

Surface Science

Room 120 - Session SS+AMS-MoM

Dynamics and Mechanisms in Heterogeneous Catalysis

Moderators: Prabhakar Kasala, Brookhaven National Laboratory, Arthur Utz, Tufts University

8:15am **SS+AMS-MoM-1 Accurate Dynamical Modelling of Vibrationally Enhanced N₂ Dissociation on Ru(0001) – Implications (Not Only) for Plasma Catalysis**, *F. van den Bosch, N. Gerrits, Jörg Meyer*, Leiden University, Netherlands

INVITED

Pioneering work of Mehta et al. [1] has quantified the efficiency of plasma-enhanced over conventional (temperature-driven) heterogeneous catalysis - nurturing hopes for a future more sustainable alternative that can be easily upscaled. The focus has been on ammonia synthesis and vibrationally excited states available in the plasma, because the dissociative chemisorption of N₂ molecules on a metal catalyst is usually the rate-limiting step. Prevalent micro-kinetic modeling based on transition state theory (TST) for the reaction rates needs to be extended by introducing vibrational-state-dependent rate constants. To do so, Mehta et al. have postulated that the computationally convenient Fridman-Macheret model often used for reactions in the gas phase [2] also works for surface reactions.

Using N₂ on Ru(0001) as a representative showcase, we scrutinize the effect of vibrational excitations of N₂ on its surface reactivity by using explicit molecular dynamics on an accurate potential energy surface using the quasi-classical trajectory method [3,4]. We compute the dissociative chemisorption probabilities as a function of the initial vibrational state ranging from 0 to 10 vibrational quanta. These calculations yield vibrational efficacies of about 1.8, i.e., vibrational excitations are more considerably more effective for promoting dissociative chemisorption reactions than equivalent amounts of translation energy. We compare our findings to TST-based models and carefully analyze why they cannot capture the vibrationally enhanced dissociation correctly. Finally, we discuss these findings in the context of thermal and plasma-enabled catalysis by critically investigating which molecules dominate the reactivity.

1. P. Mehta, P. Barboun, F. A. Herrera, J. Kim, P. Rumbach, D. B. Go, J. C. Hicks, and W. F. Schneider, Overcoming Ammonia Synthesis Scaling Relations with Plasma-Enabled Catalysis, *Nat. Catal.* 1, 269 (2018). DOI: 10.1038/s41929-018-0045-1
2. A. Fridman, *Plasma Chemistry*, Cambridge University Press, Cambridge, 2008.
3. K. Shakouri, J. Behler, J. Meyer, and G.-J. Kroes, Accurate Neural Network Description of Surface Phonons in Reactive Gas-Surface Dynamics: N₂ + Ru(0001), *J. Phys. Chem. Lett.* 8, 2131 (2017). DOI: 10.1021/acs.jpcllett.7b00784
4. P. Spiering, K. Shakouri, J. Behler, G.-J. Kroes, and J. Meyer, Orbital-Dependent Electronic Friction Significantly Affects the Description of Reactive Scattering of N₂ from Ru(0001), *J. Phys. Chem. Lett.* 10, 2957 (2019). DOI: 10.1021/acs.jpcllett.9b00523

8:45am **SS+AMS-MoM-3 SSD Morton S. Traum Award Finalist Talk: A Priori Designed NiAg Single-Atom Alloys for Selective Epoxidation Reactions**, *Elizabeth E. HappeI¹*, Tufts University; *A. Jalil*, University of California at Santa Barbara; *S. Stratton*, Tulane University; *L. Cramer*, Tufts University; *P. Christopher*, University of California at Santa Barbara; *M. Montemore*, Tulane University; *E. Sykes*, Tufts University

