## Surface Science
## Room 305 - Session SS-TuA

### Chemical Reactions

**Moderators: Fang Xu**, The University of Texas at San Antonio, **Ye Xu**, Louisiana State University

2:15pm **SS-TuA-1 Atomic-Scale Insights Into Reactivity and Excitations at Oxide Surfaces**, ***Camilla Ferreira de Sá Codeço,*** Federal University of Rio de Janeiro, Brazil **INVITED**

Oxide surfaces exhibit a strong interplay between local electronic structures, chemical reactivity, and collective excitations, governing processes relevant for catalysis, energy conversion, and infrared nanophotonics. In this work, we investigate how oxidation state, structural disorder, and local coordination affect both surface reactivity and light–matter interactions in model oxide systems.

The reactivity of transition-metal oxide surfaces was investigated considering $CO_2$ hydrogenation and hydroxylation conditions. On α-$Fe_2O_3$(012), near-ambient-pressure X-ray photoelectron spectroscopy (NAP-XPS) and low-energy electron diffraction (LEED) were used to correlate surface structure and Fe charge state with $CO_2$ activation pathways. Exposure of the reconstructed (2×1) surface to $CO_2$ + $H_2$ induces partial surface reduction and the formation of electronically enriched $Fe^{2+}$ sites, which stabilize activated $CO_2^-$ intermediates. Complementary studies on MnO(001) and $Mn_3O_4$(001) surfaces show that hydroxylation by water strongly depends on the oxidation state of surface Mn cations, with $Mn^{2+}$ sites acting as active centers for hydroxyl formation. Together, these results demonstrate that oxide surface reactivity is directly governed by local electronic structure and cation valence.

Beyond surface reactivity, oxide interfaces also support collective excitations that are highly sensitive to local chemical environments. Using synchrotron infrared nanospectroscopy (SINS), we investigated surface phonon polaritons (SPhPs) in amorphous $SiO_2$ films after fluorine-ion irradiation. The dominant SPhP associated with the asymmetric stretching mode exhibits a pronounced reduction in intensity, revealing modifications in the local dielectric response induced by ion-generated disorder. Finite dipole model calculations indicate that the measured response originates from an effective medium composed of mixed $SiO_2$ and $SiO_x$ regions, demonstrating how nanoscale chemical heterogeneity modifies polaritonic excitations at oxide surfaces.

Together, these results show how local electronic structure and chemical heterogeneity govern both catalytic activation pathways and confined light–matter interactions at oxide interfaces.

2:45pm **SS-TuA-3 In Situ AP-STM and AP-XPS Studies of $CO_2$ Activation on Oxide-Tuned PdIn(111) Surfaces**, ***Jiwon Park,*** Korea Advanced Institute of Science and Technology (KAIST), Republic of Korea; *Jeongjin Kim,* Pohang Light Source II, Pohang University of Science and Technology (POSTECH), Republic of Korea; *Jeong Young Park,* Korea Advanced Institute of Science and Technology (KAIST), Republic of Korea

The catalytic conversion of $CO_2$ into value-added products, such as methanol, presents a promising strategy towards carbon neutrality. Palladium-Indium (Pd-In) intermetallic alloys have emerged as a highly selective and effective catalyst for $CO_2$ hydrogenation. However, their surface structures and interactions with $CO_2$ at the solid-gas interface require further understandings. In this study, the $CO_2$ activation pathways on the oxide-tuned PdIn(111) single crystal alloy surfaces were investigated in situ, using ambient pressure scanning tunneling microscopy (AP-STM) and synchrotron-based ambient pressure X-ray photoelectron spectroscopy (AP-XPS).

Under ultra-high vacuum (UHV), the metastable PdIn(111) surface adopts unique surface structure consisting of clusters, atomic protrusions and ordered superstructure domains. Exposure to ambient pressure $O_2$ or $CO_2$ drives dramatic morphological transformations, as dissociated atomic oxygen triggers indium-selective oxidation, and upward segregation of $InO_x$. Such surface restructuring generates interfacial Pd-In-$O_x$ nanostructures with defect-rich oxygen vacancies, serving as highly active sites for $CO_2$ activation and the formation of carbonate intermediates. In contrast, the elevated $O_2$ pressure and temperatures accelerate the dealloying of the Pd-In lattice and the growth of passivating indium oxide overlayer with significantly lower $CO_2$ activation capabilities.

These in situ observations provide fundamental insights into the active role of the metastable PdIn(111) intermetallic alloys surface in $CO_2$ activation. Furthermore, they demonstrate that precisely tuning the PdIn(111) surface oxide structures to maintain active interfacial sites is essential for the rational design of robust Pd-In intermetallic catalyst for $CO_2$ conversion.

