

Fundamental Discoveries in Heterogeneous Catalysis Focus Topic

Room Oregon Ballroom 203-204 - Session HC-ThP

Fundamental Discoveries in Heterogeneous Catalysis Poster Session

HC-ThP-1 Insight into Synergistic Effect of Oxide-Metal Interface on Hot Electron Excitation, Eunji Lee, Korea National University of Education, Republic of Korea; B. Jeon, J. Park, Korea Advanced Institute of Science and Technology (KAIST), Republic of Korea; S. Lee, Korea National University of Education, Republic of Korea

Understanding the role of electron transfer by energy dissipation during chemical reactions on metal catalyst surfaces is significant for elucidating the fundamental phenomena at solid-gas and solid-liquid interfaces [1]. Electronic excitation by molecular interactions between reactants and catalyst surfaces generates a flow of excited electrons with an energy of 1–3 eV; these are called hot electrons [2]. To reveal the chemically induced electronic excitations on metal catalyst surfaces, metal-semiconductor catalytic nanodiodes can be used for real-time hot electron detector [3]. In addition, recently, it was found that when the metal-oxide interface is formed, the excitation of hot electrons can be amplified [4].

In this work, to understand the effect of the oxide-metal interface on hot electron excitation under exothermic chemical reactions, we employ advanced real-time detection of reaction-induced electronic excitation and report, for the first time, direct *in-situ* observations of hot electron transfer on metal-semiconductor Schottky nanodevices with well-controlled oxide-metal interfaces (CeO₂ nanocubes/Pt/TiO₂ Schottky nanodiodes). Direct measurement of the electron transfer according to the concentration of interfacial sites (by controlling the coverages of deposited CeO₂ on Pt) allows us to investigate the effect of the oxide-metal interface on non-adiabatic electronic excitation during catalytic H₂ oxidation. Surprisingly, when compared according to the concentration of the CeO₂/Pt interface, the efficiency of hot electron excitation appears to be the highest at a specific concentration of interfacial sites, proving that the inverse oxide-metal interface plays an important role in improving hot electron excitation under exothermic catalytic reactions. Our *operando* techniques using Schottky nanodiodes with well-controlled oxide-metal interfaces provide conclusive evidence about the promotional role of the inverse oxide-metal interfacial sites in heterogeneous catalysts, which contributes to the rational design of future hot electron-based catalysts [5].

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- [5] Eunji Lee *et al.*, *in preparation*

HC-ThP-2 Chemical Speciation and Structural Evolution of Rhodium and Silver Surfaces with High Oxygen Coverages, Dan Killelea, M. Turano, L. Jamka, M. Gillum, Loyola University Chicago; L. Juurlink, Leiden University, The Netherlands; T. Schäfer, University of Göttingen, Germany

The oxidation silver and rhodium surfaces are compared. In particular, I will discuss how the structures formed at high oxygen coverages differ for the metals and how their chemistries may be affected.

The interaction of oxygen with the surfaces of catalytically active transition metals has attracted much interest because of the relevance to heterogeneous catalysis. Recently, we have shown that oxygen coverages in excess of 1 ML are achievable using gas-phase atomic oxygen (AO) to dose the metal surfaces. This talk will discuss some recent results comparing the uptake of AO and O₂ on Ag(111), Rh(111), and curved Ag(111). On c-Ag(111) surfaces, the geometry of the monoatomic steps determines whether or not O will accumulate and the consequent surface

reconstruction. Conversely, on Rh(111), subsurface oxygen readily forms from exposure to AO. Finally, the uptake of oxygen on Ag(111) is discussed; unlike Rh(111), where little surface reconstruction occurs, Ag(111) undergoes several phase transformations as the oxygen coverage is increased. These results using AO demonstrate that UHV compatible dosing can prepare the same surfaces resulting high pressure O₂ exposures, allowing for quantitative and structural analysis of the oxidized surfaces.

HC-ThP-3 Exploring Field-Assisted Nitrogen Activation with Atom Probe Microscopy, Sten V Lambeets, M. Wirth, D. Perea, Pacific Northwest National Laboratory

Challenges in the development of green electricity and energy storage challenges are leading us to consider NH₃ as a promising future zero-carbon fuel. However, NH₃ production largely relies on the Haber-Bosch process requiring high temperature and pressure, making its production via this means responsible for approximately 1.5% of world CO₂ emissions. To unlock to full economic and environmental potential of NH₃, it is critical to lower energy requirements and generate a zero-carbon NH₃ by coupling with renewable electricity¹. The N₂ reduction reaction to NH₃ using electricity is extensively investigated with single atom electrocatalysts (SACs) recently showing promising results. Due to their morphology, the application of an electrical potential on SACs materials results in local High External Electric Fields (HEEFs) over the single atoms. If those effects present promising outcomes according to theoretical calculations^{2,3}, the values of those HEEFs and the mechanisms involved remains largely unknown and unexplored.

