Surface Science
Room 209 CDE W - Session SS-MoA
Photo/Electrochemistry

Moderators: Rachael Farber, University of Kansas, **Liney Arnadottir**, Oregon State University

1:30pm **SS-MoA-1 Plasmonic Catalysis: Opportunities, Challenges, and Unresolved Questions***, **Suljo Linic**,* University of Michigan, USA **INVITED**

Metallic plasmonic nanostructures have emerged as an important class of optically active materials. The initial interest in these materials was based on their nano-antenna properties where these materials concentrate electromagnetic UV-vis fields in small volumes at the surface of the nanostructure. A critical problem with using these metallic materials as nano-antennas is that they lose a significant fraction of electromagnetic energy due to the formation of energetic electron-hole (e-h) pairs in the nanostructures.

There has been a growing realization that the formation of energetic charge carriers in the nanoparticles opens avenues for a number of applications including photocatalytic chemical conversion. At the core of these applications is the need to control the rate of formation of energetic e-h pairs, the location of their formation, and their flow in the nanostructure. There has been a high degree of interest in using hybrid nanostructures containing plasmonic nanoparticles, where the plasmonic component controls the interaction of light with the material, while the non-plasmonic component uses the resultant energetic carriers to perform a function. Examples of these materials include metal-metal, metal-semiconductor or metal-molecule hybrids. The use of these multicomponent materials introduces a number of fundamental questions related to the impact of the interface between the plasmonic and non-plasmonic component as well as the presence of the non-plasmonic material on the optical properties of the system, the flow of energy and excited charge carriers in the system.

I will discuss our work in the emerging field of hybrid plasmonic materials, focusing on the underlying physical principles that govern the flow of energy and excited charge carriers in these systems, as well as on common misconceptions and fundamental questions that deserve more attention and warrant additional studies.

References

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2:00pm **SS-MoA-3 Effects of Electric Fields and Solvents on the Surface Chemistry of Catalysis and Electrocatalysis***, **Charles T. Campbell**,* University of Washington

Understanding how liquid solvents affect the adsorption energies of catalytic reaction intermediates on transition metal surfaces, compared to their better-known values in gas phase, is crucial for understanding liquid-phase catalysis and electrocatalysis. For neutral adsorbates, the dominant effect is a decrease in adsorption energy compared to the gas phase by an amount equal to the solvents' adhesion energy to the solid multiplied by the surface area of the solid (that is blocked from solvent adsorption) per mole of adsorbed reactant. However, the electric field near the surface also has a strong effect on adsorption energies. When thermal catalytic or electrocatalytic reactions occur on metal surfaces in liquid solvents like water, an electrical double layer develops near the metal surface with a large electric field which changes with reaction conditions.This electric field affects the energies of adsorbed reaction intermediates and transition states, and therefore reaction rates. I will review ultrahigh vacuum (UHV) surface science studies regarding the effects of electric fields on the energies of adsorbed catalytic reaction intermediates, and show how these can guide predictions about how changes in electric fields from the double layer affect adsorbate energies on metal surfaces in liquids, and serve as benchmarks for validating accuracy of computational studies. In UHV, the electric field felt by an adsorbate can be strongly tuned by the addition of another adsorbed species nearby. Alkali adatoms exert a very strong change in electric field near the metal surface, which changes the energies of coadsorbed catalytic reaction intermediates, their electronic character and their reaction rates, as has been studied extensively in UHV. Assuming that changes in the field have only small effects on the strength of the weak attractions between adsorbate and solvent, the change in adsorbate energy with local electric field in a liquid is the same as in UHV. This approach explains the well-known observation that the binding energy of hydrogen adatoms (H_{ad}) to many late transition metal surfaces, as probed by cyclic voltammetry in water, increases with increasing pH. This change in H_{ad} energy in turn explains pH-induced changes in thermal catalytic and electrocatalytic hydrogenation reaction rates whereby H_{ad} must add to another species.