3:00pm **SS-TuA-4 Growth, structure and surface chemistry of Pd/In surface alloys and InOx thin films on Pd(111)**, ***Aditya Patidar,*** *Om Amar Zavare, Shubham Ravan, Jason F. Weaver,* University of Florida

**Growth, structure and surface chemistry of Pd/In surface alloys and $InO_x$ thin films on Pd(111**)

Thermo-catalytic conversion of carbon dioxide ($CO_2$) to methanol ($CH_3OH$) is important for mitigating $CO_2$ emissions and producing fuels and value-added products. Indium oxide ($In_2O_3$) has emerged as a promising catalyst for activating $CO_2$ and selectively converting it to methanol while suppressing competing reactions. Adding transition metals (TMs), such as Pd, improves catalytic performance, presumably by promoting efficient $H_2$ dissociation. Under reducing conditions and during $CO_2$ hydrogenation, TMs supported on $In_2O_3$ can transform into TM-In intermetallic compounds, giving rise to complex couplings between surface chemistry and coexisting metal and metal-oxide structures. These effects motivate fundamental studies using model systems.

In this talk, I will first discuss the structural evolution of Pd-In alloys grown on Pd(111) as a function of In coverage and temperature. Temperature-programmed X-ray photoelectron spectroscopy (TP-XPS) demonstrates that Pd-In intermixing produces In-rich surface alloy phases at room temperature and that the surface In concentration decreases with increasing temperature as In dissolves into the bulk Pd. XPS fitting identifies distinct Pd 3d and In 3d components associated with In-poor and In-rich Pd-In phases, whose compositional regimes correlate with the extent of Pd-Pd connectivity within the intermetallic structures. We further show that an ordered ($\sqrt{3} \times \sqrt{3}$)R30° surface alloy ($Pd_2In$) forms upon heating 2 ML In/Pd(111) to 600 K, while InPd(110) domains develop on an initial 6 ML In/Pd(111) surface between ~400 and 525 K.

I will also discuss the formation and structure of $InO_x$ thin films on Pd(111), characterized using low-energy electron diffraction (LEED) and scanning tunneling microscopy (STM). STM shows that $InO_x$ film growth follows the Stranski–Krastanov mechanism, in which $InO_x$ initially forms a well-connected wetting layer up to an oxide coverage of 1 ML (monolayer), after which multilayer $InO_x$ islands develop. Both the wetting layer and thicker oxide islands exhibit (5 × 5) periodicity relative to Pd(111), characteristic of bixbyite $In_2O_3$(111). Lastly, I will discuss cooperative interactions between $InO_x$ islands and the exposed Pd surface regions. XPS and TPD measurements show that $H_2$ dissociates readily on Pd domains, and that a substantial fraction of the resulting H-atoms migrates to neighboring $InO_x$ domains, driving $InO_x$ hydrogenation rather than recombinative $H_2$ desorption. These findings provide molecular-level insights into cooperative metal/oxide interactions to $CO_2$ hydrogenation chemistry.

3:15pm **SS-TuA-5 Activation and Selective Hydrogenation of $CO_2$ on Inverse MgO/Cu(111) Surface**, ***Arephin Islam,*** Brookhaven National Laboratory; *Jose Rodriguez,* Brookhaven National Laboratory and State University of New York at Stony Brook

Industrial $CO_2$ emissions remain a major driver of climate change, motivating the development of efficient capture and conversion materials. Magnesium oxide (MgO) is an earth–abundant, thermally stable oxide having the capacity of $CO_2$ capture and conversion, yet the role of nanoscale structure and metal–oxide interfaces in governing its reactivity remains insufficiently understood. Here, we investigate morphology–dependent $CO_2$ activation and hydrogenation pathways on MgO nanostructures supported on metal substrates using a combined experimental and theoretical approach. Scanning tunneling microscopy (STM), ambient–pressure X–ray photoelectron spectroscopy (AP–XPS), low–energy ion scattering (ISS), temperature–programmed desorption, and catalytic measurements reveal that sub–monolayer MgO clusters (0.3–1.8 nm) dispersed on $CuO_x$/Cu(111) are 4–5 times more reactive toward $CO_2$ than bulk MgO and enable selective hydrogenation to methanol at moderate temperatures (500–550 K). MgO forms nano–islands rich in low–coordinated sites at the oxide–metal interface, producing adsorption and activation patterns distinct from bulk MgO(001), Cu(111), or $Cu_2O$/Cu(111). Spectroscopy indicates hydroxyl formation during $H_2$ activation and carbonate–like $CO_2$ binding. Density functional theory (DFT) shows that while isolated MgO motifs enhance $CO_2$ and $H_2$ activation, they are limited in promoting complete $CO_2$ → $CH_3OH$ conversion. In contrast, the MgO/Cu(111) inverse catalyst exhibits a substantially smoother reaction

