Surface Science
Room 305 - Session SS-FrM

Photo- and Electrochemical Energy Conversion

Moderators: Ashleigh Baber, James Madison University, **Bo-Hong Liu**, National Sun Yat-sen University

8:15am **SS-FrM-1 Real-Time Control of Interfacial Dynamics Enables Electrocatalytic C-H and C-C Bond Transformation and Fuel Formation***, **Marcel Schreier**,* University of Wisconsin-Madison **INVITED**

Producing fuels and chemicals using electricity has drawn considerable interest in recent decades. However, research in electrocatalysis, which allows us to link electricity to chemical transformations, remains strongly focused on the electricity-driven transformation of small inorganic molecules such as CO_2, H_2O, N_2, as well as oxygen containing molecules derived from biomass. Yet, comprehensive industrial electrification will require electrocatalytic methods that can promote the reactions that make up the core of the chemicals and fuels industry: n–alkane transformations.

In this presentation, I will show that using electricity to promote n-alkane transformations opens entirely novel avenues of reactivity with the potential to address long-standing challenges in reactivity and selectivity plaguing catalytic alkane chemistry. Specifically, I will show how our group combined fundamental understanding of the interfacial processes occurring in electrocatalytic reactionswith *in-situ* product analysis using electrochemical mass spectrometry, to gain independent control over the elementary steps of complex alkane transformation reactions in real-time. Using the real-time modulation of the electrode potential applied to an electrocatalyst, we were able to independently control the *adsorption* of n-alkanes to electrocatalyst surfaces, initiate the *transformation* of adsorbates while they are bound to the catalyst, and *selectively desorb* desired products, while leaving others bound. These methods allowed us to demonstrate the room-temperature fragmentation of ethane and butane into shorter chain fragments, achieve the dehydrogenation of n-alkanes at room temperature, open pathways towards the oxidation of n-alkanes in fuel cells, and achieve other industrially relevant hydrocarbon transformations at mild conditions. I will discuss the fundamental science behind these pathways and show how they may chart a path towards more targeted transformation of chemical compounds than is possible with current chemical technologies.

8:45am **SS-FrM-3 Exploring Non-Thiol DDQ Self-Assembly on Au(111) through Cyclic Voltammetry, AmbientSTM, and XPS***, **Nazila Hamidi**[1], Cara Plaisance, Alyssa Nanneman, Soumick Naskar, Sanwu Wang, Erin Iski,* University of Tulsa

Redox-active organic molecules on noble-metal surfaces provide model interfaces for studying molecular ordering, interfacial charge transfer, and surface-confined electrochemistry. In this study, the non-thiol adsorption and self-assembly of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ), a strongly electron-accepting quinone, on Au(111) were investigated. Clean Au(111) substrates were immersed at room temperature in a saturated DDQ solution prepared in 0.1 M $HClO_4$ for selected immersion periods, allowing the time-dependent evolution of DDQ adsorption and surface assembly to be valuated. The resulting surfaces were characterized by Cyclic Voltammetry (CV) and ambient Scanning Tunneling Microscopy (STM), while X-ray Photoelectron Spectroscopy (XPS) is being used to evaluate surface composition. CVs showed reproducible DDQ-related redox features after immersion, indicating that electroactive DDQ species are retained at the Au interface after air exposure. The redox peak currents increase systematically with immersion time, consistent with progressive adsorption and increasing surface coverage. Scan-rate-dependent measurements support a surface-confined redox process, as the peak current scales linearly with scan rate rather than with the square root of scan rate. These results indicate that immersion time provides a direct way to tune the density of DDQ molecules on the Au(111) surface. Ambient STM was used to probe Au(111) after DDQ exposure. Compared with clean Au(111), DDQ-modified surfaces show clear changes in surface appearance, suggesting adsorbed molecular features and molecule-induced restructuring of the Au surface. The time-dependent STM observations indicate that DDQ adsorption evolves from early-stage surface modification toward more developed, linear assemblies at longer immersion times. Initial analysis suggests that the molecules tilt at elevated coverages, which allows the assemblies to be stabilized through possible pi-pi stacking and H-bonding. Because DDQ contains electron-withdrawing cyano groups and quinone carbonyl groups, adsorption may involve interactions between DDQ functional groups and Au surface atoms, including cyano-mediated binding or adatom-assisted adsorption. Ongoing XPS analysis will clarify the chemical composition of the DDQ-modified Au(111) surfaces and identify changes in the local chemical environment associated with adsorption. Complementary density functional theory calculations are planned to support plausible adsorption geometries and DDQ–Au interaction pathways.

