

## Surface Science Division

### Room Oregon Ballroom 203-204 - Session SS-ThP

#### Surface Science Poster Session

##### SS-ThP-1 ESI Investigations of Melamine and Cyanuric Acid Clusters and Their Relationship to STM Experiments, *Alex Walter, K. Handy, J. Soucek, S. Kandel*, University of Notre Dame

Pulse deposition, a novel sample deposition technique for scanning tunneling microscopy (STM), sprays molecules onto a surface through a process that forces non-equilibrium, high-energy structures. Electrospray ionization mass spectrometry (ESI-MS) is a “soft” ionization method; molecules of interest do not fully fragment, but instead can be observed whole, as can dimers, trimers, and other supramolecular structures, which is often seen as a limitation. However, this clustering of molecules allows for a “screening” of sorts for pulse-deposited STM experiments, which are significantly more time-intensive. ESI-MS and pulse deposition have similar sample delivery methods, and thus can be compared to each other and used to study molecules in tandem. For example, melamine and cyanuric acid together form highly toxic complexes in the body, but the precise crystallization and intermolecular forces that drive the creation of the toxic clusters is unknown. Using the ESI-MS as a precursor to investigation with pulse deposited STM imaging, we observed several stable noncovalent clusters of melamine and cyanuric acid, ranging from mixed dimers to mixed nonamers (four melamines and five cyanuric acids). In this presentation, I will discuss the results of our ESI-MS experiments on melamine and cyanuric acid as well as comment on STM experiments of different solution ratios and how ESI-MS and STM results can be jointly interpreted.

##### SS-ThP-2 Scanning Tunneling Microscopy Study of the H<sub>2</sub>O-CO Co-Adsorbed Fe<sub>3</sub>O<sub>4</sub>(111) Surface for Understanding the Water-Gas Shift Reaction Mechanism, *Asa Kiuchi, Y. Eda, T. Hirai, T. Shimizu*, Keio University, Japan

Magnetite (Fe<sub>3</sub>O<sub>4</sub>) is an attractive catalyst for the water-gas shift reaction (WGSR), owing to its low cost and minimal environmental impact. However, the reaction path remains unclear despite extensive experimental and theoretical studies. This is partly due to the difficulty in conducting experiments and the complexity of analyzing data on co-adsorbed systems, which has resulted in a lack of clarity in the overall picture.

To clarify the mechanism of the WGSR on magnetite, we performed scanning tunneling microscopy (STM) to observe CO exposed, H<sub>2</sub>O adsorbed, and H<sub>2</sub>O-CO co-adsorbed Fe<sub>3</sub>O<sub>4</sub>(111) surfaces. Our results confirmed that CO molecules do not adsorb on the bare surface at room temperature, agreeing with the previous study based on temperature programmed desorption[1]. Two types of adsorbate species appeared on the surface after the exposure to H<sub>2</sub>O, which we attribute to dissociated species, OH, and molecular water. We also found a larger adsorbate on the H<sub>2</sub>O-CO co-adsorbed surface, although it is unclear if this species is an intermediate of the WGSR due to the complexity of the STM image. We are currently developing image analysis methods to identify and classify the adsorbed species. Our study provides a new insight into the atomistic mechanism of the WGSR on Fe<sub>3</sub>O<sub>4</sub>(111).

[1]C. Lemire *et al.*, *Surf. Sci.* **572**, 103(2004).

##### SS-ThP-3 Growth of Metal Nanoclusters on Thin Layer Moiré Pattern of Graphene and Feo on Single Crystal, *Shilpa Choyal, D. Liu, N. Jiang*, UIC

Graphene, a two-dimensional (2D) carbon crystal with sp<sup>2</sup> hybridization, has attracted much attention in recent years due to its novel chemical, electrical, and mechanical properties. Even materials that do not form cluster superlattices upon room temperature deposition may be grown into such by low-temperature deposition. A graphene monolayer was prepared on an Ir (111) single crystal, ethylene (C<sub>2</sub>H<sub>4</sub>) is pyrolytically cleaved on the surface. The resulting superstructure is examined using scanning tunneling microscopy (STM) and was identified as a well-aligned, incommensurate pattern known as moiré. This moiré pattern arises from overlapping the graphene lattice and the Ir (111) lattice, resulting in alternating bright and dark regions.