landscape, with a highest barrier of only 0.98 eV compared to 1.49 eV on unsupported MgO clusters. The MgO–Cu interface stabilizes $CO_2$–derived intermediates and lowers hydrogenation barriers, enabling both strong $CO_2$ trapping and selective conversion. Together, these findings demonstrate that MgO nanostructuring on Cu creates cooperative interfacial sites that integrate efficient $CO_2$ activation with tunable reactivity, offering a promising route for sustainable $CO_2$ utilization.

4:00pm **SS-TuA-8 Interpreting Composition-Dependent Oxygen Vacancy Mechanisms in Layered Oxides Through Machine Learning Residuals and Electronic Structure Analysis***, **Sara E. Mason**,* Brookhaven National Laboratory **INVITED**

Layered transition metal oxides underpin a wide range of functional materials, including intercalation electrodes and oxide catalysts, where oxygen activity strongly influences stability, reactivity, and electronic behavior. Understanding oxygen vacancy formation across compositionally complex chemistries remains challenging due to the interplay between local coordination, electronic structure response, and redox behavior. Here, we investigate oxygen vacancy energetics across layered oxide chemistries using density functional theory (DFT), random forest tree (RFT) models, and electronic structure analysis.

RFT models successfully capture composition-dependent trends in pristine layered oxide formation enthalpies using physically motivated descriptors, demonstrating that bulk thermodynamic properties remain transferable across chemistries. In contrast, prediction accuracy decreases for oxygen vacancy formation energetics, where systematic residual patterns emerge as a function of both local coordination environment (LCE) and host composition. These structured residuals suggest that defect-driven redox behavior introduces mechanistic complexity not fully represented within generalized descriptor-based models.

To probe the origin of these deviations, we analyze charge redistribution following oxygen vacancy formation. Vacancy accommodation mechanisms vary significantly with composition and extend beyond purely local bonding effects. Ni-rich environments preferentially stabilize vacancies through metal-centered reduction and localized electron accommodation, whereas Mn-rich systems exhibit more heterogeneous redistribution pathways. Co-rich and electronically inactive cation environments suppress conventional transition-metal redox, promoting increased oxygen-centered or delocalized compensation behavior.

Together, these results demonstrate that oxygen vacancy energetics in layered oxides arise from a continuum of composition-dependent electronic response mechanisms that are fundamentally more complex than the pristine-state properties used to describe the host lattice. More broadly, this work highlights an important limitation of transferable ML models for complex oxides: while generalized descriptors can successfully predict bulk thermodynamic properties, defect-mediated redox processes depend sensitively on emergent electronic structure effects that require more mechanistically informed representations. These findings demonstrate how structured ML residuals can serve as physically meaningful indicators of unresolved chemical complexity, providing a pathway for integrating interpretable machine learning with physics-based electronic structure analysis.

4:30pm **SS-TuA-10 Adsorption of Carbon Monoxide on the r-cut Hematite $Fe_2O_3$(012)-(1×1) Surface***, **Moritz Eder**, Johannes Filzmoser, Faith J Lewis, Adam Vavrecka, Maosheng Hao, Tun Sinner, Florian Kraushofer, Margareta Wagner, Christian Schäfer, Jiři Pavelec, Florian Libisch, Gareth S Parkinson,* TU Wien, Austria

Understanding CO adsorption on well-defined metal oxide surfaces provides critical benchmark data for interpreting catalytic processes and infrared spectroscopy on complex powder materials. We present a multi-technique study of CO on the $Fe_2O_3$(012)-(1×1) r-cut surface, which has a structurally homogeneous array of equivalent $Fe^{3+}$ and $O^{2-}$ sites. Nevertheless, temperature-programmed desorption (TPD) reveals three distinct desorption peaks (58, 100, 125 K) despite the homogeneous surface. We attribute this finding to lateral repulsive interactions between CO molecules. Infrared reflection absorption spectroscopy (IRAS) shows blue-shifted CO frequencies relative to the gas phase (>2143 $cm^{-1}$) at low coverage that red-shift with increasing coverage. This is consistent with electrostatic interaction between CO and cationic $Fe^{3+}$ sites. Light polarization-dependent measurements provide direct evidence for tilted CO orientation. Non-contact atomic force microscopy (nc-AFM) reveals protrusions consistent with adsorption atop surface $Fe^{3+}$ sites, with distinguishable static and mobile CO populations. Our multi-technique approach establishes adsorption energetics, vibrational signatures, spatial ordering, and bonding geometry, providing a molecular-level foundation for interpreting complex powder catalyst systems.

