

## Chemical Analysis and Imaging at Interfaces Room Ballroom A - Session CA-ThP

### Chemical Analysis and Imaging at Interfaces Poster Session

#### CA-ThP-1 Synthesis and Characterization of Bayerite sub-Micrometer Particles, *Mackenzie Savage, Xiao-Ying Yu*, Oak Ridge National Laboratory

The Hanford Site in Washington state houses the largest collection of nuclear waste in the United States, including 56 million gallons of mixed waste that must be processed for vitrification by the Department of Energy (DOE). However, the waste contains many insoluble micro- and nanoparticles that have unpredictable rheology due to their complex chemistry and are difficult to model. Aluminum hydroxides and oxyhydroxides such as bayerite make up a significant portion of these particles, and they must be filtered out before vitrification, as excess amounts can degrade the quality of the glass. Supply chain issues and a lack of information on their aqueous behavior has led to this study of aluminum (oxy)hydroxide nanoparticles. This research synthesizes bayerite micro- and nanoparticles using a hydrothermal synthesis process, namely by titrating and heating an aluminum hydroxide gel precursor solution. The resultant crystal structure of the largest particles was confirmed via powder x-ray diffraction, and elemental analysis confirmed that the particles were composed of bayerite. Scanning electron microscopy showed that particle sizes ranged from 70 nm needles to 4  $\mu\text{m}$  particles in a bimodal distribution depending on the crystallization interval, with particles plateauing in size after about 48 h. Future work is needed to refine synthesis methods to reduce impurities and to obtain images of smaller particles at higher resolutions. Raman and Fourier Transform Infrared (FTIR) spectroscopy are also expected to confirm crystal structure of smaller particles. In situ characterization of particles in solution is necessary to observe the aqueous behavior of bayerite nanoparticles and how their rheology at varying pH might affect processing of Hanford waste.

#### CA-ThP-2 Enhanced Nanoprojectile Secondary Ion Mass Spectrometry (NP-SIMS): Correlation Among Spatially Resolved Secondary Ions, *Pierre Hirschhahn, Stanislav Verkhoturov*, Texas A&M University; *Michael J. Eller*, University of Mississippi; *Emile A. Schweikert*, Texas A&M University

Nanoprojectile secondary ion mass spectrometry (NP-SIMS) consists in bombarding an analyte with a heavy projectile in an event-by-event fashion. Each impact is resolved in space and time. Thus, the secondary ions (SIs) from each impact can be mass analyzed and recorded as individual mass spectra. The crater size is in the range of 10-15 nm wide and depth. These dimensions enable molecular analysis with unparalleled lateral resolution. Yet more information can still be extracted from the SI, by considering their anisotropic distribution. The ejecta have distinct spatial distributions set by their axial and radial energies. These parameters in turn reflect the molecular environment, the formation-ejection mode and metastable decay. They can be accessed with the temporally and spatially correlated detection of SIs. We demonstrate spatially resolved SI analysis on a homogeneous surface of Tris(8-hydroxyquinolinato)aluminum ( $\text{Alq}_3$ ) sublimated on a silicon substrate. The NP-SIMS measurement was performed on a custom-made system equipped with a fullerene effusion source accelerated at 50 keV coupled to a linear time-of-flight mass analyzer with a 64 anodes detector. We identify analyte-specific moieties, SIs synthesized by impact chemistry by their distinct spatial distributions. The latter can be crucial for enhanced accurate identification of SIs in surfaces of unknown composition.

#### CA-ThP-3 Studying Neutron Irradiation Effects on Lithium Aluminate Pellets Using ToF-Sims, *Xiao-Ying Yu, Jiyoung Son*, Oak Ridge National Laboratory; *Tanguy Terlier*, Rice University; *Gabriel Parker*, Oak Ridge National Laboratory; *David Senor*, Pacific Northwest National Laboratory

We studied tritium breeding lithium aluminate pellets in the Tritium-Producing Burnable Absorber Rod. The selected irradiated pellets have three different thickness, namely standard, thin, and thick wall thickness, which relates to their tritium producing and retention capacities. Irradiated materials were reduced to small sizes using scanning electron microscopy (SEM) – focused ion beam (FIB). Light isotopes, such as deuterium, tritium, and lithium, are detected using highly sensitive time-of-flight secondary ion mass spectrometry (ToF-SIMS). We show that the standard and thin thickness pellets are better in retaining tritium than the thick pellet in tritium production. The thinner wall pellet retains more tritium than the standard or thick ones. Isotope exchange reactions take place and tritiated

hydrocarbons exist in all pellets. As the first study of irradiated pellets using ToF-SIMS, this work provides a new way to determine isotopic abundance of irradiated materials, improving understanding of tritium breeding in fission and fusion reactors.