The moiré patterns in graphene act as nucleation sites for the growth of plasmonic metal nanoclusters. When Ag/Au metal is deposited on graphene, it nucleates at these sites to form nanoclusters on the surface. Through STM, we have examined the nucleation and growth of these nanoclusters, studying their shape, organization, and structural evolution.

Additionally, we have also investigated the stability of these nanoclusters at different temperatures.

Moiré patterns are also found at iron oxide (FeO) thin layers on Au (111) surfaces. The FeO nanoislands are mostly truncated triangular and exhibit clear moiré superlattices. These superlattices result from the lattice mismatch between FeO and Au (111). In contrast to graphene, FeO demonstrates a preference for wetting by different metals, which imparts unique surface properties. FeO islands have two different growth sites one from Fe-edges and the other from O-edges. In addition to being located on top of FeO islands, different metal prefers to nucleate on the edge of FeO islands, where they selectively grow on the Fe-edges and O-edges.

##### SS-ThP-4 Post-Synthesis Isotopic Purification of Oxygen in TiO<sub>2</sub> via Controllable Surface Injection of Interstitial Atoms, *H. Jeong, Nabil Hilmy Abuyazid, E. Seebauer*, University of Illinois at Urbana Champaign

Isotopically pure semiconductors have important applications for cooling electronic devices and for quantum computing and sensing. Raw materials of sufficiently high isotopic purity are rare and expensive, thereby creating special opportunities for post-synthesis methods that remove isotopic impurities. Through isotopic self-diffusion measurements of oxygen in rutile TiO<sub>2</sub> single crystals immersed in water, we demonstrate fractionation of <sup>18</sup>O by a factor of three below natural abundance in a near-surface region of 10 nm or more. The specially prepared and submerged surface injects large fluxes of O interstitials, which displace lattice <sup>18</sup>O deeper into the solid due to the statistics of interstitial-mediated diffusion combined with steep interstitial gradients. Multiscale modeling offers quantitative insights into how these physical effects work together and how they might be optimized. Both ultraviolet illumination and solution pH affect the experimental injection rate, and demonstrate that adjustments to the chemistry between the surface and fluid can be used to control chemistry between the surface and defects in the bulk. The benefits of such control extend beyond isotopic fractionation to defect engineering, as the injected O interstitials also remove O vacancies and compensate donor H impurities. Importantly, all these effects occur near room temperature. This accesses a regime wherein equilibrium concentrations of native defects become vanishingly small, and where kinetic effects dominate defect behavior. It thereby becomes possible to create materials whose properties circumvent thermodynamic constraints.

##### SS-ThP-5 Analyses of Surface Structure and Chemical States of Carbon Black Nano Particles, *Mari Isagoda<sup>1</sup>, Keio University, Japan; T. Aoki, Asahi Carbon Co., Ltd., Japan; T. Shimizu*, Keio University, Japan

Carbon black (CB) is widely used as pigment and reinforcement material in tire production and is also expected to work as conductive auxiliaries of Lithium-ion batteries. To improve properties for these applications, precise control of the surface structure and chemical states of CB particles is critical. The crystallite model — particles made of small flakes of layered graphene — has been proposed based on transmission electron microscopy (TEM), and the edges of these graphene flakes are expected to be terminated with several types of functional groups [1]. However, the validity of the model and the exact types and locations of functional groups are still in debate. In this study, we employed atomic force microscopy (AFM), scanning tunneling microscopy (STM), Raman spectroscopy, and x-ray photoelectron spectroscopy (XPS) to provide a comprehensive characterization of the surface structure and chemical states of CB particles.

In AFM and STM measurements of samples prepared by the drop-drying method, we observed that carbon black exists as aggregates of particles with a variety of sizes, ranging from approximately 10 nm to 300 nm. Our STM images of small-size particles less than 10 nm in diameter cannot be adequately explained by the crystallite model. Raman spectra of powdered samples showed two peaks centered at 1340 cm<sup>-1</sup> and 1590 cm<sup>-1</sup>, which correspond to D-band and G-band[2], respectively. By comparing the spectra with those of other carbon-based materials, we concluded that the carbon black consists of graphene sheets, as in the crystallite model, but it is rather close to amorphous. XPS suggests the existence of oxygen-containing species.