4:45pm **SS-TuA-11 A Closer Look at Oxygen Reconstructions of Rh Single Crystals** *, **Elizabeth Serna-Sanchez**, Alexis Gonzalez, Dan Killelea,* Loyola University Chicago

Heavy transition metals such as Rh are commonly used for catalysis and have been heavily studied for oxidative reactions. The fundamental understanding of an oxidized Rh surface is widely investigated on low index single crystals however; limited information is available on the behavior of oxygen on high index surfaces. Using a bi-faceted Rh(111)/(322) crystal, we were able to analyze how oxidation of the two facets compared using scanning tunneling microscopy (STM) to characterize the reconstruction oxidation by surface defects and step width. Alongside with the STM, other techniques such as temperature programmed desorption, and low energy electron diffraction are used to identify the various species of oxygen present on the surface and the structures those oxygen species form.

5:00pm **SS-TuA-12 Desorption Characteristics of CO on Oxidized Rh Surfaces***, **Alexis Gonzalez**, Dan Killelea, Elizabeth Serna-Sanchez,* Loyola University Chicago

Heterogeneous catalysis plays an important role in energy production, environmental remediation, and chemical manufacturing, where reactions occur on solid surfaces. Understanding how oxygen interacts with catalytic metal surfaces is necessary for improving catalytic performance. In this study, we investigate oxygen adsorption on two Rh single-crystal surfaces: the flatRh(111) surface and a bifaceted surface with a flat (111) surface and a stepped (322) surface, which contains a high density of under-coordinated sites. Using Reflection Absorption Infrared Spectroscopy, different oxygen prepared surfaces were exposed to CO to examine how surface structure influences CO adsorption behavior. Comparisons between the two crystal single crystals revealed differences in CO vibrational peak positions, peak shifts, and relative intensities, suggesting the presence of distinct CO adsorption species. The results demonstrate that oxygen pre-coverage and surface morphology affect CO adsorption on Rh surfaces. This study provides understanding into structure sensitive adsorption behavior relevant to catalytic oxidation processes.

5:15pm **SS-TuA-13 CO on a Rh/$Fe_3O_4$ Single-Atom Catalyst: High-Resolution Infrared Spectroscopy and Near-Ambient-Pressure Scanning Tunnelling Microscopy***, Nail El Hocine Barama, Chunlei Wang, Panukorn Sombut, David Rath, Adam lagin,* Institute of Applied Physics, TU Wien, Austria*; Martin Ormos,* Department of Surface and Plasma Science, Charles University in Prague, Czechia*; Lena Puntscher, Faith Lewis,* Institute of Applied Physics, TU Wien, Austria*; Zdenek Jakub,* Central European Institute of Technology (CEITEC), Brno University of Technology, Czechia*; Florian Kraushofer, Moritz Eder,* Institute of Applied Physics, TU Wien, Austria*; Matthias Meier,* Faculty of Physics and Center for Computational Materials Science, University of Vienna, Austria*; Michael Schmid, Ulrike Diebold,* Institute of Applied Physics, TU Wien, Austria*; Cesare Franchini,* Dipartimento di Fisica e Astronomia, Università di Bologna, Italy*; Peter Matvija,* Department of Surface and Plasma Science, Charles University in Prague, Czechia*; **Jiři Pavelec**, Gareth Parkinson,* Institute of Applied Physics, TU Wien, Austria

Infrared (IR) spectroscopy is one of the most powerful techniques in heterogeneous catalysis and remains the most widely used optical spectroscopy in the field. Its various experimental modes enable the investigation of a broad range of samples, from powders studied by diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) to flat single crystals examined by infrared reflection–absorption spectroscopy (IRAS or IRRAS). Owing to this versatility, IR spectroscopy is widely regarded as a bridging technique that connects fundamental surface science with applied catalysis.