#### CA-ThP-4 In-Plasma XPS: A New Operando Metrology For Process Development and Control, *Andrei Kolmakov*, NIST

Modern near-ambient-pressure X-ray photoelectron spectroscopy (NAP or AP-XPS) instruments now cover the pressure range typical of standard plasma applications, expanding the capabilities of XPS into plasma environments. We recently demonstrated that XPS spectra can be successfully collected in these conditions, extending the application of XPS to plasma-surface interactions [1]. We highlighted the influence of plasma chamber wall reactions on sample surface chemistry and showed that plasma-XPS can capture plasma chemistry in the gas phase [2]. Plasma-induced charging and damage of wafers is a well-known challenge in semiconductor fabrication [3]. We apply plasma-XPS to model poorly conducting samples and observe anomalous XPS binding energy shifts due to sample charging during plasma exposure. We propose mechanisms that explain these shifts. Additionally, we observed plasma-induced binding-energy shifts and peak splitting in the XPS spectra of the plasma gas phase, which were related to the local plasma potential and its time alterations [4]. Overall, plasma-XPS metrology offers an opportunity to record real-time surface chemistry on plasma-exposed surfaces, along with wafer charge and plasma diagnostics, and has significant potential to advance semiconductor process development and control, as well as defect-mitigation strategies.

#### References

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#### CA-ThP-5 A High-Throughput Multimodal Workflow for Characterizing Soil Mineral-Organic Interfaces with XPS, FTIR, SEM/EDS and ToF-SIMS, *Vaithiyalingam Shutthanandan, Ajay Karakoti*, Pacific Northwest National Laboratory; *Catherine Pettinger*, University of Wisconsin-Madison; *Kavin Thangaraj*, Washington State University; *Odeta Qafoku, Mark Engelhard, Bhuvana Modachur Sivakumar, Zihua Zhu, Ravi Kukkadapu*, Pacific Northwest National Laboratory; *Erica Majumder*, University of Wisconsin - Madison

Understanding how soil carbon is stabilized as mineral-associated organic matter (MAOM) at the molecular scale is critical for more accurately representing soil processes in Earth system models. In particular, quantifying the chemistry and spatial organization of organic matter (OM) at mineral interfaces including ion-binding mechanisms, the distribution and density of reactive sites, and the arrangement of functional groups which provides key insight into the persistence, transformation, and loss of soil carbon. To address these questions, we developed a high-throughput, multimodal imaging and spectroscopy workflow that characterizes mineral-organic interfaces in terms of their elemental composition, molecular structure, and micro- to nanoscale topology. This approach integrates X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS), yielding complementary information on bonding environments, functional group chemistry, surface morphology, and mineral composition. We implemented a custom high-throughput sample preparation method based on a dot-blot style deposition system, enabling rapid and reproducible preparation of many small soil aliquots or mineral-OM extracts on a single substrate. This configuration allows all four analytical techniques to probe the same set of spots, ensuring direct comparability of chemical and structural measurements across methods. With this platform, we can analyze approximately 50 samples per day, supporting systematic, large-scale characterization of MAOM across environmental gradients. In this study, samples are drawn from the MONet project, providing a broad and spatially diverse soil inventory for

establishing quantitative relationships between mineral surface properties, organic matter chemistry, and carbon stabilization.

**CA-ThP-6 Influence of Surface Preparation on Au/P-Type HgCdTe Interfaces Investigated by Xps Depth Profiling, Alexandra Colas-Reuillon, Clément Lobre, Eugénie Martínez, Steven Bel, Marc Veillerot, Sarah Petit, Olivier Gravrand, CEA-Leti, France**

P-type metal contacts on semiconductors remain a major technological challenge, particularly in Mercury Cadmium Telluride ( $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ , MCT), a reference material for cooled infrared detectors [1]. In the context of the SWaP-C (Size, Weight, Power, and Cost) approach, current developments aim to increase the operating temperature of infrared devices. However, higher temperatures are associated with anomalous pixel responses and increased noise levels, partly attributed to non-ideal Schottky-type behavior at the metal/p-type MCT interface.

Among the different strategies proposed to improve contact quality, surface preparation prior to metallization plays a critical role through the reduction of interface defects, passivation of dangling bonds, and control of interfacial oxides. Despite previous XPS studies, the mechanisms governing contact formation remain insufficiently understood. In this work, we investigate the influence of surface preparation on metal/MCT interfacial reactivity using X-ray Photoelectron Spectroscopy (XPS) combined with gentle  $\text{Ar}^+$  sputter depth profiling.

