

Tuesday Afternoon Poster Sessions

Surface Science

Room: Hall 3 - Session SS-TuP

Surface Science Poster Session I and Mort Traum Finalists

SS-TuP1 MORTON S. TRAUM AWARD FINALIST: Morphology and Chemical Reactivity of Bi-Metallic Au-Pd Clusters. *E. Gross**, *M. Asscher*, The Hebrew University of Jerusalem, Israel

Bi-metallic clusters have unique catalytic properties, since one metal can modify the electronic and thus the catalytic properties of the other metal. Here we describe a unique preparation method of bimetallic Pd-Au nano-clusters. The growth mode is based on initial evaporation of metal atoms on top of amorphous solid water adsorbed on SiO₂/Si(100) substrate at 100K under ultra high vacuum (UHV) conditions. The ice buffer layer separates the small metallic seed clusters from the substrate. Subsequent annealing to 300K desorbs the water molecules, resulting in aggregation and growth of nano-clusters in a Buffer Layer Assisted Growth (BLAG) mechanism.

Transmission Electron Microscope (TEM), Energy Dispersive X-ray (EDX) and X-Ray Diffraction (XRD) measurements revealed that by modifying the clusters preparation procedure either segregated or alloyed Pd-Au clusters can be formed. The composition of the different clusters was also verified by optical surface plasmon resonance (SPR) absorption measurements.

Temperature Programmed Reaction (TPR) measurements have shown that adsorption of acetylene on top of Pd-Au alloy clusters has led to efficient conversion to ethylene. Benzene was also formed at an order of magnitude smaller rate. Pd-Au alloy clusters have demonstrated significantly higher reactivity than the monometallic clusters. Increasing the surface defects density by Ar⁺ ion sputtering enhanced the thermal stability and sintering resistance properties of the Pd-Au clusters.

SS-TuP2 MORTON S. TRAUM AWARD FINALIST: Direct Observation of Hydrogen-Bond Exchange within a Single Water Dimer. *T. Kumagai**, *M. Kaizu*, *H. Okuyama*, Kyoto University, Japan, *S. Hatta*, *T. Aruga*, Kyoto University, JST-CREST, Japan, *I. Hamada*, *Y. Morikawa*, Osaka University, Japan

The nature of the hydrogen bond is related to many physical, chemical and biological processes. The structure and dynamics of water dimers, which consist of hydrogen-bond donor and acceptor molecules, have been subjects of extensive research as a prototype of much more complex hydrogen-bonding systems. The water molecules in a free water dimer rearrange the hydrogen bond through quantum tunneling among equivalent structures [1]. Recently, we reported the visualization in real space of hydrogen-bond exchange process governed by quantum tunneling within a single water dimer adsorbed on a metal surface with a low-temperature scanning tunneling microscope (STM) [2].

The experiments were carried out in an ultrahigh vacuum chamber equipped with STM operating at 6 K. The Cu(110) was cleaned by repeated cycles of argon ion sputtering and annealing. The surface was exposed to H₂O or D₂O gases via a tube doser below 20 K. We conducted the experiments at very low coverages, where water molecules exist mainly as isolated monomers and dimers on the surface.

A water dimer is characterized by its bi-stable fluctuating image due to the interchange motion of the hydrogen-bond donor and acceptor molecules. The STM image of the dimer shows dramatic change upon substitution with heavy water. The interchange motion of (D₂O)₂ is much slower than that of (H₂O)₂. The interchange rate was determined to be $(6.0 \pm 0.6) \times 10^{-1}$ s⁻¹ for (H₂O)₂ and 1.0 ± 0.1 s⁻¹ for (D₂O)₂ by monitoring the interchange events in real time. The large isotope effect (~60) suggests that the rate-limiting process involves quantum tunneling. In addition, DTF calculation revealed that the barrier of the interchange on Cu(110) is 0.24 eV. This cannot be overcome via mere thermal process at 6 K, which corroborates that the interchange proceeds through tunneling.

Furthermore, the interchange rate is enhanced upon excitation of the intermolecular mode that correlates with the reaction coordinate. While the interchange motion is intrinsic at low bias voltage, as indicated by negligible tip effect, it becomes tip assisted at voltages above 40 mV. The threshold voltage is determined to be 45 ± 1 (41 ± 1) mV for H₂O (D₂O) dimers. The barrier for the interchange (0.24 eV), however, much larger than the energy transferred from a tunneling electron (45 mV).