In this work, we use a newly developed IRAS setup optimised for dielectric single crystals to investigate CO adsorption on the model single-atom catalyst Rh/$Fe_3O_4$(001).[1] Our spectra resolve three distinct Rh–CO species: monocarbonyls on isolated twofold-coordinated Rh adatoms, monocarbonyls on fivefold-coordinated Rh atoms incorporated into the surface, and gem-dicarbonyls on isolated twofold-coordinated Rh adatoms.[2] These assignments are established unambiguously by combining isotope-substitution experiments ($^{12}CO$, $^{13}CO$, and mixed $^{12}CO/^{13}CO$) with precise control of Rh coverage and surface preparation. The interpretation is further supported by our previous SPM, XPS, and TPD studies, as well as DFT calculations.[3]

Under UHV conditions, monocarbonyls at Rh adatom sites are the predominant species. Gem-dicarbonyl formation is kinetically hindered, occurring primarily through CO-induced dissociation of Rh dimers rather than sequential CO adsorption on isolated adatoms. However, NAP-STM shows that the sequential adsorption pathway becomes accessible at millibar CO pressures, linking the behaviour observed under UHV to that expected under realistic reaction conditions. Although DFT provides qualitative insights, it does not quantitatively reproduce the experimental frequencies and is highly sensitive to the computational parameters, underscoring the need for robust experimental benchmarks. The spectroscopic fingerprints established here provide benchmark reference spectra for oxide-supported single-atom catalysts and a reliable basis for assigning Rh coordination environments in practical catalysts and for evaluating theoretical predictions.

1 D. Rath, *et al.*, *Rev. Sci. Instrum.*, 2024, **95**, 065106.

2 N. E. H. Barama, *et al*., https://arxiv.org/abs/2512.15194v2,.

2 C. Wang, *et al.*, *Angew. Chem. Int. Ed.*, 2024, **63**, e202317347.

5:30pm **SS-TuA-14 Reconciling Metal Coordination in g-$C_3N_4$-based Single Atom Catalysis: A Surface Science Study of Melem-Metal Interactions on Au(111)**, ***Faith Lewis,*** TU Wien, Austria*; Nicolò Allasia,* Polytechnic University of Milan, Italy*; Ola Alayan,* IMEM-CNR and Università degli Studi di Genova, Italy*; Moritz Eder, Viktoria Waidbacher, Margareta Wagner,* TU Wien, Austria*; Letizia Savio,* IMEM-CNR and Università degli Studi di Genova, Italy*; Gianvito Vilé,* Polytechnic University of Milan, Italy*; Gareth S. Parkinson,* TU Wien, Austria

Single-atom catalysis (SAC) is a rapidly growing field of research that combines the advantages of solid supports from heterogeneous catalysis with the tunability of active sites in homogeneous catalysis. SACs also lead to well-defined single-atom centers which can achieve high selectivity. Recently, graphitic carbon nitride (g-$C_3N_4$) has been identified as a framework to stabilize SACs that facilitate reactions such as the ORR and HER. The precursor, melem (2,5,8-triamino-heptazine) is typically polymerized, and the resulting structure features a network of 6-fold nitrogen pockets which stabilize the single atoms. The g-$C_3N_4$ framework is a common conceptual baseline for these catalysts, yet its structural specifics are increasingly debated, with recent studies directly challenging the prevailing model.[1] X-ray absorption spectroscopy (XAS) suggests a 4-fold metal coordination with equal bond lengths, a geometry that cannot exist within the often-proclaimed 6-fold pocket of g-$C_3N_4$.

In this work, we use the surface science approach to reconcile this contradiction through scanning probe microscopy (SPM) and X-ray photoelectron spectroscopy (XPS). On an Au(111) single crystal, melem forms a honeycomb overlayer that is stabilized through hydrogen bonding of the amino groups on the corners with the pyridinic nitrogens on the sides of the triangular molecule. When melem and metals, specifically Ni and Fe, were co-deposited at various temperatures onto Au(111), different overlayers formed. At lower temperatures, more densely packed regions of the same triangular features form next to the honeycomb structure. At higher temperatures, two different motifs form depending on the metal. With Fe, the densely packed regions observed at lower temperatures dominate, while for Ni a new structure appears in which the triangular melem molecules are no longer recognizable on the surface. We conclude this is the result of polymerization and formation of a 2D layer.

Our results indicate that, under these conditions, the Fe-melem species forms dimeric complexes centered by a metal atom. The resulting square-planar coordination environment aligns closely with recent XAS observations, offering a structural explanation for the 4-fold coordination.

1. Allasia, Nicolò, Shuai Xu, Sadaf Fatima Jafri, et al. 2025. “Resolving the Nanostructure of Carbon Nitride-Supported Single-Atom Catalysts.” *Small* 21 (23): 2408286. https://doi.org/10.1002/smll.202408286.

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