Ethylene oxide, produced via the partial oxidation of ethylene, is among the largest volume chemicals produced by the chemical industry and has one of the largest carbon footprints. Using Ag catalysts, the reaction can achieve high selectivity ~90%, but only with a combination of promoters including Cl, Cs, and Re, and must be run at low conversions (< 15%) to avoid the total combustion of ethylene to carbon dioxide. Herein, we report a theory guided investigation demonstrating that the addition of low concentrations of Ni to Ag(111) lowers the barrier for O₂ dissociation and enables spillover of oxygen atoms to sites on the Ag surface. Temperature programmed desorption experiments quantify the facile dissociation, spillover and desorption of O₂ from NiAg(111) and demonstrate that, unlike all previous studies, Ni addition enables the population of the Ag(111) surface with atomic oxygen in near UHV pressure without having to atomize oxygen or introduce species like NO₂ or O₃. Furthermore, ambient pressure X-ray

photoelectron spectroscopy reveals that Ni not only aids in activation and spillover, but also stabilizes nucleophilic oxygen which is thought to be selective towards total oxidation. These results informed the synthesis and testing of supported catalysts which demonstrated that NiAg single-atom alloy nanoparticles produce ethylene oxide both with greater selectivity and conversion than Ag without the need for a co-flow of Cl and other promoters.

9:00am **SS+AMS-MoM-4 Effect of Surface Diffusion of Methoxy Intermediates on Methanol Decomposition on Pt/TiO₂(110)**, *C. Liu, B. Lu*, Hokkaido University, Japan; *H. Ariga-Miwa*, The University of Electro-Communications (UEC-Tokyo), Japan; *S. Ogura*, Tokyo Denki University, Japan; *K. Fukutani*, The University of Tokyo, Japan; *M. Gao, J. Hasegawa, K. Shimizu*, Hokkaido University, Japan; *K. Asakura*, Ritsumeikan University, Japan; *Satoru Takakusagi*, Hokkaido University, Japan

In oxide-supported metal catalysts, atomic-level understanding of dynamic behavior of intermediate adsorbates such as diffusion, spillover, and reverse spillover is crucial to unravel the origins of catalytic activity and product selectivity. Our previous *in situ* STM study on methanol adsorption process on a Pt/TiO₂(110) surface revealed that methoxy intermediates were formed on five-fold coordinated Ti⁴⁺ (Ti_{5c}) sites by dissociative adsorption of methanol on the Pt nanoparticles, followed by spillover to the TiO₂(110) substrate.^[1] They were mobile at room temperature. In this study, diffusion and thermal decomposition of the methoxy intermediates were examined by STM, density functional theory (DFT) calculation and temperature programmed desorption (TPD), in order to reveal how their diffusion affect activity and product selectivity in the methoxy decomposition on the Pt/TiO₂(110) surface.^[2] The TPD measurements showed that the methoxy intermediates were thermally decomposed at >350 K on the Pt sites to produce CO (dehydrogenation) and CH₄ (C-O bond scission) through their reverse spillover. We have found that activity and product selectivity for the methoxy decomposition was much dependent on the particle density, suggesting that it was controlled by diffusion of the methoxy intermediates. Decrease of the Pt nanoparticle density significantly enhanced the selectivity to CH₄, and thus we propose that Pt-TiO₂ interfacial sites are active for CH₄ formation while the other Pt sites for CO formation.

[1] S. Takakusagi, K. Fukui, R. Tero, K. Asakura, Y. Iwasawa, *Langmuir* 2010, 26, 16392.

[2] C. Liu, B. Lu, H. Ariga-Miwa, S. Ogura, T. Ozawa, K. Fukutani, M. Gao, J. Hasegawa, K. Shimizu, K. Asakura, S. Takakusagi, *J. Am. Chem. Soc.* 2023, 145, 19953.