9:00am **SS-FrM-4 Substrate Electrochemical Inertness as a Prerequisite for Reliable Electrocatalytic Characterization: Cu-Doped CNT Films Assessed by SECCM***, **Miguel Bernal**, Dario Stacchiola,* Brookhaven National Laboratory

Substrate selection is a decisive factor in the accurate electrochemical evaluation of active materials, as the support itself can obscure or distort the measured response. An ideal substrate combines electrical conductivity with chemical and electrochemical inertness under experimental conditions, ensuring that all measured signals are unambiguously ascribed to the material under investigation. In practice, however, true electrochemical neutrality is rarely achieved, since most conductive supports introduce parasitic contributions arising from double-layer capacitance, surface phase transitions, or residual catalytic activity.

In this context, we present copper-doped multi-walled carbon nanotube (Cu-CNT) films synthesized via spray pyrolysis using α-pinene as a renewable carbon precursor and ferrocene as catalyst, followed by microwave-assisted treatment to promote uniform copper incorporation at the nanotube surface. Films were deposited onto ITO substrates and characterized using scanning electrochemical cell microscopy (SECCM) coupled with cyclic voltammetry, enabling spatially resolved mapping of local electrochemical activity across thousands of discrete surface sites.

Systematic SECCM mapping of bare ITO first established its electrochemical profile as a reference baseline. CNT/ITO surfaces exhibited featureless voltammetric profiles with negligible background current, confirming that the nanotube overlayer effectively cover the ITO surface and yields a well-defined, electrochemically featureless support interface. Cu-CNT/ITO surfaces displayed a well-resolved anodic feature attributable to surface copper oxidation, clearly distinguishable from ITO contributions, alongside characteristic hydrogen and oxygen evolution onset potentials. Strikingly, statistical analysis of spatially resolved voltammograms revealed exceptional surface homogeneity across the Cu-CNT film, with markedly lower site-to-site variability in peak potential and current density than typically observed for heterogeneous metal–carbon interfaces.

These results demonstrate that spray pyrolysis combined with microwave treatment produces Cu-CNT surfaces with uniform electroactive site distribution and reproducible interfacial behavior. The electrochemical cleanliness of the CNT support, confirmed through rigorous substrate mapping, positions Cu-CNT films as model platforms for mechanistic studies of copper-mediated electrocatalysis.

9:15am **SS-FrM-5 Atomic-Scale Investigation of Li/Ag(111) Interfaces: New Phases, Li-Ag Alloy Formation and Lithiophilicity***, **Yuchen Niu**, Janice Reutt-Robey,* University of Maryland, College Park

Bimetallic Li alloys are very promising for solid-state battery applications, particularly as thin film interlayer in next-generation Li metal anodes. Exceptional lithiophilicity and Li-transport properties derive from the structure and reversible formation of Li-Ag alloys across a range of compositions. However, atomic-scale knowledge of the structure and Li-transport dynamics of Li-Ag alloy surfaces is limited.