Atom Probe Microscopy (APM) such as Field Ion Microscopy (FIM) and Operando Atom Probe (OAP) are ideal techniques to unravel those mechanisms at the nanoscale since they inherently rely on HEEFs for imaging. In this work, we will illustrate those capabilities with the room temperature N₂ dissociation over Ru single nanoparticle case imaged at the nanoscale using FIM and OAP.

We use the recently developed OAP technique⁴ to effectively measure this dissociation over a 0001-oriented Ru specimen. After fixing a constant HEEF between 15 and 25 V/nm and the temperature at 300K, 1.4x10⁻⁷mbar N₂ pure gas is introduced in the analytic chamber. The electric field either directly ionize N₂ or provoke its dissociation. Dissociated N(ads) are mainly detected over the Ru{1012} facets while ionized N₂⁺⁺ are detected on large areas in the periphery of the imaged apex. The occurrence of one or the other processes is intimately linked to the local surface structures and, subsequently, the local HEEFs.

APM are capable to observe and estimate the HEEF necessary to trigger specific chemical reaction steps such as the N₂ dissociative adsorption (i.e. activation). With an accurate calculation of those HEEF, those values can be extrapolated to create new chemical and reactor system designed to perform N₂ activation at relatively low energy cost. In a context of electrification of chemical processes, APM can help pave the way to a deeper understanding of the physical laws involved in electrochemistry, as well as in chemistry in general.

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HC-ThP-4 Adsorption and Hydrogenation of 1,3-Butadiene on Cu (111) and a Pd/Cu (111) Single-Atom-Alloy, Mohammad Rahat Hossain, M. Trenary, University of Illinois - Chicago

A single atom alloy (SAA) is made by substituting catalytically active dopant metal atoms into the topmost atomic layer of a relatively inert host metal. Commonly, Pd and Pt are used for hydrogenation reactions due to their virtually zero barrier for H₂ activation. Yet, these catalysts are easily coked by CO and their high activity often limits their selectivity. Industrially, these catalysts are often doped with a less active metal to prevent coking and enhance their selectivity. One of the most important steps in the refinement of alkene streams for the industrial-scale production of high-quality polymers is the selective hydrogenation of 1,3-butadiene. In the literature, Pd (111) shows higher selective control over hydrogenation of 1,3-butadiene to butene than Pt (111). Therefore, the presence of isolated

Pd atoms on a Cu host surface can be a suitable SAA catalyst model for this particular reaction. In this study, we used reflection absorption infrared spectroscopy (RAIRS) and temperature programmed desorption (TPD) to investigate the adsorption of butadiene and butene, as well as the hydrogenation of butadiene on Cu (111) and a Pd/Cu (111) SAA. The TPD study shows butadiene binds strongly to the Cu (111) surface compared to 1-butene. The desorption peak in the 120-130 K temperature range indicates the production of 1-butene from the hydrogenation of butadiene on the Pd/Cu (111) SAA. Auger electron spectroscopy (AES) shows no accumulation of surface carbon demonstrating molecular desorption of butadiene from the surface under UHV. The observation of both in-plane (CH_2 -wag at 908 cm^{-1}) and out-of-plane (C-H bend at 1023 cm^{-1}) modes with RAIRS at monolayer coverage indicates that butadiene is adsorbed neither parallel nor perpendicular to the surface. In addition, IR peaks at 1464 cm^{-1} for ($-\text{CH}_3$) antisymmetric deformation and 1412 cm^{-1} for ($=\text{CH}_2$) scissors modes are observed on the Pd/Cu (111) SAA, indicating 1-butene formation from butadiene hydrogenation. The ambient pressure RAIRS studies are currently underway to calculate the turnover frequency (TOF) of the above-mentioned catalyst for the hydrogenation of 1,3-butadiene.