[1] S. Khodabakhshi, P. F. Fulvio, and E. Andreoli, *Carbon* **162**, 604-649 (2020).

[2] M. A. Pimenta, *et al.*, *Phys. Chem. Chem. Phys.* **9**, 1276-1291 (2007)."

<sup>1</sup> SSD Morton S. Traum Award Finalist

**SS-ThP-6 Surface Chemistry of Zirconium Borohydride on Zirconium Diboride (0001), Ayoyele Ologun, M. Trenary, University of Illinois - Chicago**  
Zirconium diboride ZrB<sub>2</sub> is an extremely hard material with a high melting point of 3246 °C; given these properties, ZrB<sub>2</sub> can be used for various applications, such as high-resistant coatings for body armors and tanks. In addition, it has also been explored as a diffusion barrier in microelectronics. Industrially, highly conformal thin films of ZrB<sub>2</sub> are grown via chemical vapor deposition (CVD), using zirconium borohydride Zr(BH<sub>4</sub>)<sub>4</sub> as a precursor. While surface chemistry plays a central role in the CVD of ZrB<sub>2</sub> from the Zr(BH<sub>4</sub>)<sub>4</sub> precursor, the surface mechanism is yet to be explored. In this study, we investigated the surface mechanism of Zr(BH<sub>4</sub>)<sub>4</sub> decomposition on a ZrB<sub>2</sub>(0001) surface with reflection absorption infrared spectroscopy (RAIRS), temperature-programmed desorption (TPD), and X-ray photoelectron spectroscopy (XPS). The RAIRS spectra obtained on exposing the ZrB<sub>2</sub>(0001) surface at 90K to Zr(BH<sub>4</sub>)<sub>4</sub>(g) closely matched that of the pure compound, indicating adsorption of Zr(BH<sub>4</sub>)<sub>4</sub> without decomposition. However, new surface intermediates were formed upon heating to 280 K, as shown by the retention of the νB-Ht stretch (2569 cm<sup>-1</sup>) and δH-B-H bend (1228 & 1057 cm<sup>-1</sup>) in the RAIRS spectra. These surface intermediates were tentatively identified as either BH<sub>4</sub> or BH<sub>3</sub> and were found stable up to 330 K. Temperature-programmed desorption studies revealed the desorption of B<sub>2</sub>H<sub>6</sub> and H<sub>2</sub> at around 470 K.

**SS-ThP-7 An Annotated Compendium of X-Ray Photoelectron Spectroscopy (XPS) Spectra, Samira Jafari, M. Linford, A. Dean, B. Kulbacki, S. Ko, Brigham Young University**

X-Ray photoelectron spectroscopy (XPS) is a powerful tool for studying surfaces, where, in its conventional embodiment, it is sensitive to the upper 5 – 10 nm of materials. XPS is widely used throughout science and technology. In XPS, photoemission of core electrons is a result of X-rays striking a surface. Two types of XPS spectra are collected: survey spectra and narrow scans. Survey spectra reveal the elements at surfaces. Narrow scans provide chemical/oxidation state information about those elements. XPS spectra are sometimes inappropriately acquired because of (a) contamination on the surface, (b) contamination in the instrument, e.g., due to a previously-analyzed fluorine-containing sample, and (c) poor method development, e.g., a failure to take a sufficient number of scans. For these reasons, it is important for analysts to have comparison spectra in their work. We intend to collect a number of XPS spectra of different materials and compile them into an annotated compendium that can help other XPS users. Spectral processing will largely be with CasaXPS. Data acquisition of narrow scans will be undertaken at low pass energies that will minimize peak FWHM values, while still providing good

statistics for the spectra. We hope this compendium will be a good resource for other researchers and scientists in their interpretation of their XPS spectra.

**SS-ThP-8 Determination of Band Alignment in Semiconductor Heterojunctions by X-Ray Photoelectron Spectroscopy (XPS), Mohamed Nejjib Hedhili, T. Ng, K. Lee, B. Ooi, KAUST, Saudi Arabia; O. Bakr, Kaust, Saudi Arabia**

Heterojunctions are widely used as an essential building block in advanced semiconductor devices because of their multiple functionalities. The electrical and optical properties of heterojunctions are strongly governed by their electronic band alignment.