We present an atomically detailed investigation of the formation and evolution of surface and near surface $Li_xAg(111)$ alloys. Lithium is vapor deposited on a well-characterized Ag(111) single crystal substrate under UHV conditions. Lithium films, from sub-monolayer to multilayer thickness, spontaneously form surface alloys at room temperature. Surface alloys, in turn, support and stabilize lithium metallic islands, reflecting their lithiophilicity. The atomic-scale structures, energetics and evolution of Li-Ag surface alloys are mapped with scanning tunneling microscopy (STM), scanning tunneling spectroscopy (STS), low energy electron diffraction (LEED). Unreported Li-Ag phases are revealed, including a $Li_xAg(111)$ solid solution wetting layer (which supports Li islands) and local $Li_1Ag_1(111)$-($\sqrt{3}\times1$) and Li_xAg herringbone phases. Mechanisms of Li_xAg alloy formation, lithiophilicity, stability of $Li/Li_xAg(111)$ interface, and the growth mode of Li islands are discussed. We further explore electric-field driven Li transport,

[1] **SSD Morton S. Traum Award Finalist**

using the STM tip for both introducing a localized field and subsequent characterization tool. Together, these results bridge atomic-scale surface science and device-level interface engineering, offering mechanistic insights for next-generation Li-metal batteries.

9:30am **SS-FrM-6 Tracking Conducting Oxygen Ions in Solid Oxide Fuel CellElectrolytes by NAP-XPS**, *Po-Chiao Li,* National Synchrotron Radiation Research Center, Taiwan*;* ***Bo-Hong Liu,*** National Sun Yat-sen University, Taiwan

Solid oxide fuel cells (SOFCs) are an important energy technology for stationary power generation, distributed energy systems, and integration into future hydrogen economies.[1] Yttria-stabilized zirconia (YSZ), which conducts oxygen ions O^{2-}, is the most common solid electrolyte. The high operating temperatures of 600–1000 °C is considered a drawback, as it results in heat lost, thermal stress, and materials degradation.[2] Fundamental understanding to the behavior of the conducting ion of the electrolyte is a key for the design and improvement of novel solid electrolytes. In this talk, I will present the study of YSZ-based SOFC model systems using Near-Ambient Pressure X-ray Photoelectron Spectroscopy (NAP-XPS). On a YSZ single crystal, we confirmed the presence of the thermal activated oxygen ion. The oxygen ion induces chemical shift in the Y 3d binding energy while marginal change is shown in the Zr 3p spectra. This evidences the local electronic environment modulation of yttrium during the oxygen ion conducting. We further elucidate the influence of gas environments, temperature, and the presence of electrode materials on the oxygen ion. The result provides basic understanding for designing advanced electrolytes with improved performance, durability, and lower-temperature operation.

Keywords:

Solid Oxide Fuel Cell (SOFC) Yttria-Stabilized Zirconia (YSZ)Oxygen Ion ConductivityNear-Ambient Pressure X-Ray Photoelectron Spectroscopy (NAP-XPS)(1) Shi, H.; Su, C.; Ran, R.; Cao, J.; Shao, Z. Electrolyte materials for intermediate-temperature solid oxide fuel cells. Progress in Natural Science: Materials International 2020, 30 (6), 764–774.(2) Kim, S. K.; Lee, H. J.; Moon, J. Y.; Jo, Y.-R.; Lee, J.; Park, J.-H.; Kim, S.-D.; Joo, J. H. Understanding the phase stability of yttria stabilized zirconia electrolyte under solid oxide electrolysis cell operation conditions. Journal of Materials Chemistry A 2024, 12 (14), 8319–8330.

9:45am **SS-FrM-7 Translating Interfacial Ion Activity to Electrochemical Reactivity**, ***Samuel Johnstone***[1]*, Seth Anderson, Evan Grothen, Matthew Gebbie,* University of Wisconsin - Madison