HC-ThP-5 Studying C-H Activation on RhCu Single-Atom Alloys Using Molecular Beams, Molly Powers, J. Rosenstein, L. Joseph, A. Utz, Tufts University

Single-atom alloys (SAAs) are a new class of heterogeneous catalysts drawing immense research interest due to their unique catalytic activity. An active dopant metal is atomically dispersed in a more inert, selective host metal to produce a catalyst that has a unique potential to simultaneously improve catalytic selectivity and activity compared to their single metal counterparts. To date, detailed surface science studies of SAAs have been limited to relatively low-barrier reactions. The poster will summarize recent work in our lab that extends the range of detailed mechanistic studies to higher barrier reactions, including C-H activation. Our study focuses on the reaction of simple hydrocarbons, such as methane, on a RhCu SAA catalyst. While prior work has examined this system using reflection-absorption IR spectroscopy, temperature programmed desorption, and scanning tunneling microscopy, our supersonic molecular beam experiments permit the study of high energetic barriers to examine the dynamics, kinetics, and energetics of the initial C-H bond cleavage reaction, and the subsequent chemistry and diffusion of the surface-bound products of dissociative chemisorption.

HC-ThP-6 Investigating the Dissociative Chemisorption of Methane on Ru(0001) via Supersonic Molecular Beam, Matthew Kalan, Y. Li, A. Utz, Tufts University

Recent theoretical studies have proposed the creation of a database of experimentally determined barrier heights for model reactions on transition metal surfaces as a tool for the development of chemically accurate computational methods for heterogeneously catalyzed reactions¹. This approach has been effectively applied to the dissociative chemisorption of CH_4 on Pt and Ni surfaces using semi-empirical specific reaction parameter density functional theory (SRP-DFT)². Current work on SRP-DFT involves expanding its application to other surfaces, including Ru(0001). There is, however, substantial disagreement in the current literature as to the height of the barrier on Ru(0001), with experimentally determined values ranging from 0.38 to $0.85^{3,4}\text{ eV}$. Our work applies supersonic molecular beam reactivity measurements to this system in order to provide an important experimental benchmark for additional SRP-DFT studies. By measuring reactivity across a wide range of translational energies, surface temperatures, and vibrational states, our data will also serve as a rigorous test of the theoretical predictions of any future first-principles methods. We will present the results from ongoing reactivity measurements of the dissociative chemisorption of CH_4 on Ru(0001) and their implications for the dynamics of the reaction.

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HC-ThP-7 Size-Selected Pt Alloy Cluster Catalysts for the Dehydrogenation of Light Alkanes, Autumn Fuchs, M. Malek, S. Anderson, University of Utah

Size-selected Pt_nGe_m clusters supported on alumina have been developed and are being investigated under ultra-high vacuum for the selective dehydrogenation of light alkanes. Surface analysis techniques such as temperature programmed desorption (TPD), x-ray photoelectron spectroscopy (XPS), and ion scattering spectroscopy (ISS), are used to characterize the clusters and probe binding sites and energies of small alkanes. TPD experiments have shown that the addition of Ge to Pt_n limits catalyst deactivation by carbon deposition (coking) and sintering. The number of Pt atoms in the cluster affects the number of available binding sites for ethylene on the surface. We will present a study of butane and isobutane dehydrogenation and cracking $\text{Pt}_n/\text{alumina}$ and $\text{Pt}_n/\text{Ge}_m/\text{alumina}$. In addition to the surface science experiments, we will present data from a MEMS microreactor device that allows reactants to be flowed over a sample of deposited clusters at pressures up to 1 atm and temperatures to 1000 K. Reactions on clusters will be compared to reactions on Pt nanoparticles made via chloroplatinate drop-casting and reduction.

HC-ThP-8 Switching between Hot Electron and Hot Hole Transfer during Chemical Reaction, Hyekyung Kwon, Korea National University of Education, Republic of Korea; B. Jeon, J. Park, Korea Advanced Institute of Science and Technology (KAIST), Republic of Korea; S. Lee, Korea National University of Education, Republic of Korea

Understanding hot carrier dynamics is crucial for the rapid development of nanocatalysts [1]. By observing the transfer of carriers quantitatively in real-time during a catalytic reaction, a correlation between catalytic performance and charge transfer can be identified [2]. When a chemical reaction occurs on a metal catalyst, electronic excitation is caused by the non-adiabatic dissipation of exothermic energy, leading to the generation of electron-hole pairs (*i.e.*, hot carriers) [3]. In order to study the surface dynamics of these hot carriers, a metal/semiconductor Schottky (*i.e.*, catalytic nanodiode) needs to be coupled with heterogeneous catalysts [4].