High-resolution X-ray photoemission spectroscopy (HR-XPS) is proven to be a powerful way of measuring the valence band offsets in semiconductor heterojunctions. This study aims to determine the band alignment to different semiconductor heterojunctions by direct X-ray photoelectron spectroscopy (XPS) measurements. Type-I and Type-II band alignment were obtained.

Design of heterojunction based electronic/photonic devices requires an accurate determination of the band offset. This parameter is crucial to tailor heterojunction based devices as per the operating requirement.

**SS-ThP-9 Localized Plasmon-Controlled Chemistry at and Beyond the Nanoscale, Chamath Siribaddana, S. Rajak, S. Choyal, D. Liu, S. Mahapatra, L. Li, N. Jiang, University of Illinois Chicago**

Probing the effect of the local chemical environment of surface nanostructures is a challenge because the spatial resolution of conventional spectroscopic techniques is limited by the diffraction limit of light. Coupling

light with plasmonic nano-objects creates highly localized surface plasmons (LSPs), which allows us to break this limit. Tip-enhanced Raman spectroscopy (TERS) is one such surface spectroscopic technique that uses the apex of the tip of a scanning tunneling microscope made from a plasmonic metal as a nano object to couple light to the near field. The Raman modes of the nanostructure underneath this tip are greatly enhanced which allows us to obtain chemical information with Angstrom scale spatial resolution. Thus, TERS can probe intermolecular interactions, molecule-substrate interactions, organic-2D material heterostructures, and the reactivity of 2-D materials to reveal how the local chemical environment affects the chemical and physical interactions of a molecule or a nanostructure on a surface. Apart from probing the local environment, the highly localized nature of LSPs can be used to drive energy-intensive and unselective thermally activated chemical reactions using light with a lower energy input and a site-selective manner. The controllability of LSPRs was demonstrated by dissociating a single bond inside a molecule in the presence of multiple equivalent bonds. The insights obtained into the local environment enable precise control of self-assemblies, on-surface reactions, and LSPRs and expand the ability to synthesize nanostructures with tailored electronic, optical, and magnetic properties required for next-generation nanodevices.

**SS-ThP-10 Heterostructured Nanomaterials Fabrication Using a Modular MBE Research Platform, Lukasz Walczak, Research and Development Division, PREVAC sp. z o.o., Raciborska 61, 44-362 Rogow, Poland; M. Florek, Research and Development Division, PREVAC sp. z o.o, Poland; M. Kwoka, Department of Microelectronics, Silesian University of Technology, Poland**

Many important processes such as energy conversion, electrochemical, corrosion, and biological processes take place at solid-gas and solid-liquid interfaces [1-3]. The molecular beam epitaxy (MBE) method is the one of powerful techniques for creating new nanomaterials and it is the key to improving the performance of novel battery generation or renewable energy sources such as solar, wind, or hydropower energy conversion devices. We would like to promote an original, modular ultra-high vacuum system for the fabrication of heterostructured nanomaterials by the molecular beam epitaxy (MBE) method. Its basic element is a vacuum installation, which consists of a sample loading chamber, a substrate preparation (cleaning) chamber, and a proper MBE chamber for the deposition of selected nanomaterials. All vacuum chambers are connected by appropriate vacuum locks and magnetic sample transfers between the above-mentioned chambers, which enables the implementation of all technological and research works without contact of deposited nanomaterials with the atmospheric environment. Vacuum conditions in all of the above-mentioned vacuum chambers are created using independent systems of various types of vacuum pumps. For the control of the substrate cleaning process, the deposition of nano-layers of selected electronic materials, and their initial characterization, the above-mentioned vacuum installation is equipped with electronic control systems and measurement data acquisition systems. Correct operation of the designed and the completed installation has been verified on the example of the deposition of Mg nano-layers on the Si substrate. The conducted technological works and preliminary research works as well as the obtained results confirmed that the designed and assembled modular vacuum system can be very useful for the deposition of nanolayers of selected electronic materials using the MBE method, on the one hand on terms of their potential research applications, and on the other - in terms of their potential industrial applications, incl. for the production of photovoltaic renewable energy sources.