2:15pm **SS-MoA-4 In Air STM Observation of Au(111) Surface Disturbance Including Au Magic Fingers as Modified by Solvent Choice***, **Nazila Hamidi**, Erin Iski, Dillon Dodge, Rowan Dirks, Lauren Hornbrook,* University of Tulsa

The formation of Au magic fingers on Au(111) surfaces, a phenomenon first observed under "pristine" ultra-high vacuum (UHV) conditions, has long fascinated researchers due to its implications for nanoscale surface manipulation and quantum effects. This study explores the formation of these structures under more relevant conditions, specifically in air and at room temperature, using Scanning Tunneling Microscopy (STM). After exposing Au(111) to a 0.1 M solvent solution, three types of surface disturbances were observed, including the formation of Au magic fingers. The disturbance mechanism involved strong tip-surface interactions and the mass transport of Au atoms, influenced by the solvent choice. Various solvents, including $HClO_4$, HNO_3, HCl, NaOH, CH_2O_2, $CHCl_3$, C_5H_5N, and $C_2H_4O_2$, were tested for their effects on the surface disturbances. The degree of disturbance was categorized into significant & organized disturbance (SOD), significant & disorganized disturbance (SDD), and insignificant disturbance (IND).

The formation of Au magic fingers was primarily observed with solvents classified under SOD, such as $HClO_4$ and HNO_3, which facilitated the diffusion of Au atoms at a rate of 9.9 ± 1.1 nm^2/min. Solvents in the SDD category, like CH_2O_2, showed a diffusion rate of 4.0 ± 1.9 nm^2/min. Our findings reveal that the chemical properties of the solvent, particularly its total charge and electronegativity, play a pivotal role in modulating the surface dynamics of Au(111). The mechanism for the formation of Au magic fingers likely involved the adsorption of solvent molecules to the STM tip, which then picked up and moved Au atoms from the step edges to form nanowires. This study advances our understanding of solvent-surface interactions and demonstrates the potential for ambient STM to probe and manipulate nanostructures under practical, real-world conditions. The ability to form organized nanostructures like Au magic fingers in the air, without needing UHV or protective liquid layers opens new avenues for nanotechnology, catalysis, and surface chemistry applications. By bridging the gap between fundamental surface science and applied chemistry, this work underscores the importance of solvent choice in controlling nanoscale phenomena and paves the way for future innovations in nanomaterial design.

2:30pm **SS-MoA-5 Size-Selected Pt Clusters on N-Implanted HOPG: Oxygen Reduction Reaction and the Effect of Nitrogen Anchor Sites***, **Lokesh Saravanan**,* University of Utah*; Tsugunosuke Masubuchi,* Keio University, Japan*; Zihan Wang,* University of Utah*; Pavel Rublev, Yu Wei, Anastassia N Alexandrova,* University of California at Los Angeles*; Scott L Anderson,* University of Utah

Platinum clusters (Pt_n) have been considered as an effective electrocatalyst for the oxygen reduction reaction (ORR), the hydrogen evolution reaction and alcohol oxidation. In this work, atomically size-selected Pt_n were deposited under soft landing conditions (≤2 eV/atom) on indium tin oxide (ITO) and highly-oriented pyrolytic graphite (HOPG) electrodes in ultra-high vacuum, then studied electrochemically. The ORR activity exhibited strong substrate dependence, with Pt_n on HOPG showing up to a ~5 times increase in mass activity compared to Pt_n on ITO. However, clusters on pristine HOPG are unstable due to sintering, dissolution, and surface poisoning. To overcome the stability issue, nitrogen implantation has been employed to create anchoring sites on HOPG (N-HOPG). Pt_n deposited on N-HOPG showed improved stability against sintering and dissolution while retaining their high catalytic activity. The catalysts have been characterized by a combination of scanning tunneling microscopy (STM) and scanning transmission electron microscopy. X-ray photoelectron spectroscopy (XPS) provided direct evidence of Pt-N interactions, revealing the nitrogen species that serve as chemically active binding sites. Density functional theory (DFT) calculations were performed to simulate both the cluster-surface interaction and the impact dynamics of N/N_2 ions during implantation. These simulations suggests that ion bombardment leads to penetration and modification of surface graphite layers, promoting the formation of anchor or defect-rich sites favorable for Pt_n stabilization. These findings can provide fundamental insights into cluster stabilization mechanisms and the role of nitrogen anchoring sites on HOPG, offering a broader understanding of electrochemical transformations involving various metal clusters. I would like to acknowledge the support from the U.S. Department of Energy, Office of Science, under grant number DE-SC0020125.