Consequently, we propose that the interchange tunneling is assisted by vibrational assisted tunneling process.

[1] R. S. Fellers, C. Leforestier, L. B. Braly, M. G. Brown, and R. J. Saykally, *Science* 284, 945 (1999).

[2] T. Kumagai *et al. Phys. Rev. Lett.* 100, 166101 (2008).

SS-TuP3 MORTON S. TRAUM AWARD FINALIST: A Surface-Chemical Perspective on the Success (and Failure) of Metalorganic Compounds as Precursors for Thin Film Deposition. *J.C.F. Rodriguez-Reyes**, *A.V. Teplyakov*, University of Delaware

Fundamental studies focused on understanding the key surface processes that introduce contaminants into a growing film are essential for achieving a contaminant-free thin film deposition. In the case of the metal alkylamide precursor Ti[N(CH₃)₂]₄, we have obtained experimental evidence of two chemical reactions on a Si(100) surface that explain the uncontrolled incorporation of carbon during deposition, namely a surface attachment through a ligand and a feasible C-H bond scission on the surface. For both reactions, the nitrogen p-orbital in the alkylamide precursor plays a significant role, not only favoring the interaction of a N atom with electron-deficient surface sites, but also weakening anti-periplanar C-H bonds. The realization of the important role that the heteroatom in the ligand plays during surface reactions prompted us to start a comprehensive computational investigation of N-containing and O-containing metalorganic precursors, such as amidinates, guanidinates, alkoxides and acetonates. In addition, we consider metal alkyls and cyclopentadienyl precursors. An effective comparison of the surface reactivity of these compounds and their ability to adsorb and decompose on a surface is achieved by simulating the reactions on a silicon surface cluster model using the same methods. Our results corroborate the model established for the alkylamide precursors, where metalorganic precursors adsorb preferentially through the formation of a N- or O- dative bond to the surface. However, further decomposition mechanisms are less favorable for certain types of precursors, such as amidinates and guanidinates, where the p-orbital of the N atoms is delocalized and therefore does not favor the scission of nearby C-H bonds. Oppositely, alkylamides, alkoxides and acetonates are found to readily decompose on the surface. When heteroatoms are absent (e.g. in the case of cyclopentadienyls) adsorption is a significantly less favorable process and decomposition pathways leading to carbon incorporation are less feasible than for other compounds. This investigation is intended to set a ground work for future investigations devoted to understanding and controlling contamination mechanisms during film deposition.

SS-TuP4 MORTON S. TRAUM AWARD FINALIST: Nanoparticle Growth from Copper Metal-Organic Precursors on H-terminated Silicon Surfaces. *K.A. Perrine**, *A.V. Teplyakov*, University of Delaware

Scaling down of the features needed for microelectronic industry has been redefined with the advances in atomic layer deposition (ALD) methods. At the same time, chemical vapor deposition (CVD) of copper has been used in interconnect devices in the electronics industry for several decades. For a controlled copper deposition, it is desirable to combine the clean ligand removal from the precursor molecules (such as, Cu^I (hexafluoroacetylacetonato) vinyltrimethylsilane or Cu(hfac)VTMS) with the control of the ALD process.

In this work, copper nanoparticles were grown by CVD on hydrogen-terminated silicon surfaces to minimize interfacial contamination and effectively remove the hfac and VTMS ligands for clean Cu deposition. During the deposition process, the amount of copper deposited at room temperature is limited by the amount of hydrogen available for ligand removal. Both H-terminated Si(111) and Si(100) surfaces exhibit the growth of nanoparticles, as observed by AFM, and the amount of copper deposited depends on the amount of surface hydrogen present, showing similarity to ALD growth mechanism. Both AFM and SEM confirm copper nucleation. In addition, the growth kinetics of Cu(hfac)VTMS is compared for monohydrogen-terminated Si(100) and hydrogen-terminated Si(100)-3x1 using *in situ* MIR-FTIR. The growth on OH- and NH- terminated surfaces will be compared to that on H-terminated surfaces. *In situ* FTIR spectroscopic studies indicate surface reaction by analysis of the Si-H region and are compared to *in situ* and *ex situ* XPS studies to confirm metallic copper deposition. Silicon surfaces, Si(111) and Si(100), yield different results and indicate that copper growth starts at defect sites. These results set a foundation for growth of clean copper on reducing solid substrates.

