Applied Surface Science
Room 319 - Session AS-TuA

Applied Surface Science in Action II

Moderators: Tanguy Terlier, Rice University, **Jeffrey Terry**, Illinois Institute of Technology

2:15pm **AS-TuA-1 Quantification of Surface Functional Groups on Nano- and 2D Materials***, Jörg Radnik,* Federal Institute for Material Research and Testing (BAM), Germany **INVITED**

Surface chemistry plays a crucial role in determining the properties of nanoforms. Incorporation into a matrix, solubility, and dispersibility are all strongly influenced by their surface. Therefore, measurements of surface functionalization are essential for nanoforms, both in terms of applicability and regarding safety and sustainability. Despite this importance, the reliable quantification of functional groups on nanoforms remains a challenge. Documentary standards have recently been published or are currently under development, proposing X-ray photoelectron spectroscopy (XPS) as one of the key methods.[1]

In developing protocols for the quantification of surface functionalities, we chose two approaches: (i) correlative measurements, in which the same samples were analyzed using at least two different methods, and (ii) interlaboratory comparisons (ILCs) carried out across multiple laboratories. Both approaches were successfully applied to functionalized graphene nanoplatelets. The comparison of XPS and energy-dispersive X-ray spectroscopy (EDS@SEM) results shows good agreement between the two methods when the different analysis volumes are considered.[2] The international ILC with XPS as method revealed a pronounced influence of excessively high humidity and sample preparation on the results.

XPS and quantitative nuclear magnetic resonance (qNMR) measurements were carried out on aminated silica nanoparticles, enabling a correlation between the N/Si ratio determined by XPS and the amine coverage on the nanoparticle surface derived from NMR.[3] Comparative measurements performed in two laboratories yielded comparable results. Based on the results obtained, uncertainty budgets could also be established.

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[3] Tavernaro, I. et al. Nanoscale Adv. 7 (2025) 6888 DOI: 10.1039/D5NA00794A

2:45pm **AS-TuA-3 Compositional Analysis of Al1-XScXN Thin Films***, Jeffrey Shallenberger, Saeed Almishal, Jon-Paul Maria,* Penn State University*; G. Bruce Rayner,* Kurt J. Lesker Company

Aluminum scandium nitride ($Al_{1-x}Sc_xN$) has received a lot of attention because of its ferroelectric properties and ability to be integrated on silicon. It has been synthesized over a wide compositional range by a variety of growth and deposition techniques including sputtering, molecular beam epitaxy (MBE) and atomic layer deposition (ALD). Determination of the composition of $Al_{1-x}Sc_xN$ thin films has been done by a variety of techniques including: SEM-EDS, XRF, RBS, SIMS and XPS. This talk will focus on the use of XPS to characterize both thin (~30 nm) and thick (300 nm) films produced by ALD and sputtering, respectively. XPS offers depth-resolved compositional information which is important as these films frequently have AlN layers to improve epitaxial growth or are grown on sapphire substrates. EDS, XRF and RBS have relatively poor depth resolution making it difficult or impossible to determine the stoichiometry in these cases, particularly on very thin films. Accurate measurement of oxygen content is also important as high levels of oxygen can degrade the ferroelectric properties. We utilize Rutherford backscattering (RBS) to help calibrate the XPS-derived stoichiometry of a series of $Al_{1-x}Sc_xN$ films ($0.58<x<0.15$). This talk will also discuss the relative advantages and disadvantages of the commonly utilized thin film compositional techniques with a goal of providing guidance to users when selecting an analytical tool.