In this work, we observed hot carriers excited in real time during the H_2O_2 decomposition reaction using Pt/Si Schottky nanodiodes. When the Pt/Si nanodiode was immersed in the H_2O_2 solution, the H_2O_2 decomposition occurred on the Pt catalyst surface, and electron-hole pairs were excited. This could be detected as a current signal (*i.e.*, chemicurrent; a current induced by a chemical reaction) flowing through the nanodevice. Interestingly, the flow of hot electrons was detected in the Pt/n-Si nanodiode, whereas the transfer of hot holes was observed in the Pt/p-Si nanodiode. The chemicurrent caused by hot carrier transfer linearly depended on the concentration of the H_2O_2 solution, confirming that the observed chemicurrent was indeed a result of the H_2O_2 decomposition occurring on the Pt catalyst. In order to electronically control the hot electron and hot hole transfers, we detected the currents while applying a reverse bias (*i.e.*, an electric field on the Schottky junction) to both the Pt/n-Si and Pt/p-Si nanodevices. In the Pt/n-Si nanodiode, through which hot electrons flowed, electron transfer was amplified under the reverse bias condition due to Schottky barrier lowering. However, when reverse bias was applied to the Pt/p-Si nanodevice, where hot hole transfer was dominant under open circuit, hot electron transfer surprisingly emerged instead of hot hole transfer. Accordingly, we can conclude that the movement of the hot hole in the Pt/p-Si nanodiode was due to the remaining holes after the electrons were consumed for inducing the chemical reaction. Our techniques to control and switch the transfer of hot carriers during a chemical reaction using metal-semiconductor Schottky junctions may shed light on potential applications of hot carriers in catalytic devices, energy conversion-based devices, or sensors.

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Thursday Evening, November 9, 2023

HC-ThP-9 Tracking Elementary Steps in Conversion of Carboxylic Acids on Single Crystalline and Nanofaceted TiO₂(101), Xingyu Wang, Pacific Northwest National Laboratory; *W. Debenedetti,* Los Alamos National Laboratory; *C. O'Connor,* Harvard University; *Z. Dohnalek, G. Kimmel,* Pacific Northwest National Laboratory

In catalysis research, the material gap is one of several that the surface science community is trying to bridge, along with the pressure and temperature gaps. In the specific case of carboxylic acids on TiO₂, previous studies discovered that for acetic acid reacting with anatase nanoparticles at ambient pressure, an environment very much like real world catalytic processes, acetone could be produced. However, under ultrahigh vacuum (UHV) conditions, with a few monolayers of acetic acid on anatase(101) single crystals, acetone was not observed as a product. In contrast, formic acid reactions with either nanoparticles or single crystals showed no evidence of C-C bond formation. Here, we prepared a sample with layers of synthesized anatase nanoparticles with mostly (101) facets in an UHV system and compared the reactivity of acetic acid with that on an anatase(101) single crystal, using TPD (temperature programmed desorption) and RAIRS (reflection absorption infrared spectroscopy) to track the elementary steps, with the goal to bridge this material gap and fully understand the reaction mechanisms of carboxylic acids on metal oxides.

HC-ThP-10 ZnO Nanoparticles as an Effective Rhodamine B Dye Mineralization Under Direct Sunlight Irradiation, Jose Alberto Alvarado Garcia, BENEMERITA UNIVERSIDAD AUTONOMA DE PUEBLA, Mexico; *G. ANAYA GONZALEZ,* Universidad Autónoma de Mexico; *R. PEREZ CUAPIO, H. JUAREZ SANTIESTEBAN,* BENEMERITA UNIVERSIDAD AUTONOMA DE PUEBLA, Mexico; *A. ARCE PLAZA,* INSTITUTO POLITÉCNICO NACIONAL, Mexico

In this research ZnO at different Zinc acetate concentration from 0.1-0.4 M was synthesized through colloidal synthesis and applied to mineralize Rhodamine B dye under direct sunlight irradiation, showing that this is strongly related to the particle size and defects presence at the particle. This relationship is well correlated to the UV-Vis absorbance spectrum and photoluminescence (PL), showing up to 90 % of photocatalyst efficiency after 100 min for sample obtained at 0.3M, this behavior also is showed for those samples obtained at 0.1, 0.2 and 0.4 molar concentration. The XRD data analysis let the structure and crystallite average size (18- 27 nm) to be determined meanwhile the morphology and composition was obtained from HRSEM and EDS respectively. From the FTIR results the organic dye mineralization evolution was determined. Albeit the defects contribution was determined by PL.