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SS-TuP5 MORTON S. TRAUM AWARD FINALIST: Room Temperature Molecular-Resolution Characterization of Self-Assembled Organic Monolayers on Epitaxial Graphene, Q.H. Wang*, M.C. Hersam, Northwestern University

Graphene has attracted significant attention due to its unique electronic structure, high carrier mobilities, and quantum relativistic phenomena. Epitaxial graphene grown on SiC(0001) is a promising material for next-generation technology because it allows for wafer-scale processing. In order to realize its full potential for a diverse range of devices, the bare graphene sheet must be incorporated with other materials via chemical functionalization schemes. Recent progress reported in the literature include the demonstration of atomic layer deposition of Al₂O₃ on mechanically exfoliated graphene sheets functionalized by carboxylate-terminated perylene derivatives,¹ and the formation of monolayers of 3,4,9,10-perylene-tetracarboxylic dianhydride (PTCDA) at cryogenic temperatures on epitaxial graphene.² In this study, we report the room-temperature formation of self-assembled monolayers of PTCDA on epitaxial graphene.³ We characterize the molecular ordering and electronic properties of these monolayers using ultrahigh vacuum (UHV) scanning tunneling microscopy (STM). The molecules self-assemble into stable, well-ordered monolayers that are arranged in a herringbone phase with extended domains spanning hundreds of nanometers. At submonolayer coverage, PTCDA forms stable, isolated molecular islands. The molecular ordering is unperturbed by either defects in the epitaxial graphene or atomic steps in the underlying SiC surface. Scanning tunneling spectroscopy (STS) performed on the PTCDA monolayers reveals strong features in the electronic density of states that are distinct from the pristine graphene regions. The demonstration of robust, uniform organic functionalization of epitaxial graphene presents opportunities for exploring self-assembly chemistry on graphene, tailoring the chemical functionality of graphene, and templated growth and deposition of other materials as potential routes toward realizing graphene-based molecular electronic and sensing devices.

¹ X.R. Wang, S.M. Tabakman, and H.J. Dai, *J. Am. Chem. Soc.* **130**, 8152-8153 (2008).

² P. Lauffer, K.V. Emtsev, R. Graupner, T. Seyller, and L. Ley, *Phys. Status Solidi B*, **245**, 2064-2067 (2008).

³ Q.H. Wang and M.C. Hersam, *Nature Chemistry*, in press (2009).

SS-TuP6 MORTON S. TRAUM AWARD FINALIST: Size-Dependent Catalytic Properties of Pt Nanoparticles Synthesized by Reverse Micelle Encapsulation: Oxidation, Reduction, and Reactivity, J. Croy*, L.K. Ono, S. Mostafa, H. Heinrich, B. Roldan Cuenya, University of Central Florida

The oxidation of Pt and the reactivity of Pt oxides are of fundamental importance in catalysis, and although much research has been conducted on the interaction of oxygen with Pt surfaces, most of the work is confined to UHV with bulk materials such as Pt(111). In this work we will explore the size-dependent oxidation and reduction properties of Pt nanoparticles (NPs) synthesized by reverse micelle encapsulation. We use in-situ O₂-plasma treatments in conjunction with e-beam heating and X-ray photoelectron spectroscopy (XPS) to monitor the temperature-dependent reduction of Pt^{δ+} species in NPs with distinct size distributions. In addition temperature programmed desorption (TPD) was conducted to monitor the oxygen phases developed on our NP samples. These results are compared with the same experiments conducted on a Pt(111) crystal. In parallel to UHV studies, the activity of pre-oxidized Pt NPs, supported on nanocrystalline ZrO₂, was tested for methanol (MeOH) decomposition and oxidation reactions at atmospheric pressure using a packed-bed reactor coupled to a quadrupole mass spectrometer (QMS). Transmission electron microscopy (TEM) was conducted for morphological information and XPS was used to characterize samples both before and after interaction with MeOH.