Gold thin-film contacts ( $\sim 10$  nm) were deposited on arsenic-doped p-type  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  ( $x = 0.29$ ) with a carrier concentration of  $\sim 10^{18} \text{ cm}^{-3}$ . Two surface preparation routes were compared prior to metallization: (i) a deoxidized surface obtained by dry plasma treatment followed by wet chemical etching, and (ii) a deliberately oxidized surface prepared by plasma treatment only. Depth profiles were acquired by XPS (Al K $\alpha$  source, Quantes, ULVAC-PH) combined with low-energy  $\text{Ar}^+$  sputtering. Quantification was corrected using sputter sensitivity factors determined from a non-metallized reference MCT sample.

The measurements revealed strong preferential sputtering effects following the sequence  $\text{Hg} \gg \text{Te} > \text{Cd}$ , attributed to the fragility of Hg-Te bonds. After correction, the Au/MCT interface formed on the intentionally oxidized surface appeared significantly sharper than that obtained on the deoxidized surface. Surface deoxidation promotes Au diffusion into the MCT as well as reciprocal interdiffusion processes. In contrast, the plasma-grown oxide layer, richer in HgO and CdTeO species than the native oxide formed after air exposure, likely stabilizes the surface and acts as a barrier against Au interdiffusion during deposition.

Such diffusion phenomena may strongly affect the electrical quality of the contacts through the formation of interfacial defects, local stoichiometry changes, and Au-induced doping effects.

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**CA-ThP-7 Multi-modal analysis of NIST standard Iron Ore for establishment of the LIOS database, Gabriel Parker, Mackenzie Savage, Oak Ridge National Laboratory, USA; Michael Mulholland, Idaho National Laboratory; Xiao-Ying Yu, Oak Ridge National Laboratory, USA**

**Abstract:** The US Department of Energy (DOE) recently announced \$155 million to advance American industrial innovation toward enhancing the capabilities of US national laboratories to develop technologies which will improve technological innovation, reduce costs, and increase prosperity for American workers and consumers. The DOE Office of Critical Minerals and Energy Innovation (CMEI) invested in new programs to address critical industrial challenges in areas such as energy intensive industry, cross sector technologies, data center collaboratives, and national library for iron ore and scrap. This library for iron ore and scrap (LIOS) project focuses on the iron ore and steel scrap to develop a national resource center and to deliver advanced characterization of iron ore and steel scrap samples of shared or donated by users for boosting American industrial innovations. The ORNL team's efforts are to develop analytical methods using X-ray diffractometry (XRD), inductive coupled plasma mass spectrometry (ICP-MS), and scanning electron microscopy (SEM) with energy dispersive x-ray spectroscopy (EDS) and electron backscatter detection (EBSD) to provide detailed characterization of the natural and processed/recycled feedstocks. In the first step, several iron ore reference materials available from National Institute of Standards (NIST) will be used to establish the analytical benchmarking for XRD, ICP-MS, and SEM/EBSD. Additionally, time-of-flight secondary ion mass spectrometry and atom probe tomography will be used

as complementary techniques to verify composition and microstructures at the nanoscale. Our results provide the necessary baseline measurement to build the foundation for a resource library, which will provide important technical information for industrial needs.

**Keywords:** iron ore, SEM/EBSD, ICP-MS, XRD, ToF-SIMS, chemical imaging

**CA-ThP-8 Exploring Radiolytic Chemistry of Tin Oxide Photoresists for Extreme Ultra-Violet (EUV) Photolithography Using Plasma-XPS, Trey Diulus, NIST-Gaithersburg; Priyanka Ketkar, Conan Weiland, Cherno Jaye, NIST; J. Anibal Boscoboinik, Ashley Head, Brookhaven National Laboratory; Andrei Kolmakov, Daniel Sunday, R. Joseph Kline, NIST-Gaithersburg**

