlative atomic to nanoscale imaging of the surface of a very sharp metal needle revealing a complex network of crystallographic facets, the nanoscale apex size and hemispherical like shape of which models that of a metal nanoparticle. While APT and Operando Atom Probe (OAP) is used to track the chemical interaction between the complex surface with reactive gases such as O_2 or N_2 through composition mapping.

Compared to other metals, Ru specimen preparation is relatively challenging due to fragility related to its hexagonal close packed (HCP) crystal structure. This makes Ru particularly sensitive to mechanical stresses during FIM and APT experiments. Additionally, Ga implantation from a FIB-based needle specimen preparation approach can lead to the formation surface defects, complicating the indexing of the atomically resolved surface structure. In this work we will focus on the Ru specimen fabrication by FIB and the surface imaging by FIM with the indexing of Miller indices mapped across various surface features.

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