SS-TuP7 A Novel Spectrometer for Wide Angle and High Pressure Photoemission, A. Thissen, S. Mähl, T. Kampen, O. Schaff, SPECS GmbH, Germany

New scientific opportunities in key research fields like catalysis, research on liquid and aqueous samples, and living matter demand a combination of in-situ x-ray photoemission experiments (XPS) with high-pressure environments of up to 10 mbar.

To address these exciting new challenges we have developed a novel photoemission spectrometer that enables angle resolved photoelectron spectroscopy (XPS) at pressures up to 10 mbar with an angular acceptance of +/- 22°. This new set-up not only dramatically increases the spectrometer transmission but also allows non-destructive depth profiling exploiting the angular dependence of the XPS signal.

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The instrument consists of a differentially pumped PHOIBOS 150 hemispherical analyzer with a pre-lens. This newly developed pre-lens increases the acceptance angle of the PHOIBOS analyzer by a factor of 5. At the same time it allows extremely efficient differential pumping of the gas atmosphere above the sample.

SS-TuP8 In Situ High Pressure X-ray Photoelectron Spectroscopy Studies of the Adsorption of CO and O₂ on Au Nanoparticles Grown on TiO₂(110) Surfaces, P. Jiang, S. Porsgaard, F. Borondics, M. Kober, Z. Liu, M. Salmeron, H. Bluhm, Lawrence Berkeley National Laboratory

Gold nanoparticles supported on TiO₂ surfaces are effective catalysts for CO oxidation. Despite numerous studies on this system, the fundamental mechanism is still controversial, partly due to the challenging pressure gap. Using our special in situ high pressure X-ray photoelectron spectroscopy (HP-XPS), we systematically investigated the adsorption of CO and O₂ on Au nanoparticles grown on TiO₂(110) surfaces with different stoichiometries under real reaction conditions. We found that the synergism between Au and TiO₂ surfaces plays the key role for the whole reaction process.

SS-TuP10 DFT Study of Oxygen Vacancy Formation in a Diesel Oxidation Catalyst: Pt/CeO₂(111), T.Q. Nguyen, M.C.S. Escanho, S. Kunikata, H. Nakanishi, H. Kasai, Osaka University, Japan, H. Maekawa, K. Osumi, Y. Tashiro, Isuzu Advanced Engineering Center, Ltd., Japan

Air pollution from automobile exhaust is one of the major environmental problems in modern civilization. In order to reduce the amount of noxious pollutants, the diesel oxidation catalyst (DOC) systems are widely used. In the DOC system, CO and Hydrocarbon (HC) are oxidized to harmless chemical substances such as H₂O, CO₂, and NO is converted to NO₂.¹⁻⁴ DOCs have a honeycomb-like, monolithic structure. The monolith support is made either from metallic (stainless steel) or ceramic material and coated with a so-called "washcoat". The washcoat consists of high porous oxides, such as γ -Al₂O₃ (alumina), and precious metals, such as Pt, Pd, and Rh plus more additives (e.g., CeO₂ and ZrO₂) to increase the catalytic activity or to stabilize the structure of the catalyst. In DOCs, Cerium is present in high quantities in the form of CeO₂. Cerium renders multiple functions, the most vital of which is to store and to release oxygen under fuel-lean and fuel-rich conditions. This is so-called the oxygen storage capacity (OSC), which allows the catalyst to operate over wider air to fuel ratio.⁵

In this work, we investigate the adsorption of Pt atom on unreduced and reduced CeO₂(111) surface by using first-principles method. With the aim of having a better understanding of the catalytic properties of Pt/CeO₂ for further development and improvement of DOCs, we concentrated on the details of Pt adsorption on CeO₂, the formation of oxygen vacancies at Pt/CeO₂ surfaces and how it affects the geometric and electronic structures as well as chemical properties of surface. We found that, some metal induced gap states appeared under the adsorption of Pt, which is due to the strong interaction between Pt 5d and O 2p. This is the factor that makes the Pt/CeO₂ system more active than the clean CeO₂ surface. In addition, the adsorption of Pt accelerates the formation of surface vacancies and enhances the OSC. A more detailed discussion of these results will be conducted in the meeting.