2:45pm **SS-MoA-6 Surface Analysis of Planetary Regolith Particles: X-Ray Photoelectron Spectroscopy Results from the Asteroid Bennu***, **Catherine Dukes**, Lianis Reyes Rosa, Adam Woodson, Jackson Glass, Annabel Li,* University of Virginia*; Christopher Snead, Lindsay Keller,* NASA Johnson Space Center*; Michelle Thompson,* Purdue University*; Harold Connelly, Jr.,* Rowen University*; Dante Lauretta,* University of Arizona*; OSIRIS-REx Sample Analysis Team,* NASA

NASA's OSIRIS-REx mission to near-Earth primitive asteroid Bennu returned >120 grams of regolith. The rims of planetary regolith particles can exhibit a composition that is significantly different from the bulk, due either to geochemical processes on the parent body—such as interactions with aqueous environments—or to space weathering processes, including meteoritic impacts or solar wind irradiation. We investigate the surface composition of multiple Bennu particles from aggregate sample OREX-803205-0 via X-ray photoelectron spectroscopy (XPS). We compare these values with published bulk elemental abundances as well as energy-dispersive X-ray (EDS) spectroscopic measurements. XPS analyses were performed on a PHI Versaprobe III small spot instrument; SEM/EDS measurements were made with an FEI Quanta 650 with Oxford silicon drift detector.

The near-surface compositions of the >1 mm particles we analyzed are relatively homogeneous and comparable to the bulk elemental content [1], containing Mg, Si, O, Na, Fe, S, Al, Cr, Mn, C, Cl, and F [2]. The Bennu particle surfaces exhibit lower carbon and oxygen signatures (C: 11–13.4 at-%, O: 56–60 at-%) than the Murchison meteorite surfacial concentrations (C: 16–19 at-%, O: 60–64 at-%) — presumably the result of controlled sample handling and/or indicative of space weathering [3]. The surface abundances of Na (0.6–2.8 at-%), Mg (10.1–13.4 at-%), and Si (10.1–11.9 at-%) are significantly greater for Bennu than Murchison, where <<1 at-% Na has been observed and Mg content is only 3–5 at-% [3]. No Ca was observed on the Bennu particle rims, which is surprising because ICP-MS bulk analysis identifies Ca at concentrations similar to Na [1]. These differences may be the result of sample inhomogeneities, but are most probably indicators of aqueous exposure.

The <1 mm Bennu regolith particles that we analyzed have surface constituents identical to those of the >1 mm particles but exhibit more compositional variation. In particular, S (to 2.1%) and Na (to 3.8%) are highly enriched on some particles. Na enrichment may have originated from salty precipitates generated by a subsurface brine within Bennu's parent body [4], such exposure can also preferentially leach elements such as calcium from particle rims [5].

Acknowledgments: This material is based upon work supported by under contract no. NNM10AA11C issued through the New Frontiers Program.

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3:00pm **SS-MoA-7 X-Ray Photoelectron Spectroscopy informs on Hybrid Photoelectrode Surface Passivation and Durability***, **Jillian Dempsey**,* University of North Carolina at Chapel Hill **INVITED**

Integration of CO_2 reduction molecular catalysts with silicon photoelectrodes can be achieved through covalent attachment methods, such as hydrosilylation, where hydrogen-terminated silicon is reacted to form silicon-carbon bonds. These hybrid photoelectrodes use sunlight to drive the conversion of CO_2 to value added fuels and chemicals. Incomplete reaction of the atop silicon sites can leave reactive sites that are prone to corrosion and lead to the formation of defects that trap photogenerated carriers. X-ray photoelectron spectroscopy provides critical information both on the success of the catalyst attachment, as well as the extent of passivation through detection of SiO_2 growth. Further, XP spectra recorded before and after controlled potential photoelectrolysis inform on the durability of the covalently modified silicon surface and guide the development of improved hybrid photoelectrodes.