Electrochemical reactivity hinges on the local environment at electrode-electrolyte interfaces, where interactions between the charged electrode, reactive species, and electrolyte ions combine to dictate charge transfer. Of significant interest is how interfacial ion assembly can be leveraged to control reactivity, and many recent studies have worked to identify how particular ions can influence the rate and selectivity of reactions such as hydrogen/oxygen evolution and CO_2 reduction. Electrostatically, ions with the opposite charge as the electrode (counterions) are expected to have a large presence at the interface, while ions with the same charge (co-ions) are expected to have a minor presence. However, many reaction systems utilize large polarizations and highly concentrated electrolytes, leading to correlated, charge-dense interfaces containing both counterions and co-ions. Therefore, it is important to consider the influence of both the counterion and the co-ion on reactivity and determine whether the combined effect of correlated ions differs from what would be expected from each ion individually. Here, we study the hydrogen evolution reaction in mixed salt electrolytes and show that counterions and co-ions act in tandem to significantly alter the interfacial water structure and influence the rate of reaction. We use a combination of interfacial techniques, including X-ray absorption spectroscopy and atomic force microscopy, to connect reactivity to changes in the structural and chemical makeup of the interface and again show that both ionic species together determine interfacial properties. Overall, our work provides fundamental insight into how correlated interfaces play a key role in modulating electrocatalytic reactions.

10:00am **SS-FrM-8 Chemisorbed Oxygen Enables a Bicarbonate-Mediated Pathway for Formic Acid Oxidation on IrO2(110)**, *Jungwon Yun,* The Ohio State University*; Connor Pope, Somraj Patil,* University of Florida*; Aravind Asthagiri,* The Ohio State University*;* ***Jason Weaver,*** University of Florida

Understanding the mechanisms for formic acid oxidation on transition-metal oxides is essential for designing catalysts with improved activity and resistance to CO poisoning. Here, we investigated how chemisorbed oxygen (O_t) influences formic acid oxidation on IrO_2(110) using temperature programmed reaction spectroscopy (TPRS), reflection absorption infrared spectroscopy (RAIRS), ^{18}O-labeling and density functional theory (DFT) calculations. On the stoichiometric surface, RAIRS and DFT show that formic acid undergoes facile deprotonation by lattice oxygen (O_{br}) to form a bridging formate intermediate that subsequently produces CO_2 during TPRS at temperatures near 500 K. In contrast to O_{br}, chemisorbed oxygen (O_t) redirects HCOOH oxidation through a distinct bicarbonate-mediated pathway that becomes increasingly dominant with increasing O_t coverage. In this pathway, HCOOH reacts with adjacent O_t and O_{br} atoms to produce a bridging bicarbonate intermediate that subsequently decomposes to CO_2 near room temperature. Both DFT and ^{18}O-labeling experiments show that an O_t atom is incorporated into the HCO_3 intermediate and that O_t atoms are also incorporated into a large fraction of the room-temperature CO_2 products, providing direct evidence that chemisorbed oxygen participates in the elementary steps leading to low-temperature CO_2 formation. These results demonstrate that chemisorbed oxygen can fundamentally alter oxidation chemistry on IrO_2(110) by creating a new reaction pathway rather than simply promoting oxidation of pre-existing intermediates. These findings expand the mechanistic understanding of formic acid oxidation on transition-metal oxides and provide design principles for oxide catalysts or co-catalysts that exploit chemisorbed oxygen to suppress CO poisoning through alternative oxidation pathways.

10:30am **SS-FrM-10 Caught in the Act: Correlating Chemical Transformations and Structural Evolution Under Reaction Conditions**, ***Slavomir Nemsak,*** Lawrence Berkeley National Laboratory **INVITED**

Understanding how catalysts behave under operating conditions remains a central challenge in surface science. This talk presents a multimodal platform combining ambient pressure X-ray photoelectron spectroscopy (AP-XPS) and grazing incidence X-ray scattering (AP-GIXS) at the APPEXS endstation of the Advanced Light Source, enabling simultaneous chemical and structural characterization at the same sample spot under identical conditions. Case studies include Ni nanoparticle exsolution from perovskite hosts, Ag-Cu bimetallic nanoparticles restructuring during photocatalytic CO_2 reduction, hydrogen storage in Pd-Ni nanoparticles, ligand oxidation on $NaYF_4$ nanoparticles, and nanopatterned ceria under H_2 and CO_2 atmospheres. Through this examples, we demonstrate how coupled chemical and morphological dynamics are revealed only through simultaneous measurement. Neither technique alone captures the full picture, but together they establish mechanistic links between surface chemistry and structural evolution that are directly relevant to catalyst design.