9:15am **SS+AMS-MoM-5 Simultaneous Tracking of Ultrafast Surface and Gas-Phase Dynamics in Solid-Gas Interfacial Reactions**, *Keith Blackman, E. Segrest, G. Turner, K. Machamer, A. Gupta, M. Pathan*, University of Central Florida, Department of Physics; *N. Berriel*, University of Central Florida, Department of Material Science and Engineering; *P. Banerjee*, University of Central Florida, Department of Material Science and Engineering, Renewable Energy and Chemical Transformations Cluster (REACT); *M. Vaida*, University of Central Florida, Department of Physics, Renewable Energy and Chemical Transformations Cluster (REACT)

ABSTRACT

Real-time detection of intermediate species and final products at the surface and near-surface in interfacial solid-gas reactions is critical for an accurate understanding of heterogeneous reaction mechanisms. In this contribution, an experimental method that can simultaneously monitor the ultrafast dynamics at the surface and above the surface in photoinduced heterogeneous reactions is presented. The method relies on a combination of mass spectrometry and femtosecond pump-probe spectroscopy. As a model system, the photoinduced reaction of methyl iodide on and above a cerium oxide surface is investigated. The species that are simultaneously detected from the surface and gas-phase present distinct features in the mass spectra, such as a sharp peak followed by an adjacent broad shoulder. The sharp peak is attributed to the species detected from the surface while the broad shoulder is due to the detection of gas-phase species above the surface, as confirmed by multiple experiments. By monitoring the evolution of the sharp peak and broad shoulder as a function of the pump-probe time delay, transient signals are obtained that describe the ultrafast photoinduced reaction dynamics of methyl iodide on the surface and in gas-phase. Finally, SimION simulations are performed to confirm the origin of the ions produced on the surface and gas-phase.

¹ SSD Morton S. Traum Award Finalist

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9:30am **SS+AMS-MoM-6 In situ Photoemission of Ru(0001) Model Catalyst in Plasma-activated N₂-H₂ Mixtures, Roland Bliem**, Advanced Research Center for Nanolithography, Netherlands

In plasma-assisted catalysis, reactants are activated by a plasma discharge, resulting in vibrational excitation and the production of radicals and ions. Plasma catalysis with low-energy excitations has been reported to allow for remarkable efficiencies, under specific conditions even beyond the limits of thermal catalysis. More generally, the activation of strong molecular bonds in a plasma can soften the requirements on the operating conditions for important reactions, such as ammonia synthesis, and contribute options for their electrification and operation at small scales. While plasma-activation can facilitate certain reaction steps, the reaction pathway and product selectivity is still largely defined by the catalyst surface, making it key to catalyst design. However, at present the active state of catalyst surfaces in plasma is unknown, and *in situ* information on surfaces exposed to plasma is fully lacking.

Here, we follow the surface composition and chemistry of the Ru, one of the most active elements for conventional ammonia synthesis, *in situ* during exposure to plasma-activated N₂-H₂ mixtures using near-ambient pressure X-ray photoelectron spectroscopy (NAP-XPS). The activation of N₂ by a microwave discharge source results in extremely efficient sticking of nitrogen species to Ru(0001) and polycrystalline Ru surfaces, where they form nitrogen-metal bonds. The temperature-dependent stability of nitrogen species indicates the presence of at least two different configurations. The comparison of XPS spectra of plasma-exposed Ru to Ru nitride films grown using pulsed laser deposition suggests that the nitrogen species formed in plasma likely correspond to adsorbates on terraces and at extended defects. In the presence of hydrogen, the nitrogen peak reveals significant hydrogenation, and residual gas analysis shows the presence of NH₃ and N₂H_n. In a temperature-dependent study up to 300°C, we detect ammonia in the residual gas at all times, whereas the hydrogenated surface species in XPS spectra decrease with increasing temperature, indicating a shorter residence time or their decomposition.