#### References:

- [1]S. Choudhury et al. C 2021, 7, 28.
- [2]A. Asyuda et al. Phys. Chem. Chem. Phys., 2020,22, 10957-10967.
- [3]H. Aldahhak et al. . J. Phys. Chem. C 2020, 124, 11, 6090–6102.

**SS-ThP-12 Angular and Velocity Distributions of NO<sub>2</sub> and O<sub>2</sub> Desorption from an Oxidized Ag(111) Surface, Arved Cedric Dorst, Georg-August Universität, Göttingen, Germany; R. Dissanayake, Max Planck Institute for Multidisciplinary Sciences, Germany; D. Schauer mann, Georg-August Universität, Göttingen, Germany; D. Killelea, Loyola University Chicago; T. Schäfer, Georg-August Universität, Göttingen, Germany**  
Transition group metals are used as catalysts in various oxidation reactions. A common example is silver which found industrial usage in the epoxidation of ethylene to the ethylene oxide. To optimize such processes it is required to understand the dynamics and microscopic details. In this talk, the velocity and angular distribution of NO<sub>2</sub> and

recombinatively-desorbing oxygen from Ag(111) will be presented. Experimentally, we combined velocity-map imaging (VMI) and temperature-programmed desorption (TPD). NO<sub>2</sub> decomposes into NO and O after adsorption on silver. At 510 K, a clean p(4 × 4)-O reconstruction with a maximum oxygen coverage of  $\theta_0 = 0.375$  ML forms on Ag(111).<sup>1</sup> The reaction probability *S* of NO<sub>2</sub> decomposition was studied at this temperature as a function oxygen coverage  $\theta_0$ . *S* is coverage-independent up to  $\theta_0 = 0.3$  ML which is a clear indication for a precursor-mediated mechanism.

In TPD spectra, the recombinative desorption of O atoms occurs as a defined O<sub>2</sub> desorption feature around 600 K. In contrast to NO<sub>2</sub> whose desorption appears thermal, these oxygen molecules exhibit a clearly hyper-thermal velocity distribution; (*v*) is >200 m · s<sup>-1</sup> above a flux-weighted, thermal velocity distribution. Compared to Rh(111),<sup>2</sup> we observe a significantly narrower cos<sup>8</sup>( $\theta$ ) angular distribution for the flux density of O<sub>2</sub> and the velocity distribution differs stronger from a thermal one. Finally, first results for the epoxidation of styrene to styrene oxide will be shown. We observe that the epoxide forms only at high oxygen coverage.

[1] A. Michaelides, K. Reuter, and M. Scheffler, *J. Vac. Sci. Technol. A* **23**, 1487 (2005).

[2] A.C. Dorst, F. Güthoff, D. Schauerma, A.M. Wodtke, D.R. Killelea, and T. Schäfer, *Phys. Chem. Chem. Phys.* **24**, 26421 (2022).

## SS-ThP-14 Growth and Characterization of Bimetallic NiCo Particles on CeO<sub>2</sub>(111) Thin Film Surfaces, *T. Ara, Nishan Paudyal, J. Zhou*, University of Wyoming

Ceria-supported Ni and Co have been of great interest as economical and promising catalysts for chemical reactions including CO oxidation, CO<sub>2</sub> hydrogenation, ethanol reforming, and dry reforming of methane. They can exhibit promising reactivity owing to the strong metal-support interaction. Bimetallic NiCo could provide interesting properties compared to individual metal counterparts due to the synergistic effects between two metals as well as the interaction between the metal and ceria. To elucidate the nature of the activity, we investigated the nucleation, growth, and sintering of metal particles of Ni, Co, and NiCo over well-ordered CeO<sub>2</sub>(111) thin films using scanning tunneling microscopy and x-ray photoelectron spectroscopy under ultrahigh vacuum conditions. Our results indicate that oxidation of the metal (Co, Ni) occurs at the cost of Ce<sup>4+</sup> reduction to Ce<sup>3+</sup> upon deposition of low coverages (< 0.2 ML) of Co or Ni over CeO<sub>2</sub>(111) at room temperature. Both Ni and Co form small particles that are less than two-atomic layer high with no clear preferential nucleation at step edges, suggesting a strong metal-support interaction. Compared to Ni, Co forms relatively smaller particles with a higher particle density on CeO<sub>2</sub>(111) at 300 K that experiences less sintering with heating up to 800 K. Our studies show that bimetallic NiCo particles can be prepared by deposition of Ni followed by Co on CeO<sub>2</sub>. As demonstrated by scanning tunneling microscopy data, Co primarily deposits onto the pre-dosed Ni particles on CeO<sub>2</sub> at 300 K to produce NiCo bimetallic particles and addition of Co can inhibit the sintering of Ni and enhance its thermal stability on ceria with heating.