4:00pm **SS-MoA-11 Investigation of the Interaction between Water Vapor and Ni Nanoparticles Supported Over Ceria***, Nishan Paudyal, Erik Peterson, Yinghui Zhou,* University of Wyoming*; Sanjaya Senanayake,* Brookhaven National Laboratory*; **Jing Zhou**[1],* University of Wyoming

Understanding the role of water vapor in the growth and sintering of ceria-supported Ni nanoparticles is important since it can provide insights into their promising activity towards reactions in which water is a key reactant. In our study, we prepared fully oxidized $CeO_2(111)$ and partially reduced $CeO_{1.75}(111)$ thin films as model catalytic supports for Ni and studied the detailed nucleation, size, and distribution of Ni nanoparticles under ultrahigh vacuum (UHV) conditions using scanning tunneling microscopy (STM) and x-ray photoelectron spectroscopy (XPS). Deposition of Ni with submonolayer coverages at 300 K produces small particles that are uniformly distributed over both oxidized and reduced ceria surfaces. The particles agglomerate into large three-dimensional features that are on average 1.0 nm high after heating to 700 K. Furthermore, the particles show a clear preferential decoration at step edges on CeO_2 due to enhanced diffusion at elevated temperatures. The Ni particle growth with heating was also observed over reduced ceria. However, due to a stronger interaction between Ni and reduced ceria with defects (e.g. oxygen vacancies/Ce^{3+}), smaller Ni particles are formed over $CeO_{1.75}$. Exposure of water vapor to the Ni/ceria surface at 300 K can influence the sintering behavior of Ni, causing the formation of significantly flatter particles with heating compared to Ni over adsorbate-free ceria that are attributed to unique adsorbate-metal-oxide interactions. We also conducted the experiments when water was first dosed onto ceria supports followed by subsequent Ni deposition at 300 K. Surface defects (e.g. oxygen vacancies) over reduced ceria are the active sites for water dissociation to form hydroxyl groups at 300 K. This creates fewer nucleation sites for Ni upon deposition over $CeO_{1.75}$ at room temperatures and an extensive decrease in its particle density. Predosed water over $CeO_{1.75}$ also causes significant sintering of Ni nanoparticles with subsequent heating to higher temperatures. Our experimental findings suggest distinct adsorbate-metal-oxide interactions are key to tune the properties of Ni nanoparticles over reducible $CeO_x(111)$ surfaces caused by water exposure. Such interactions are essential in consideration for further modification of Ni-based catalysts for improved reactivity and stability.

4:15pm **SS-MoA-12 The Effect of Surface Reaction Intermediates on the Interaction of Solvent Molecules with Pt(111) Model Catalyst***, **Valeria Chesnyak**,* Oregon State University*; Arjan Saha,* Washington State University*; Marcus Sharp,* University of Washington*; Zbynek Novotny,* Pacific Northwest National Laboratory*; Nida Janulaitis, Charles T. Campbell,* University of Washington*; Zdenek Dohnalek,* Pacific Northwest National Laboratory*; Liney Arnadottir,* Oregon State University

The interaction energies of molecules with solvents on metal surfaces contribute to processes in heterogeneous catalysis and electrocatalysis. Such interactions are essential in catalytic biomass conversion reactions, pollution cleanup, and energy transition. The impact of solvents on the adsorption energetics of reactants, reaction intermediates, and transition states determines reaction rates and selectivity. While adsorption enthalpies of many reactants on metal surfaces in vacuum have been reported, how they change in the presence of liquids remains poorly understood.[1] Computational methods such as density functional theory (DFT) can predict adsorption energies, including coadsorption and solvation effects at the reactant-solvent-catalyst interface. However, these models require experimental benchmarks for validation, as significant discrepancies persist between calculated and experimental adsorption enthalpies. We present here results aimed to provide such benchmarks for the interaction energies between adsorbed intermediates and solvents on metal surfaces.

Single Crystal Adsorption Calorimetry (SCAC) is the only technique capable of directly measuring the molecular and dissociative enthalpies of adsorption on well-ordered single crystalline surfaces. We employ a state-of-the-art microcalorimeter, developed by the Campbell group,[2] to understand coadsorption-induced effects of common agents in catalysis on surfaces. Specifically, we measure the differential heats of adsorption for water and methanol coadsorbed with CO or hydrogen on clean Pt(111). On Pt(111), CO forms a $\sqrt{3}\times\sqrt{3}$ R30° structure, while molecular hydrogen dissociates to form a (1×1) hydrogen atom adlayer at cryogenic

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[1] **JVST Highlighted Talk**

temperatures (100 K). Understanding the effects of these pre-adsorbed catalytic intermediates on the adsorption energies of the solvents provides important benchmarks for validating computational models.