3:00pm **AS-TuA-4 Surface-Engineered Nanostructures for Wastewater Contaminants Sensing and Its Treatment by Thermocatalysts***, Zong-Hong Lin,* National Taiwan University, Taiwan*; **Jinn P. Chu,*** National Taiwan University of Science and Technology, Taiwan

Ensuring access to clean water is a critical global health challenge, yet the energy required for contaminant monitoring and treatment intensifies resource pressures. Self-powered sensing and remediation systems offer a sustainable path forward. This study introduces multifunctional surface-engineered metallic nanotube arrays (MeNTAs) as a tunable platform for both pollutant detection and degradation. By tailoring composition and structure, we controlled surface wettability across a wide range (5°–144°) to suit specific functions.For detection, a Ni-W-Ni MeNTA with optimal triboelectric output was integrated into a droplet-mode liquid-solid triboelectric nanosensor (DLS-TENS). Modified with ion-selective membranes, it rapidly (20 ms) and sensitively detected heavy-metal ions (Cr^{6+}, Pb^{2+}, Hg^{2+}). For bacterial sensing and thermocatalytic treatment, W-SS MeNTAs were employed. Their hydrophilic stainless-steel nanoparticles enabled in-situ synthesis of aptamer-functionalized gold nanoparticles for highly sensitive E. coli detection (163 mV/decade). Simultaneously, surface Fe^{2+} ions catalyzed •OH radical generation via the Fenton reaction, while adhered Bi_2Te_3 nanoflakes generated H_2O_2 under a small thermal gradient (10 °C). This system achieved significant bacterial inactivation (E. coli: 97%; S. aureus: 95%) and 95% Cr^{6+} reduction.To demonstrate practical utility, we constructed a wireless sensing bottle for real-time wastewater analysis and integrated it with a treatment module for on-site, simultaneous monitoring and purification. This work presents a cost-effective, portable, and self-powered platform, highlighting the strong real-world potential of multifunctional MeNTAs for advanced water purification.

3:15pm **AS-TuA-5 Multi-Technique Structural and Chemical Characterization of Native Oxide Evolution on Niobium Thin Films for Nasa Astrophysics Detectors***, **FEMI AKINRINOLA**, Robbyn Trappen, Nethmi Loku Kankanamge, Tap Raj Bhandari,* West Virginia University, USA*; Thomas Stevenson, Emily Barrentine,* NASA Goddard Space Flight Center*; Mikel Holcomb,* West Virginia University, USA

Niobium-based superconducting detectors, including microwave kinetic inductance detectors (MKIDs) and transition-edge sensors (TESs), are critical for future NASA astrophysics missions operating in the far-infrared to microwave spectral range. Their performance can be strongly influenced by native oxide formation and surface chemical modifications introduced during fabrication and post-processing. Building on our previous X-ray absorption spectroscopy (XAS) investigation of niobium oxide formation in superconducting device structures, we present an expanded study combining XAS with grazing-incidence X-ray diffraction (GIXRD) and X-ray reflectivity (XRR) to obtain complementary chemical and structural information from the near-surface region of Nb thin films.

This work examines Nb films subjected to multiple surface-processing conditions relevant to superconducting device fabrication, including pristine films, buffered oxide etch (BOE) treatments, unbuffered HF treatments, and hydrogen-exposure conditions. Oxygen K-edge XAS is used to probe changes in the local electronic structure and bonding environment associated with oxide evolution, while GIXRD measurements at multiple incidence angles are used to evaluate crystalline phase formation and structural changes near the film surface. XRR measurements provide additional information on film thickness, interface quality, density, and surface roughness. Preliminary XRR analysis indicates that the films remain structurally dense after processing while supporting thin oxide-like surface layers, whereas GIXRD measurements show that crystalline metallic Nb remains the dominant phase in the films studied thus far.

Rather than relying on a single characterization technique, this combined approach is intended to provide a more complete understanding of how different fabrication and surface-treatment conditions influence oxide evolution, crystallinity, and near-surface structure in Nb-based superconducting materials. Ongoing analysis is focused on correlating spectroscopic trends observed in XAS with structural information obtained from GIXRD and XRR across the different processing conditions. These results contribute to NASA's broader effort to better understand and optimize superconducting detector fabrication processes for future space-based astrophysics missions.

We acknowledge support from NASA 80NSSC22M0173 and NSF 2417349.