## SS-ThP-15 DFT Calculations of Cyanuric Acid and Melamine from ESI-MS, *Kaitlyn Handy, A. Walter, J. Soucek, S. Kandel, S. Corcelli*, University of Notre Dame

With the use of electrospray ionization mass spectroscopy (ESI-MS) we are able to observe clusters of cyanuric acid and melamine in solution. The clusters consist of homogenous and heterogenous mixtures with varying ratios of molecules. Several of these clusters are observed forming in high concentrations. Using density functional theory (DFT), calculations are run to model possible cluster structures. Both homogenous and heterogenous clusters arrange in a lattice structure. When there are clusters with disproportionality more cyanuric acid or melamine the molecular structure has variations leading to the molecule not lying flat. Larger clusters show increased stability when compared to smaller clusters.

## SS-ThP-17 Scanning Tunneling Microscopy Studies of Diarylethene Monolayer and Cluster Formation on Noble Metal Surfaces, *Tomoko K. Shimizu, T. Kaneko*, Keio University, Japan; *K. Sagisaka*, National Institute for Materials Science, Japan

The supramolecular assembly on metal surfaces is governed by a subtle balance between intermolecular interaction and molecule-substrate interaction. Even on chemically similar metals, such as Cu, Ag, and Au, the deposition of the same molecule under the same condition may result in different types of assembled structures. We have observed such a case with

a molecule called diarylethene, famous for its photochromism and expected to work as single molecule switching device.

After depositing molecular powder of the open-form isomer on metal surfaces at room temperature, scanning tunneling microscopy (STM) was performed in ultra-high vacuum at liquid helium temperature. Our STM images revealed the presence of both the open-form and closed-form isomers on all three metals. This is due to a stability reversal on metals compared to the gas phase, arising from charge transfer between molecules and metallic substrates[1]. Isolated adsorption was predominant on Cu(111) even after annealing. Co-deposition of NaCl was necessary to form a homogeneous monolayer, which was achieved via ion-dipole interaction[2]. On Au(111), closed-form isomers formed small clusters, such as trimers and tetramers, while the open-form remained isolated. In contrast, larger clusters made of both the open- and closed-form isomers were found on Ag(111), including chain-type clusters and three-fold symmetric chiral clusters. Mild annealing transformed all the open-form isomers to the closed-form isomers, and only three-fold symmetric clusters made of nine or more closed-form isomers were observed. The clusters found on Ag(111) have three-dimensional structures, suggesting local and weak intermolecular interaction. Theoretical analysis is underway to clarify the exact structures and interactions involved.

## References

[1] T. K. Shimizu, et al., *Chem. Commun.* **49**, 8710-8712 (2013).

[2] T. K. Shimizu, et al., *Angew. Chem. Int. Ed.* **53**, 13729-13733 (2014).

## SS-ThP-18 Distinguishing Elements at the sub-Nanometer Scale on the Surface of a High Entropy Alloy, *Lauren Kim, W. Scougal*, University of Wyoming; *P. Sharma*, Lehigh University; *N. Shirato, S. Wiegold*, Argonne National Laboratory; *W. Chen*, Northwestern University; *V. Rose*, Argonne National Laboratory; *G. Balasubramanian*, Lehigh University; *T. Chien*, University of Wyoming

High entropy materials, including high entropy alloys (HEAs), high entropy Van der Waals materials (HEX), and high entropy oxides (HEOs), have drawn the attention of scientists and engineers for their various functionalities and properties. While a wide variety of properties are being studied in these materials, a microscopic understanding is still missing. In this work, the spatial resolving power of scanning tunneling microscopy (STM) is combined with the elemental resolving X-ray absorption spectroscopy (XAS) to achieve this goal. With the unique X-ray assisted tunneling effect, the elemental distributions on the surface of a HEA at the sub-nm scale were revealed by a synchrotron X-ray scanning tunneling microscope (SX-STM). The elemental distribution at sub-nm scale was revealed by maximizing the correlation coefficient between the collected XAS mappings and the atomic scale elemental modeling. The results shown here demonstrate that SX-STM is a promising tool to reveal elements at the sub-nm scale, even for high entropy materials.