Recent developments in semiconductor nanomanufacturing towards next generation extreme ultraviolet (EUV) lithography now allows for fabrication of leading edge microchips with even smaller features. EUV lithography utilizes higher photon energy than the previous industry standard, which allows for patterning with higher resolution. This is compromised by low photoabsorption cross-sections for elements commonly found in conventional polymer-based photoresists. As a result, tin oxide organometallic nanoclusters are being optimized as photoresist materials, as Sn has a cross section 30x higher than carbon. Unfortunately, this transition to metal oxides yields a different radiation mechanism that results in a solubility contrast compared to their polymer counterparts, while further contrasting in post exposure processing. To identify chemical changes upon EUV exposure, we prepared spin coated thin films of a well-known  $\text{Sn}_{12}$  dodecamer cluster and treated the films with flood exposures for a range of EUV dosages to correlate changes in solubility with structural changes in the resist, including loss of ligands and cross-linking between clusters. In addition to EUV exposure, we also exposed samples to  $\text{O}_2$  and  $\text{H}_2$  plasmas using an AC-driven cold cathode 22 kHz plasma to simulate basic post exposure etch processing. Recently, Plasma-XPS has been developed where x-ray photoelectron spectroscopy (XPS) can be collected during exposure to plasma, allowing for studies of chemical changes in operando. We additionally utilize x-ray absorption spectroscopy (XAS) to examine the electronic structure of the elemental components ex situ, after EUV and/or plasma exposure. XPS before and after EUV irradiation shows a decrease in C 1s intensity along with an increase in the O 1s corresponding to the metal oxide, which is expected as butyl ligands are cleaved and the clusters cross-link. Comparing the C and O K-edges for the unexposed versus EUV exposed samples display a clear change suggestive of an increase in carbonyl chemistry. Furthermore, exposure to  $\text{H}_2$  plasma seems to further pattern the film, likely due to UV emission from the hydrogen Lyman series lines. Overall, we provide a more detailed understanding of the radiation induced chemistry that we anticipate will allow for the design of more efficient EUV photoresists and can lead to advances in EUV lithography.

**CA-ThP-10 ToF-SIMS Analysis of Surface Deposited Reaction Species in Selectively Passivated MXene Catalysts, Tobias Misicko, Gabriel Parker, Oak Ridge National Laboratory, USA; Yang Xiao, Louisiana Tech University; Xiao-Ying Yu, Oak Ridge National Laboratory, USA**

Heterogeneous catalysis is controlled by the chemistry of the outermost atomic layers of catalyst surfaces and interfaces, where active metals, promoters, supports, and adsorbed reactants interact to form the catalytic system. A key deactivation pathway is coking, leading to deposition of carbonaceous species onto active surfaces, which causes a progressive loss of catalytic activity over time. Selective passivation of heterogeneous catalysts, the intentional neutralization of unselective and unproductive sites<sup>[1]</sup>, has been applied to platinum catalysts using MXene. MXene is a class of two-dimensional (2D) metal carbides with the empirical formula of  $\text{M}_{n+1}\text{X}_n\text{T}_x$ , where M is an early transition metal, X is a carbon or nitrogen, and T is a surface functional group (such as F or OH). In prior work,<sup>[2,3]</sup> platinum was deposited onto  $\text{Mo}_2\text{TiC}_2$  MXene via incipient wetness impregnation to yield a 0.5 wt.% Pt/ $\text{Mo}_2\text{TiC}_2$  nanolayer catalyst. This catalyst demonstrated excellent activity, with turnover frequencies (TOFs) of  $0.4\text{--}1.2 \text{ s}^{-1}$  for the conversion of methane<sup>[2]</sup> and ethane<sup>[3]</sup>. It also exhibited high selectivity, >98% toward  $\text{C}_2$  products in non-oxidative coupling of methane (NOCM) and >95% toward ethylene in catalytic dehydrogenation of ethane, along with robust stability, showing no loss of activity over 72 h and 24 h time-on-stream (TOS) for NOCM and ethane dehydrogenation, respectively. This stability is attributed to the catalyst's strong coke resistance. Following kinetic performance evaluation, time-of-flight secondary ion mass spectrometry (ToF-SIMS) measurements were conducted to probe surface-deposited species formed during the catalytic conversion of short-chain alkanes. Surface spectral analysis, depth profiling, and mass spectral imaging were performed to elucidate the identities and

spatial distribution of carbonaceous species on the 0.5 wt.% Pt/Mo<sub>2</sub>TiC<sub>2</sub> surface. Surface spectral analysis revealed the presence of many hydrocarbon species, including known coke precursors such as tropylium (C<sub>7</sub>H<sub>7</sub>).