4:00pm **AS-TuA-8 Interfacial Solution Structure Impacts Crystal Growth, Ice Nucleation, and Particle Aggregation***, **Elias Nakouzi,*** Pacific Northwest National Laboratory*; Mingyi Zhang,* University of Oklahoma*; Kuan-Ting Liu, Dong June Jang,* Cornell University*; Philip Brahana, Bhuvnesh Bharti,* Louisiana State University*; Jaehun Chun,* Pacific Northwest National Laboratory*; Ulrich Wiesner, Lara Estroff,* Cornell University **INVITED**

Solution structure at solid-liquid interfaces creates inter-particle forces and chemical potential gradients that influence particle stability, reactivity, and aggregation. However, despite this relevance to a variety of research fields, significant knowledge gaps remain in our understanding of solid-liquid interfaces at the molecular scale. Using a combination of atomic force microscopy, confocal fluorescence microscopy, colloidal theory, and atomistic simulations, we present three case studies examining the effect of interfacial solution structure on particle aggregation, crystal growth, and heterogeneous nucleation. Firstly, we determined that extreme salt concentrations enhance ion correlations at the surface of boehmite nanoparticles, resulting in strong repulsive forces at short particle separations that decrease the rate of particle aggregation. In the second study, we investigated the incorporation of ligand-coated nanoparticles in growing calcite crystals. This process is governed by the interactions experienced by an incoming particle as it penetrates the interfacial hydration layers, as well as the binding energy upon adsorption to the growing crystal surface. Thirdly, we determined that oxidizing a polymer surface results in the assembly of hydration layers at the polymer-water interface, which increase the surface hydrophilicity and the propensity for nucleating ice. We anticipate that these studies will provide insights into the molecular nature of solid-liquid interfaces with important implications on relevant problems in geochemistry, electrochemistry, and materials synthesis.

4:30pm **AS-TuA-10 Surface Distribution, and Thermal Evolution in Nuclear Graphite: What We Can Learn Using XPS***, **Jonathan Counsell,*** Kratos Analytical Limited, UK*; Chris Moffitt,* Kratos Analytical Inc.

Analysis of material samples related to nuclear industry remains an important research area considering the rapid evolution of new nuclear power plants planned for the coming century. Critical in these systems is the neutron moderator material graphite. Understanding the interaction of high-energy particles radiation and these effects on structure and chemistry of the graphitic lattice.

XPS is used to analyse the changes in sp2/sp3 character under high-energy Cs ion bombardment and the relationship between dose and depth of change for impinging ions. We will discuss not just structural changes but also chemical relationships between the ions present in the near-surface region and the constituent graphite. We will show how HAXPES can be used to gain insight also.

The life cycle of a graphitic rods in a reactor involves withstanding harsh environments, therefore, considering the operating environment, we explore the effects of heating to determine possible thermal effects from thermal cycling. Here we will exploit different options for analysis including in-situ thermal treatments of the surface to understand the processes occurring in the surface and the stabilisation of incorporated ions. We will introduce best practices and experimental design considerations to optimise and probe the process taking place.

4:45pm **AS-TuA-11 Energy Materials and the Importance of Cryo-XPS***, **Liam Soomary,*** Kratos Analytical Limited, UK*; Chris Moffitt,* Kratos Analytical Inc.

The development of advanced energy storage materials is crucial for the transition to sustainable energy solutions. Cryogenic X-ray Photoelectron Spectroscopy (cryo-XPS) plays a pivotal role in understanding the surface chemistry and electronic properties of these materials under extreme conditions. This study focuses on vitrification techniques to preserve the transient states of energy storage materials, allowing for accurate characterization before degradation occurs.

Using cryo-XPS, we investigate the surface distribution and chemical composition of various energy storage materials, capturing transient states and reducing beam damage through optimized sample preparation and analysis protocols. Software workflows, developed for specific sample types are utilized to streamline the data acquisition and post-processing steps, ensuring reproducibility and reliability of the results. Additionally, vacuum damage is minimized by employing cryo conditions, which are critical for maintaining the integrity of the sample during analysis.