## SS-ThP-19 Soft X-Ray Spectro-Microscopy for Electrochemical Interfaces, *Xiao Zhao, E. Carlson, T. Mefford, W. Chueh*, Stanford University

Most electrochemical reactions occur at interfaces. In response to applied voltage, electron and energy transfer between the first few atomic layers of electrochemical active materials and absorbates. Electrochemical reactions are also highly heterogeneous, as most electrochemical processes preferentially take place around active sites with certain facets, coordination structure and chemical environment. Characterizing these reactions requires a spectroscopic imaging platform with interfacial sensitivity and chemical sensitivity. **Direct characterization of electrochemical interfaces** is further complicated by the presence of liquid electrolyte, which requires the measurement performed *in-situ/operando*.

Here we are presenting the recent development of Scanning Total Electron Yield X-ray Microscopy (STEYXM) based on the Scanning Transmission X-ray Microscopy (STXM), which enables *in-situ* X-ray spectroscopic imaging of various electrochemical interfaces with **25nm spatial resolution and ~5nm surface sensitivity**. A custom electrochemical flow cell is used with three electrodes setup. The traditional transmission mode could provide complimentary bulk information of electrodes, while the electron yield mode maps the surface structure and oxidation state of electrodes, as well as the local electrochemical double layer structure. We anticipate that the development of STEYXM will enable the investigation of surface electrochemistry and advance our fundamental understanding of electrochemical reactions.

# Thursday Evening, November 9, 2023

**SS-ThP-20 Effect of Heat Treatment on Silicon Carbide Reinforced Aluminum Matrix Composite Fabricated Through an Optimized Stir Casting Process, Conner Neely, D. Madiraju, M. Rabea, California State Polytechnic University, Pomona**

The purpose of this study is to determine the additional effect of heat treating on the hardness, corrosion resistance, and mechanical strength of cast aluminum matrix composites reinforced with SiC. The effects of heat treatment will be compared to an identical but untreated cast aluminum matrix composite reinforced by SiC, and additionally to pure aluminum. Accordingly, both composite samples were created from the same batch of matrix alloy in combination with SiC, melted and homogenized in an induction furnace to ensure stability and consistency of crystal structure. Microstructure and morphology analyses were conducted, the hardness was measured by the Micro Vickers hardness Tester (HM-200), corrosion of the samples was tested using salt spray chamber, and the tensile strength by a universal test machine. It was found that the heat treatment increased the hardness in the way of wear resistance of the Al-SiC composite, improved the corrosion resistance, the tensile strength, and showed more favorable material properties than the unaltered test sample of the Al-SiC composite.

**SS-ThP-22 Atomic-Scale Hydration Structures Visualized by Three-Dimensional Atomic Force Microscopy (3D-AFM), Keisuke Miyazawa, Kanazawa University, Japan**

Water molecules on a surface influence the chemical reactivity and molecular adsorption behavior of a surface. Given that these interfacial properties are influenced by local interactions between the material surface and water molecules, a deep understanding of the atomic-scale surface structures and their hydration structures is crucial for the design of surface functions. Recently, three-dimensional atomic force microscopy (3D-AFM) was developed as a method for investigating Subnanometer-scale hydration and flexible molecular structures on various surfaces. In 3D-AFM, an interaction force applied to an AFM tip is measured during the AFM tip 3D scanning at a solid-liquid interface to generate a 3D force image with atomic-scale local contrasts reflecting the local density distributions of molecules on a surface. Recent studies revealed the hydration structures of calcite (Fukuma et al., PRB 92 (2015) 155412), fluorite (Miyazawa et al., Nanoscale 8 (2016) 7334), sapphire and quartz (Nagai et al., Nanoscale 15 (2023) 13262). In this presentation, we present the recent studies of atomic-scale hydration structures investigations, and future prospects of 3D visualization of various solid-liquid interfacial phenomena using 3D-AFM.