HC-ThP-11 Role of Vacancies and Absorbed Hydrogen Atoms on the Formation of Peroxides and Superoxides on CeO₂ Surfaces, M. Brites Helu, M. Vecchiotti, S. Collins, Instituto de Desarrollo Tecnológico para la Industria Química, Argentina; *M. Calatayud,* Laboratoire de Chimie Théorique, Sorbonne Université, France; *Jorge Anibal Boscoboinik, D. Stacchiola,* Center for Functional Nanomaterials, Brookhaven National Laboratory; *F. Calaza,* Instituto de Desarrollo Tecnológico para la Industria Química, Argentina

It is well known that VOCs being recognized as major responsible for the increase in global air pollution. Catalytic combustion is an efficient technology for the abatement of VOC, which are oxidized over a catalyst at temperatures much lower than those of the thermal process. Specifically, gold supported catalysts on CeO₂ have shown a great performance in the oxidation of CO, methanol, toluene, etc. Besides, it is important to clarify the role of the support in such reaction. Ceria has the key property of high oxygen storage capacity which originates in its ability to rapidly switch from Ce to Ce oxidation states as the environment changes from reducing to oxidizing and vice versa. Its redox behavior is influenced by the substituent lattice groups that could be incorporated during different catalyst pretreatments and could affect the oxidation of VOC. This could be understood as the influence of oxygen vacancies and/or absorbed or coadsorbed H on the activation of oxygen molecules. The latter leads to the formation of superoxide and peroxide molecules on the surface, which could in principle be highly reactive towards oxidation of organic molecules.

In this context, we study, by IR spectroscopy and mass spectrometry, the interaction of O₂ with the modified CeO₂ based material, by creating vacancies following different reduction treatments. The possible role of the vacancies and/or presence of H atoms in the electron transfer from the surface to the oxygen molecule is discussed. Using AP-XPS we are able to prove that the surface/near surface of CeOx presents a charging effect which could be due to extra charge/electrons which then transfer to O₂ to form superoxide and peroxide species sequentially and presenting different

thermal stabilities. Furthermore, the presence of different facets on the surface of the material could change the stability or amount of active species, thus comparison of results from polycrystalline ceria and CeO₂ nanocubes provide information about the structure-activity relationship for the rational design of catalytic materials.

HC-ThP-12 Small Alcohol Reactivity Over TiO₂/Au(111) Inverse Model Catalysts, Ashleigh Baber, James Madison University

Gold-based catalysts have received tremendous attention as supports and nanoparticles for heterogeneous catalysis, in part due to the ability of nanoscale Au to catalyze reactions at low temperatures in oxidative environments. Surface defects are known active sites for low temperature Au chemistry, so a full understanding of the interplay between intermolecular interactions and surface morphology is essential to an advanced understanding of catalytic behavior and efficiency. Our undergraduate research lab uses ultrahigh vacuum temperature programmed desorption (UHV-TPD) to investigate the fundamental interactions between small alcohols on Au(111) and the reactivity of TiO₂/Au(111) inverse model catalysts on small alcohol redox behavior. In a systematic study to better understand the adsorption and intermolecular behavior of small alcohols (C₁-C₄) on Au(111) defect sites, coverage studies of methanol, ethanol, 1-propanol, 1-butanol, 2-butanol, and isobutanol have been conducted on Au(111). These small alcohols molecularly adsorb on the Au(111) surface and high resolution experiments reveal distinct terrace, step edge, and kink adsorption features for each molecule. The desorption energy of small primary alcohols was shown to trend linearly with increasing C₁-C₄ carbon chain length, indicating that the H-bonded molecular packing of 1-butanol resembles that of methanol, ethanol, and 1-propanol, while isobutanol and 2-butanol deviate from the trend. These energy insights are particularly interesting when studying the redox behavior of small alcohols over TiO₂/Au(111). Depending on the surface preparation conditions, Au(111) supported TiO₂ nanoparticles react with small alcohols to form either reduced and oxidized products. The reactivity of the surface for ethanol oxidation was altered by controlling the oxidation state of TiO_x (x<2) and coverage of TiO₂. Low coverages of fully oxidized TiO₂ nanoparticles on Au(111) are active for the selective oxidation of ethanol to form acetaldehyde, but not all small alcohols behave similarly.

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