9:45am **SS+AMS-MoM-7 Velocity Map Imaging of Desorbing Oxygen from sub-Surface States of Single Crystals**, A. Dorst, R. Dissanayake, Georg-August Universität, Göttingen, Germany; D. Killelea, Loyola University Chicago; Tim Schäfer, Georg-August Universität, Göttingen, Germany

We combine velocity map imaging (VMI) with temperature-programmed desorption (TPD) and molecular beam surface scattering experiments to record the angular-resolved velocity distributions of recombinatively-desorbing oxygen from single crystal surfaces. We assign the velocity distributions to desorption from specific surface and sub-surface states by matching the recorded distributions to the desorption temperature. These results provide insight into the recombinative desorption mechanisms and the availability of oxygen for surface-catalyzed reactions. We use concepts of detailed balance to analyze translational energy distributions of O₂ when shifted towards hyperthermal energies. These distribution indicate desorption from intermediate activated molecular chemisorption states.

10:30am **SS+AMS-MoM-10 Designing the Local Environment of Single Atom Catalysts for Product Selectivity: Theory Meets Experiment**, Talat Shahnaz Rahman, University of Central Florida

INVITED

Singly dispersed transition metal atoms on oxide surfaces, the so-called single atom catalyst (SAC) have recently been shown to attain chemical activity and selectivity for several technologically important reactions that surpass those of Pt single crystal surfaces, the prototype exemplary catalyst but with a large price tag. Apart from being cost-effective, single atom catalyst offer excellent opportunities for tuning their local environment and thereby their oxidation state, local coordination, and electronic structure. In this talk, I will present results of collaborative work with several experimental groups on singly-dispersed transition metal atoms anchored on metal oxide surfaces, with and without ligands, that have the potential to be cost-effective catalysts with high activity and product selectivity. Examples will include Pd and Pt atoms anchored on ZnO that form a bimetallic local environment consisting of one Pd and three Zn atoms with high catalytic activity for generation of H₂ through methanol partial oxidation (MPO) [1] and Pt atoms stabilized in specific fine-tuned local coordination environments that exhibit strikingly distinct catalytic behaviors in reactions as varied as CO oxidation and NH₃ oxidation [2]. I will also pay attention to the special role played by ligands (1,10-phenanthroline-5,6-dione (PDO)) in emergent catalytic properties of Pd single atoms stabilized on ceria surfaces [3]. I will also draw attention to some factors that control the emerging functionalities of the above systems in controlled confinement.

[1] Y. Tang, et al., Nano Lett. 20, 6255 (2020); T.B. Rawal, et al., ACS Catalysis 8, 5553-5569 (2018).

[2] W. Tan, et al., Nat Commun. 13, 7070 (2022)

[3] E. Wasim, N. Ud Din, D. Le, et al., J. Catalysis 413, 81 (2022)

* The work is supported by NSF grant CHE-1955343 and performed in collaboration with D. Le, N. U. Din, D. Austin, T. Rawal, T. Jiang, and the research groups of F. Tao (U of Kansas), F. Liu (UCF), S. Tait (Indiana U).

11:00am **SS+AMS-MoM-12 Stabilizing and Characterizing Single-Atom Catalysts: Rhodium on Titania**, Faith J Lewis, M. Eder, J. Hütner, D. Rath, J. Balajka, J. Pavelec, G. Parkinson, TU Wien, Austria

Single-atom catalysis (SAC) aims to minimize the amount of precious metals in catalysts while maintaining catalytic activity. SAC often uses oxide supports to stabilize individual transition metals as isolated active sites. A multi-technique surface science approach allows characterization of these sites to determine their coordination structure. In this work, rhodium adatoms, stabilized by carbon monoxide, were studied on a rutile titania (r-TiO₂(110)) surface.

