Monday Morning, November 4, 2024

Surface Science

Room 120 - Session SS+AMS-MoM

Dynamics and Mechanisms in Heterogeneous Catalysis

Moderators: Abner de Siervo, University of Campinas (UNICAMP), Arthur Utz, Tufts University

8:15am SS+AMS-MoM-1 Accurate Dynamical Modelling of Vibrationally Enhanced N_2 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 plasmaenhanced 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 ratelimiting 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 TSTbased 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.

- 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.
- 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.jpclett.7b00784

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. Happel*¹, 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 conversation 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+ (Ti5c) 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 CH_4 (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.

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9:30am SS+AMS-MoM-6 Controlling Surface Sites on CuO Nanoparticles by Annealing Treatments after Synthesis, *S. Rodriguez Bonet*, Instituto de Desarrollo Tecnológico para la Industria Química (CONICET-UNL), Argentina; *K. Hanium Maria*, University of Dhaka, Bangladesh; *M. Bosco*, Universidad Nacional del Litoral, Argentina; *Florencia C. Calaza*, Instituto de Desarrollo Tecnológico para la Industria Química (CONICET-UNL), Argentina

In recent years, transition metal oxides thin films have gained a great attention from material scientists and engineers due to their different properties which in turn provide promising applications in various fields of technology. Cu_xO has beenidentified as promising materials for solar energy conversion and heterogeneous catalysis. In addition to their favorable band gap energies that allow for the utilization of visible light, the low cost, earth abundance, and non-toxicity of Cu are additional advantages for developing Cu-based materials. The performance of the copper oxide thin films can be enhanced by improving the crystal quality and surface morphology of the material. Among different synthesis strategies for thin film fabrications, solution-processed methods, such as hydrothermal and electrophoretic deposition, are attractive in terms of their scalability, financial advantages and eco-friendliness. In this work the synthesis of Cu_xO nanoparticles by solution-processed methods followed by annealing treatments, shows the stabilization of different available surface sites. In summary, results will be presented for CuO nanoparticle synthesis where the materials were calcined at different temperatures ranging from 300 to 500 C, showing mainly CuO chemical composition and structure in the bulk, but a hint to the presence of Cu₂O on the surface is observed by CO IR titration experiments at ambient conditions.

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 detailled balance to analyze translational energy distributions of O_2 when shifted towards hyperthermal energies. These distribution indicate desorption from intermediate activated molecular chemisorption states.

10:00am SS+AMS-MoM-8 Kinetic Monte Carlo Modelling of Hydrogen Oxidation on Pt/Pd Surfaces, Alexander Kandratsenka, MPI for Multidisciplinary Sciences, Germany

Recent velocity-resolved kinetics measurements of water production from gas-phase H_2 and O_2 at Pt and Pd surfaces revealed the complex dependence of reaction rates on the oxygen coverage and step density. We aim to clarify the detailed mechanisms of these oxidation reactions by means of Kinetic Monte Carlo approach with adsorption energies, reaction barriers and transition state geometries determined from *ab initio* calculations, and rate constants derived from the Transition State Theory.

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-5,6dione (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 microcopy (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).
- 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 (MOS_2) has previously demonstrated its

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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].

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 oxygencontaining compounds.

[1] Cao J, Zhang Y, Wang L, Zhang C, Zhou C. Unsupported MoS_2 -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 MoS_2 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 CeO_2 lattice with one surface oxygen vacancy nearby is the most favorable on the $CeO_2(110)$ surface while the structure in which Ag embeds in the $ZrO_2(-111)$ 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 ceriasupported 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-TiO_2(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 Monday Morning, November 4, 2024

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

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