## Author Index

### Bold page numbers indicate presenter

— A —

Abuyazid, N.: SS-ThP-4, **1**

Aoki, T.: SS-ThP-5, **1**

Ara, T.: SS-ThP-14, **3**

— B —

Bakr, O.: SS-ThP-8, **2**

Balasubramanian, G.: SS-ThP-18, **3**

— C —

Carlson, E.: SS-ThP-19, **3**

Chen, W.: SS-ThP-18, **3**

Chien, T.: SS-ThP-18, **3**

Choyal, S.: SS-ThP-3, **1**; SS-ThP-9, **2**

Chueh, W.: SS-ThP-19, **3**

Corcellii, S.: SS-ThP-15, **3**

— D —

Dean, A.: SS-ThP-7, **2**

Dissanayake, R.: SS-ThP-12, **2**

Dorst, A.: SS-ThP-12, **2**

— E —

Eda, Y.: SS-ThP-2, **1**

— F —

Florek, M.: SS-ThP-10, **2**

— H —

Handy, K.: SS-ThP-1, **1**; SS-ThP-15, **3**

Hedhili, M.: SS-ThP-8, **2**

Hirai, T.: SS-ThP-2, **1**

— I —

Isagoda, M.: SS-ThP-5, **1**

— J —

Jafari, S.: SS-ThP-7, **2**

Jeong, H.: SS-ThP-4, **1**

Jiang, N.: SS-ThP-3, **1**; SS-ThP-9, **2**

— K —

Kandel, S.: SS-ThP-1, **1**; SS-ThP-15, **3**

Kaneko, T.: SS-ThP-17, **3**

Killelea, D.: SS-ThP-12, **2**

Kim, L.: SS-ThP-18, **3**

Kiuchi, A.: SS-ThP-2, **1**

Ko, S.: SS-ThP-7, **2**

Kulbacki, B.: SS-ThP-7, **2**

Kwoka, M.: SS-ThP-10, **2**

— L —

Lee, K.: SS-ThP-8, **2**

Li, L.: SS-ThP-9, **2**

Linford, M.: SS-ThP-7, **2**

Liu, D.: SS-ThP-3, **1**; SS-ThP-9, **2**

— M —

Madiraju, D.: SS-ThP-20, **4**

Mahapatra, S.: SS-ThP-9, **2**

Mefford, T.: SS-ThP-19, **3**

Miyazawa, K.: SS-ThP-22, **4**

— N —

Neely, C.: SS-ThP-20, **4**

Ng, T.: SS-ThP-8, **2**

— O —

Ologun, A.: SS-ThP-6, **2**

Ooi, B.: SS-ThP-8, **2**

— P —

Paudyal, N.: SS-ThP-14, **3**

— R —

Rabea, M.: SS-ThP-20, **4**

Rajak, S.: SS-ThP-9, **2**

Rose, V.: SS-ThP-18, **3**

— S —

Sagisaka, K.: SS-ThP-17, **3**

Schäfer, T.: SS-ThP-12, **2**

Schauer mann, D.: SS-ThP-12, **2**

Scougale, W.: SS-ThP-18, **3**

Seebauer, E.: SS-ThP-4, **1**

Sharma, P.: SS-ThP-18, **3**

Shimizu, T.: SS-ThP-17, **3**; SS-ThP-2, **1**; SS-

ThP-5, **1**

Shirato, N.: SS-ThP-18, **3**

Siribaddana, C.: SS-ThP-9, **2**

Soucek, J.: SS-ThP-1, **1**; SS-ThP-15, **3**

— T —

Trenary, M.: SS-ThP-6, **2**

— W —

Walczak, L.: SS-ThP-10, **2**

Walter, A.: SS-ThP-1, **1**; SS-ThP-15, **3**

Wieghold, S.: SS-ThP-18, **3**

— Z —

Zhao, X.: SS-ThP-19, **3**

Zhou, J.: SS-ThP-14, **3**