In idealized SAC systems, metal atoms are assumed to stay isolated on oxide surfaces. In reality, this is not always the case. On titania, rhodium adatoms readily sinter into larger clusters above cryogenic temperatures. It has been suggested that adding ligands stabilizes single rhodium atoms on the surface; carbon monoxide has been proposed to form a geminal dicarbonyl (Rh(CO)₂) structure to stabilize the individual rhodium atoms on r-TiO₂(110).¹

The rhodium gem-dicarbonyl infrared (IR) stretch has been used to signify single rhodium atoms on titania surfaces since the turn of the century, but no scanning probe images have been published of this Rh(CO)₂/r-TiO₂(110) system. Using a newly integrated infrared reflection absorption spectroscopy (IRAS) system, a protocol for forming the Rh(CO)₂ species on r-TiO₂(110) in UHV was established. In this talk, I will present scanning tunneling microscopy (STM) and non-contact atomic force microscopy (nc-AFM) images of the rhodium gem-dicarbonyl system after annealing to various temperatures with complementary IRAS and X-ray photoelectron spectroscopy (XPS) data. The images show two distinct double-lobed species, one parallel to the [001] direction and one perpendicular, at a low coverage (0.005 ML). Contradictory to theoretical predictions,² the images show an asymmetry of the two lobes in both confirmations. Our results illustrate that the surface science approach provides unique information about single atom catalysts and is a prerequisite for their accurate theoretical description.

1. Frank, M.; Bäumer, M.; Kühnemuth, R.; Freund, H.-J., Metal Atoms and Particles on Oxide Supports: Probing Structure and Charge by Infrared Spectroscopy. *The Journal of Physical Chemistry B*, 8569-8576 (2001).

2. Tang, Y., Asokan, C., Xu, M. et al., Rh single atoms on TiO₂ dynamically respond to reaction conditions by adapting their site. *Nature Communications*, 4488 (2019).

11:15am **SS+AMS-MoM-13 In-Situ Observation of the Effects of Oxygen-Containing Compounds on MoS₂-Based Catalysts Using Near-Ambient Pressure Scanning Tunnelling Microscopy**, Kerry Hazeldine, M. Hedevang, Aarhus University, Denmark; L. Mohrhusen, Carl von Ossietzky University of Oldenburg, Germany; J. Vang Lauritsen, Aarhus University, Denmark

More than 20% of greenhouse gas emissions in the European Union are produced by the heavy transport and aviation sector. Pyrolysis oil derived from biomass is a promising replacement for fossil fuels for aviation and heavy transport applications and is one of the technologies being researched for the generation of green aviation fuel. Fast pyrolysis is a process of decomposing biomass into pyrolysis oil by rapidly heating it in an oxygen-free atmosphere. An advantage of pyrolysis oil derived from biomass is that it is compatible with the existing infrastructure that is used in the catalysis and distillation of jet fuels and diesel. However, the oxygen content in pyrolysis oil derived from biomass is unacceptably high (up to 50%) and can lead to corrosion and instability [1].

To reduce the oxygen content and produce useful and efficient hydrocarbons from the pyrolysis oil, a pre-treatment and hydrodeoxygenation (HDO) step is required to be added to the refining process. Molybdenum disulphide (MoS₂) has previously demonstrated its effectiveness as a catalyst in hydrodesulphurisation (HDS) and has further shown to be a promising candidate as a catalyst in HDO and is therefore the primary material of interest in this study [2].

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To develop atomistic structure determination of the catalyst and elucidate to the reaction pathways for oxygen-containing molecules during HDO, model studies are required. In this study, the model system based on MoS₂ growth on Au(111), has been exposed to oxygen-containing molecules and characterised in-situ using the surface-sensitive techniques, near-ambient pressure scanning tunnelling microscopy (NAP-STM), and near-ambient pressure X-ray photoelectron spectroscopy (NAP-XPS). By using a gold substrate, the oxygen uptake can be evaluated without background from the oxide support. NAP-XPS shows evidence of O exchange on the sulphide phase, consistent with current theoretical models, whilst the corresponding NAP-STM shows structural changes to the particle shape and size of the sulphide phase. For example, preliminary work shows restructuring in the MoS₂ clusters when exposed to elevated pressures of methanol vapour. By using these complementary techniques, we can gain insight into both the chemical and physical changes of MoS₂ upon exposure to oxygen-containing compounds.

late transition metal nanoparticles across oxides streamlines development of optimal catalysts.