References:

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[2] Ajo, Henry M., et al. "Calorimeter for adsorption energies of larger molecules on single crystal surfaces." **2004** *Review of Scientific Instruments* 75(11) 4471-4480.

4:30pm **SS-MoA-13 Mechanistic Insights into C–C Bond Formation in Photoinduced and Photocatalytic Reactions on Reducible and Non-Reducible Oxide Surfaces***, Aakash Gupta,* University of Central Fllorida*; Keith Blackman, Eric Segrest,* ***Mihai Vaida,*** University of Central Florida

The formation of carbon–carbon (C–C) bonds is a fundamental step in numerous catalytic processes that drive the synthesis of diverse materials, including commodity chemicals, polymers, cosmetics, and pharmaceuticals. Understanding how C–C bonds are created in heterogeneous reactions at the gas–solid interface, and how this process depends on the properties of the solid surface, is essential for improving the efficiency of these reactions.

This study examines the transformation of single-carbon species into two-carbon species through the coupling of CH_3 radicals and tracks the subsequent evolution of these intermediates on various oxide surfaces. The investigation employs an experimental strategy that integrates time-of-flight mass spectrometry with pump–probe spectroscopy, along with rapid preparation of the surface with molecules.

On irreducible oxide surfaces such as silicon oxide, the reaction is initiated by a pump laser pulse at a central wavelength of 266 nm. This pulse excites the CH_3I precursor molecules into the dissociative A-band, resulting in the formation of CH_3 and I fragments. A subsequent probe laser pulse in the ultraviolet range ionizes the reaction intermediates and final products, which are then detected and analyzed using time-of-flight mass spectrometry. Alongside bimolecular processes that yield I_2 and regenerate CH_3I, the formation of C_2H_6 can be monitored with femtosecond resolution.

On reducible metal oxide surfaces, including TiO_2(110) and amorphous TiO_2, the reaction pathway is more complex and extends over considerably longer timescales. In the case of TiO_2(110), the process begins with photodissociation of adsorbed CH_3I precursor molecules. The resulting CH_3 radicals can couple on the surface to produce C_2H_6, which subsequently undergoes photocatalytic oxidative dehydrogenation to form C_2H_5, C_2H_4, and H_2O. An additional reaction channel involves dehydrogenation of surface-adsorbed CH_3 radicals, leading to the generation of CH_2 species. On amorphous TiO_2 surfaces, however, C–C bond formation predominantly proceeds through dissociative adsorption of CH_3I molecules.

Such investigations provide detailed insights into C–C bond formation across a variety of surfaces and precursors, offering new opportunities to understand and control surface reactions of significant industrial and technological relevance.

4:45pm **SS-MoA-14 Studying the Intermolecular Forces That Drive Magic Number Clustering***,* ***Alex Walter****, S. Alex Kandel,* University of Notre Dame

The intermolecular forces that drive crystallization also drive cluster formation in solution. These clusters can be measured using electrospray ionization mass spectrometry (ESI-MS) as the soft ionization technique does not break up supramolecular clustering, and the information can be used to study pathways to bulk crystallization. Clusters that appear at higher frequencies than would be expected are called magic number clusters and have highly favorable geometries, charge distributions, or energies. In a solution together, guanine and cytosine form magic number tetramers (3:1 guanine: cytosine). During MS/MS experiments, the tetramers break into guanine dimers and 1:1 guanine: cytosine clusters. This is not the expected Watson-Crick binding pattern, but rather a Hoogsteen binding pattern where the N7 purine and the C4 amino group are binding to the N3 and C4 positions of the other guanine. Replacing a guanine with a cytosine stretches this geometry. Substituting the guanines with 9-methylguanine shows the same binding pattern, as the N9 position does not participate in the Hoogsteen binding, but replacing the guanines with 6-O-methylguanine breaks this clustering entirely as the methyl group sterically blocks magic number cluster formation. This technique is applied to other systems, including melamine with cyanuric acid and adenine with thymine/uracil to learn more about magic number clustering, the intermolecular forces that drive solution assembly, and crystallization.