This work highlights the importance of cryo-XPS in providing detailed insights into the surface properties of energy storage materials, enabling the identification of key mechanisms governing their performance. By integrating vitrification, software workflows, and vacuum optimization, we aim to advance the understanding and development of next-generation energy storage technologies.

5:00pm **AS-TuA-12 In-Vacuo Detection of Ti Hydride Using Advanced XPS Technique***, **Ravi Prakash,*** *Jacqueline van Veldhoven,* Semicon Equipment Lifetime, TNO, the Netherlands Organisation for Applied Scientific Research, Netherlands*; Herman Bekman, Leon Arkesteijn,* Semicon Equipment Metrology, TNO, the Netherlands Organisation for Applied Scientific Research, Netherlands*; Ronald Hultermans, Violeta Navarro Paredes, Mark van de Kerkhof,* ASML, Veldhoven, Netherlands

Titanium has an outstanding strength-to-density ratio, high tensile strength, and is naturally resistant to chemical corrosion. Due to the favourable mechanical properties in harsh environments, titanium is widely used in chemical processing equipment, aerospace structures and marine systems. However, when it is exposed to environments with H_2-gas/H-radicals/H-ions, hydrogen may diffuse into the bulk titanium and start forming hydrides which leads to embrittlement, posing a significant risk under extreme conditions.

To measure hydride formation in titanium in-vacuo is crucial. ToF-SIMS and EBSD are ideal techniques to detect the hydrides, but using these techniques requires a very specific combination of exposure and analysis techniques which is not very common. If a hydride containing Ti sample is exposed to air, the surface state may alter, which limits the ability to detect early stage hydride formation on the surface. Also, our investigation shows that titanium oxidizes rapidly even under UHV conditions.

This work introduces a novel in-situ methodology for detecting titanium hydride using X-ray photoelectron spectroscopy (XPS) focused on valence band measurements. A plasma/radical exposure tool attached to an XPS setup enables in-vacuum sample transfer, which allows to study hydrogen ingress in a controlled environment during exposure to H-plasma/H-radicals/H_2-gas. The approach uses a peak fitting model to isolate the overlapping oxygen and titanium hydride signals in the valence band region. Correlating this signal with the oxygen O 1s content enables the identification of hydride formation. This method reliably differentiates between hydride-containing and hydride-free samples, allowing for the early-stage detection of embrittlement precursors after hydrogen exposure. This method not only improves detection sensitivity but also streamlines the experimental workflow by eliminating the need for additional ex-situ measurements. This allows for quicker testing of more representative samples that could contribute to enhancing lifetime.

References;

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5:15pm **AS-TuA-13 Chemical Mapping of sub-Monolayer Oxygen Functionalities on Acid Treated HOPG Surfaces Through Photo-Induced Force Microscopy***, **David Morgan,*** Cardiff University, UK

Carbon is widely used as a support for heterogeneous catalysts because of their relatively low cost, high surface area and the ease with which precious metals can be reclaimed from the support at the end of the catalyst's active life [1]. In most cases, these carbons are acid washed, before the adsorption of the active component. The wash serves two purposes; it removes unwanted inorganic contaminants present in the carbon; and it introduces oxygenated functionalities that modify the behaviour of the carbon towards the adsorption of the active component and the reaction solvent [2].

Our previous work using HOPG as a model substrate has shown HCl and HNO_3 has a significant impact on the surface topography through intrusion by the acid into the graphite, causing weakening and breakage of the interplanar bonds [3]. Chemical derivatisation studies via XPS, has shown that depending on the acid, the surface forms hydroxyl, carbonyl and nitrate containing functions, however, to date the location of these features, especially with relation to the protrusions are unknown [4].

Herein, we present the first Photo-Induced Force Microscopy (PiFM) studies of acid treated HOPG surfaces allowing the unambiguous correlation of surface topography with sub-monolayer oxygen functionalities through chemically distinct vibrational spectra.

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