[1] Cao J, Zhang Y, Wang L, Zhang C, Zhou C. Unsupported MoS₂-Based Catalysts for Bio-Oil Hydrodeoxygenation: Recent Advances and Future Perspectives. *Front Chem.* 10, 928806 (2022).

[2] Salazar, N., Rangarajan, S., Rodríguez-Fernández, J. *et al.* Site-dependent reactivity of MoS₂ nanoparticles in hydrodesulfurization of thiophene. *Nat Commun.* 11, 4369 (2020).

11:30am **SS+AMS-MoM-14 Revealing Local Coordination of Ag Single Atom Catalyst Supported on CeO₂(110) and ZrO₂(-111), Syeda Sherazi, D. Le, K. Ye, S. Xie, F. Liu, T. Rahman, University of Central Florida**

Single atom catalyst (SAC) supported on metal oxide surfaces is a promising candidate for various reactions as it possesses high temperature stability and potentially high selectivity. Determining the local atomic coordination and geometric structure of the SAC is important for the understanding of its catalytic performance. In this work, we apply the ab initio thermodynamics approach to investigate the coordination environment of Ag SAC supported on CeO₂(110) and ZrO₂(-111), so chosen as accompanying experimental observations find the former to be a more viable support than the latter. We find that the Ag SAC structure in which Ag is embedded in the CeO₂ lattice with one surface oxygen vacancy nearby is the most favorable on the CeO₂(110) surface while the structure in which Ag embeds in the ZrO₂(-111) lattice without any oxygen vacancy nearby is the most favorable on the ZrO₂(-111) surface. Our results also show that it is easier to create oxygen vacancy near the Ag atom when the support is CeO₂(110) than ZrO₂(-111). We compare the trends in the energetics of NH₃ adsorption and dissociation on Ag SAC supported on CeO₂(110) with those on ZrO₂(-111) to compare with accompanying experimental observations that find the ceria-supported Ag SAC to exhibit a pronounced selectivity in ammonia oxidation. We will report experimental data to compare with our finding and comment on their implications for the catalytic performance of the Ag SAC.

Work is supported by National Science Foundation grant CHE-1955343.

11:45am **SS+AMS-MoM-15 Trends for Predicting Adhesion Energies of Catalytic Late Transition Metal Nanoparticles on Oxide Supports, Nida Janulaitis, The University of Washington; K. Zhao, C. Campbell, University of Washington**

Understanding the energetics of late transition metal nanoparticles dispersed on oxide-based catalyst support materials is important for the development of high-performance catalysts. Metal/support adhesion energies, which are used to estimate the metal chemical potential as a function of metal nanoparticle size, which in turn correlates with the surface reactivity and sintering kinetics of the metal nanoparticles. Single crystal adsorption calorimetry (SCAC) was used to directly measure Cu vapor adsorption energies and Cu chemical potential as a function of Cu coverage on the clean rutile-TiO₂(100) surface, while He⁺ low-energy ion scattering (LEIS) was used to measure the average size of the Cu nanoparticles. By fitting these data to a theoretical model, we extracted the adhesion energy of Cu nanoparticles on rutile-TiO₂(100). By comparing to earlier results for Ag, we find that the adhesion energies of metals on the rutile-TiO₂(100) surface correlate proportionally to the oxophilicity of the metal element. Similar proportional correlations for the adhesion energy of metals to MgO(100) and CeO₂(111) surfaces as a function metal oxophilicity have been previously published. Expanding upon these existing oxide adhesion energy trends with the new rutile-TiO₂(100) data clarifies the structure-function relationship between the physical properties of the oxide supports and their metal adhesion energetics. The ability to predict the adhesion energy, and thus the metal chemical potential versus size, of

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