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## **CA-ThP-11 Multimodal Raman and Luminescence Techniques for Defect Characterization in Wide Band Gap Semiconductors., Praveena Manimunda, HORIBA**

Wide band gap semiconductors such as SiC, Ga<sub>2</sub>O<sub>3</sub>, and GaN, are essential for next-generation power electronics, optoelectronics, and quantum technologies, yet their performance is strongly influenced by strain and crystallographic defects introduced during growth and processing. In this work, we demonstrate how Raman spectroscopy, Photoluminescence (PL), Time-Resolved PL (TRPL), and Cathodoluminescence (CL) provide a comprehensive, non-destructive characterization platform for evaluating these materials with high spatial and spectral resolution. Case studies on 4H-SiC and SnO<sub>2</sub> doped Ga<sub>2</sub>O<sub>3</sub> nanowires illustrate how this multimodal approach enables the identification of defect-related optical signatures and microstructural non-uniformities that impact material quality and device reliability. Raman spectroscopy reveals crystal quality and strain distributions, while PL and TRPL provide insight into impurity levels, recombination dynamics, and defect-related transitions. CL further correlates luminescence features with microstructural variations at the microscale. Together, these results highlight how integrated optical and electron-beam characterization supports optimization of epitaxial growth, improves device yield, and accelerates materials development across high-performance wide band gap semiconductor technologies.

## **CA-ThP-12 High-Intensity HAXPES Brought to Your Lab: Deepcore-X, Patrick Lomker, Scienta Omicron, Sweden; Daniel A Beaton, Scienta Omicron; Marcus Lundwall, Scienta Omicron, Sweden**

Buried interfaces in complex materials systems play a critical role in determining performance, stability, and functionality. Traditional surface-sensitive techniques struggle to access these interfaces without destructive sample preparation across a multitude of applications: energy storage devices, catalysts, thin-film coatings, quantum materials, and semiconductor heterostructures. However, Hard X-ray photoelectron spectroscopy (HAXPES) has emerged as a powerful non-destructive characterization technique for buried layers, interfaces, and subsurface chemical states.

Requirements for pristine samples and avoiding exposure to air has driven demand for in-situ, local characterization. In response we have developed the next-generation laboratory instrument that integrates soft and hard X-ray photoelectron spectroscopy (XPS & HAXPES) in a single solution, delivering synchrotron-like HAXPES intensities directly in the laboratory for surface and interface science applications - the DeepCore-X system. At the heart of this instrument is the Excillum Ga K $\alpha$  MetalJet source (9.25keV), enabling 1000W continuous operation. The system delivers a several-hundred-fold intensity advantage over conventional solid-anode laboratory HAXPES instruments while maintaining a monochromated beam with a 50 $\mu$ m spot size. This enables deep, high-resolution probing of buried interfaces and subsurface chemistry up to 50nm without sputtering. Coupled with the ScientaOmicron EW4000 hemispherical analyzer (featuring 60° angular acceptance, 200mm radius, and image-corrected wide-angle detection) the DeepCore-X delivers high-throughput, high-fidelity measurements across complex multilayer structures. The system supports automated sample handling, multi-point acquisition, and *in-operando* bias measurement.

We will present recent results and application examples, including comparison measurements with BL46XU at Spring-8 (supplemental Fig. 2), demonstrating how DeepCore-X bridges the gap between conventional surface analysis and depth-resolved characterization of buried interfaces and multilayer materials systems—without requiring synchrotron access. Demonstrating this instrument will play a critical role in future studies of electronic structure, chemical-state evolution, interfacial reactions, charge transfer, and buried-interface phenomena under realistic operating conditions.

## **CA-ThP-13 Machine Learning-Assisted Multimodal Chemical Sensing at Functional Metal-Organic Framework Interfaces, Muhammad Azhar Hayat Nawaz, Muhammad Mansha, Safyan Akram Khan, Shahid Ali, King Fahd University of Petroleum and Minerals, Saudi Arabia**

The integration of functional nanomaterials with artificial intelligence is driving the development of next-generation analytical platforms capable of enhanced sensing accuracy and reliability. Herein, a machine learning-assisted multimodal sensing strategy is developed by coupling fluorescence and electrochemical transduction at functional lanthanide-based metal-organic framework (MOF) interfaces. The multidimensional analytical responses are processed using machine learning algorithms to improve signal discrimination, quantitative prediction, and analytical robustness in complex matrices. The synergistic combination of optical and electrochemical sensing provides complementary information, enabling enhanced analytical confidence compared with single-mode detection. The sensing platform is demonstrated through the analysis of a representative pesticide contaminant in food samples, highlighting its practical applicability as a proof-of-concept. More broadly, this work establishes a versatile framework that integrates functional MOF interfaces, multimodal signal transduction, and data-driven analytics for intelligent chemical sensing, with future potential for applications in environmental surveillance and monitoring of chemical species associated with carbon utilization processes. The proposed strategy provides new opportunities for developing adaptive analytical systems that combine advanced materials with machine learning for next-generation sensing technologies.

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