5:00pm **SS-MoA-15 Application of XPS to Detect Low-Energy Electron (1–25 eV) Irradiation-Induced DNA Degradation***,* ***Hao Yu,*** University of Notre Dame*; João Pereira-da-Silva,* Universidade NOVA de Lisboa, Portugal*; Jackson King, Thejaswini Basappa, Bo-An Chen, Pitambar Sapkota,* University of Notre Dame*; Filipe Ferreira da Silva,* Universidade NOVA de Lisboa, Portugal*; Sylwia Ptasińska,* University of Notre Dame

Low-energy electrons (LEEs, typically 1–30 eV) are known to induce DNA damage through surface-localized interactions occurring within only a few nanometers of depth, primarily via dissociative electron attachment (DEA). These processes can lead to localized bond scission and chemical modifications in specific molecular components. To examine these changes, we employed X-ray Photoelectron Spectroscopy (XPS). In this study, we investigated various components of DNA irradiated with LEEs in the 1–25 eV range, using XPS to monitor energy-dependent chemical modifications.

The investigated targets included double-stranded calf thymus DNA and plasmid DNA (pUC18), as well as four nucleosides together with their corresponding nucleobases, representing eukaryotic DNA, prokaryotic DNA, and individual base components, respectively. Samples were deposited on substrates to form dry thin films for surface analysis. The films were then irradiated with LEEs for durations ranging from 30 minutes to 8 hours under ultrahigh vacuum conditions (base pressure ~2×10^{-10} mbar). XPS measurements using an Al Kα X-ray source (70–100 W), focusing on the O 1s, C 1s, N 1s, and P 2p core levels, were performed both during and after irradiation under the same UHV conditions.

At 5 and 10 eV, DNA undergoes core-excited dissociative electron attachment (DEA), and we observed selective cleavage of C–N (N-glycosidic) and C–O bonds in plasmid DNA. Damage patterns at these energies were similar, with the extent of modification increasing progressively with irradiation time. In contrast, at 1 eV, where shape-resonant DEA dominates, chemical modifications were minimal, consistent with the limited effectiveness of DEA at such low energies. The TE buffer remained stable under extended X-ray and LEE exposure, but appeared to enhance the yield of site-selective DNA damage under LEE irradiation. At 25 eV, LEEs promoted DNA degradation through combined ionization and DEA pathways. Analysis of calf thymus DNA showed evidence consistent with cleavage of C–O and C–N bonds and disruption of the phosphate backbone. Comparison among four nucleosides revealed that thymidine and guanosine exhibited the most pronounced spectral changes, while cytidine and adenosine were more resistant. Additionally, nucleosides were more susceptible to electron-induced damage than their corresponding nucleobases, indicating that LEEs may preferentially target sugar moieties and N-glycosidic bonds.

5:15pm **SS-MoA-16 Unmasking Local Phenomena in Electrochemistry through Scanning Electrochemical Cell Microscopy***,* ***Miguel Bernal,*** *Dario Stacchiola,* Center for Functional Nanomaterials, BNL

Electrochemical reactions at solid–liquid interfaces are at the heart of catalysis, energy conversion, and materials durability. However, the traditional way we study them—using electrodes with macroscopic dimensions—tends to blur the picture. Averaging over large surfaces hides the fact that many processes, from the nucleation of a nanoparticle to its eventual dissolution, actually take place in a highly heterogeneous and stochastic manner at the micro- and nanoscale.

In our work, we use scanning electrochemical cell microscopy (SECCM) to confine the electrochemical reaction to tiny, micrometer- and submicrometer-sized regions of interest. This localized probing allows us to capture electrochemical “snapshots” that reveal just how variable processes such as metal nanoparticle growth and dissolution can be from one spot to the next.

By examining hundreds of such localized measurements, we can build statistical maps of activity across a surface, linking electrochemical behavior with substrate heterogeneity and particle-scale transformations. Even more insight emerges when SECCM is combined with complementary high-resolution imaging, such as in-situ electrochemical transmission electron microscopy (EC-TEM), which directly visualizes dissolution one nanoparticle at a time.

Taken together, these approaches show that electrochemical reactivity is not uniform but intrinsically local and dynamic. Understanding this hidden complexity opens new opportunities for the design of durable electrocatalysts and functional nanomaterials, where performance and stability hinge on events occurring at the level of single entities. Local electrochemical probing thus provides a powerful lens to bridge the gap between ensemble electrochemistry and the reality of nanoscale processes.

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