11:00am **SS-FrM-12 The Importance of Interfacial Water for Achieving Long-Range Ordered Assemblies**, ***Kevin Rosso***[2]*, Xiaoxu Li,* Pacific Northwest National Laboratory*; Tuan Ho,* Sandia National Laboratories, USA*; Duo Song, Sebastian Mergelsberg, Jianbin Zhou, Yining Wang, Narendra Adhikari, Nabajit Lahiri, Yatong Zhao,* Pacific Northwest National Laboratory*; Honghu Zhang,* Brookhaven National Laboratory*; Lili Liu,* Pacific Northwest National Laboratory*; Ruipeng Li,* Brookhaven National Laboratory*; Ping Chen, Mark Bowden, James De Yoreo, Zheming Wang, Carolyn Pearce, Xin Zhang,* Pacific Northwest National Laboratory

Self-assembly of nanocrystals by oriented attachment (OA) is a basis for growing larger crystals with defects organized along relict particle boundaries. A critical stage during OA events is the solvent separated state, in which the Angstrom-scale intervening water layers coating the particles provide the time in close proximity needed for interparticle torque acting across the gap to co-align particles.

This talk will feature new experimental and computational insights into how water structure on particle surfaces impacts particle interactions, using two illustrative systems.First, we examine the dispersion/aggregation of three morphologies of hematite nanocrystals in varied aqueous solutions using ex situ electron microscopy and in situ small-angle x-ray scattering. We demonstrate a unique tendency of (104) hematite nanoparticles to maintain a monodisperse state across a wide range of solution conditions

[1] **SSD Morton S. Traum Award Finalist**

[2] **JVST Highlighted Talk**

not observed with (001)-and (116)-dominated particles, which instead form disordered aggregates. Density functional theory calculations reveal an inert, densely hydrogen-bonded first water layer on the (104) facet that favors interparticle dispersion.The finding clearly shows that facet-engineering is a route to controlling the hydration forces that influence the lifetime of the solvent-separated state.

Second, we demonstrate how macroscopic gibbsite mesocrystals emerge from nanoplates guided into staggered positions by directional sliding determined by intervening water layers. Electron microscopy and X-ray scattering reveal the monoclinic superlattice structure, based on nanoplate stacking with a uniform ≈50° stagger along the gibbsite [010] direction. In situ liquid-cell transmission electron microscopy captures preferential sliding along the gibbsite [010] direction, decelerating with increasing particle overlap. Molecular dynamics simulations reveal that this staggered arrangement corresponds to a global free-energy minimum, rather than full alignment. The simulations also confirm that with 1-2 intervening water layers, particle sliding along the [010] direction is energetically favored.The finding clearly shows the critical role of interfacial water for achieving long-range ordered assemblies.

Our collective insights highlight the central role that interfacial water plays in determining the residence time and energy landscape for interparticle configurational searching during aggregation, in turn determining the extent of order achieved and the corresponding properties of the aggregates themselves.

11:15am **SS-FrM-13 Exploring Reactive Oxygen Species for Epoxidation on AgCu Bimetallic Surfaces***, **Ashleigh Baber**,* James Madison University

Surface science provides a powerful framework for exploring the fundamental nature of model heterogeneous catalysis, with the goal of enhancing reactivity, improving selectivity, and identifying active sites. Our research group utilizes ultrahigh vacuum temperature-programmed reaction spectroscopy to probe catalytic reactivity, complemented by low-temperature scanning tunneling microscopy for atomic-scale visualization of model catalysts. We explore the role of hydroxyl groups as reactive oxygen species in the epoxidation of ethylene and propylene over Ag/Cu(111) model catalysts. The presence of Ag and hydroxyls promotes selective partial oxidation to epoxides while suppressing overoxidation to combustion products. By elucidating the relationship between surface structure and catalytic activity, this work contributes to the rational design of more efficient catalysts for industrial applications enhancing performance, reducing waste, and mitigating CO_2 emission.

11:30am **SS-FrM-14 Energetics of Water and Methanol Adsorption on CO-Precovered Pt (111) Surfaces: Solvent - Adsorbate Interactions***, **Arjan Saha**,* Washington State University*; Valeria Chesnyak,* Oregon State University*; Marcus Sharp,* Pacific Northwest National Laboratory*; Nida Janulaitis,* University of Washington*; Zbynek Novotny,* Pacific Northwest National Laboratory*; Charles T. Campbell,* University of Washington*; Líney Árnadóttir,* Oregon State University*; Zdenek Dohnálek,* Pacific Northwest National Laboratory

Carbon monoxide (CO) is among the most widely studied molecules in surface science, serving as a probe for surface structure, a reactant in catalytic processes, and a model adsorbate for investigating adsorbate–adsorbate interactions. Here we employ single-crystal adsorption calorimetry (SCAC) and sticking coefficient measurements to understand the influence of preadsorbed CO on the interaction of Pt(111) with prototypical solvents, methanol and water (D2O). At 100 K, the initial sticking probability of D2O on CO/Pt(111) is high (~0.9) and increases asymptotically to unity with increasing D2O coverage. The corresponding D2O heat of adsorption remains constant over submonolayer coverages and is identical to that of water multilayers. This suggests that D2O binds more weakly on the CO/Pt(111) as compared with bare Pt(111). Therefore, the measured heat arises mainly from D2O–D2O hydrogen bonding, as CO blocks the Pt sites. As the temperature increases from 100 to 130 K, the initial sticking probability decreases from ~0.9 to ~0.2. This indicates that isolated water monomers become unstable and desorb with high probability before they can find other waters by diffusion and be stabilized via cluster formation. With increasing D2O coverage, the sticking probability again increases asymptotically to unity, but only at coverage well above 1 ML. This behavior indicates the formation of three-dimensional D2O clusters, demonstrating that the CO-covered Pt surface behaves as a non-wetting surface for water adsorption. Analogous studies for methanol on CO/Pt(111) reveal a similar trend in the heat of adsorption, indicating that methanol adsorption is also dominated by methanol–methanol hydrogen bonding rather than methanol–Pt interactions. These results show the impact of coadsorbed species on solvent binding and provide important insights into surface interactions relevant to electrodes and catalytic metal nanoparticles.

11:45am **SS-FrM-15 Vibrational Relaxation of Bromine-Water Complexes on a Metal Surface***, **Huijun Han**,* Ulsan National Institute of Science and Technology, Republic of Korea*; Fajar Prihatno,* University of Ulsan, Republic of Korea*; Junseop Noh, Narae Yun,* Ulsan National Institute of Science and Technology, Republic of Korea*; Jaehoon Jung,* University of Ulsan, Republic of Korea*; Hyung-Joon Shin,* Ulsan National Institute of Science and Technology, Republic of Korea

Vibrational energy of an excited molecule is released through intramolecular relaxation or to neighboring molecules or atoms, and the pathway for relaxation can determine the subsequent reaction. Relaxation pathways in aqueous solutions, however, are complicated to resolve because vibrational spectroscopic techniques inherently probe ensembles in crowded environments. This hinders the examination of the relaxation dynamics of an individual molecule. Here, we investigate mode-specific vibrational energy relaxation of a single water molecule coordinated with bromine atoms on Ag(100) using scanning tunneling microscopy (STM) and density functional theory (DFT) calculations. The molecule was vibrationally excited by inelastic tunneling electrons, resulting in either surface hopping of the molecule or migration of a coordinating Br atom. Action spectra of 2·Br-H_2O and 2·Br-D_2O complexes reveal not only fundamental vibrational modes but also bending overtones and combination modes. Notably, the bending overtone emerges in the absence of Fermi resonance, indicating that the Br-water interaction perturbs the harmonic potential. Excitation of the bending overtone switches intramolecular relaxation to interadsorbate relaxation accompanied by Br migration. Our results demonstrate that coupling between the vibrational mode and the reaction coordinate is crucial for controlling the reaction of an individual molecule.

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