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- ⁵ H. C. Yao, Y. F. Yu. Yao, *J. Catal.* **86** (1984) 254

SS-TuP11 Surface Electronic Structures and Chemical States of O₂ on Pt₃M [111], (M = Fe,Co,Ni), Y.-S. Kim, Lawrence Berkeley National Laboratory and Hanyang University, Korea, **B.S. Mun,** Hanyang University, Korea and Lawrence Berkeley National Laboratory, **V. Stamenkovic, N. Markovic,** Argonne National Laboratory, **A. Bostwick, E. Rotenberg, P.N. Ross,** Lawrence Berkeley National Laboratory

With x-ray photoemission spectroscopy (XPS), surface electronic structures and chemical states of O₂ on Pt₃M [111] single crystals are investigated at various surface temperature, T = 30~150K. At T=30K, both physisorbed and chemisorbed oxygen molecules are present on the surfaces. As the surface temperature is increased to 150K, molecular oxygen on the surface undergoes a phase transition from weakly bounded physisorbed states to chemisorbed states, i.e. an atomic oxygen states. From the measurements of Fermi surface with angle-resolved XPS, Pt [111] surface shows a clear signature of charge transfer along M direction upon the adsorption of oxygen molecules. However, all other Pt₃M surfaces exhibit little changes

near Fermi surface at various temperatures. The correlation between the electrochemical reactivity and surface electronic structures on Pt_3M will be presented.

SS-TuP13 Improvement of Metal Oxide Catalyst Reactivity by Modification of Surface Fermi Level. *M.C. Kratzer*, University of Illinois at Urbana-Champaign, *F. Nasim*, *A.S. Bhatti*, Comsats Institute of Information Technology, Pakistan, *E.G. Seebauer*, University of Illinois at Urbana-Champaign

There is good reason to believe that the properties of semiconducting metal oxide catalysts can be improved when designed according to the principles of microelectronic devices. Since metal oxide semiconductors support space charge, it is possible for surface electronic properties to couple to bulk electronic properties. Consequently, electronic "band engineering" can be employed to optimize surface reactivity and affect either thermal catalysis or photocatalysis. For instance, hydroxyl group acidity on the TiO_2 surface can be tuned via the electron richness of the semiconductor, which can be manipulated via controlled doping. Alternatively, the direction and magnitude of the near-surface electric field within the space charge region can be adjusted by bulk doping which, in turn, affects the flow of photogenerated charge carriers toward the surface in photocatalysis. The present work describes the applicability of photoreflectance (PR), a type of modulation spectroscopy, to understanding semiconductor surface-bulk coupling in the context of catalysis using TiO_2 as an example metal oxide. The approach involves the synthesis of a thin film of the semiconductor on a silicon substrate by chemical vapor deposition or atomic layer deposition. N- and p-type dopants are introduced into TiO_2 during deposition to produce samples of varying doping levels. The physical and chemical properties of the thin films are characterized using ellipsometry, x-ray diffraction, and x-ray photoelectron spectroscopy. Detailed electrical characterization employing a Schottky diode test structure is undertaken to obtain a precise estimate of carrier concentration. PR is then utilized to better understand the effect of film thickness and uniformity, crystal structure, and doping on the position of the surface Fermi level. The results shed light on the properties of the TiO_2 surface as it relates to thermal catalysis and photocatalysis, and enable unprecedented precision in the tailoring of metal oxide catalysts to ensure optimal surface reactivity.

SS-TuP18 Transition of the Molecule Orientation during Adsorption of Terephthalic Acid on Rutile TiO_2 . *A. Nefedov*, Ruhr University of Bochum, Germany, *P. Rahe*, *M. Nimrich*, University of Osnabruck, Germany, *M. Naboka*, Ruhr University of Bochum, Germany, *A. Kuehnle*, University of Osnabruck, Germany, *Ch. Woell*, University of Bochum, Germany

Chemically functionalized surfaces play an important role in many nanotechnological applications. However, only few attempts have been made so far to create a chemically functionalized metal oxide surface by adsorption of organic molecules, even though metal oxide surfaces provide anisotropy and specific adsorption sites that lead to well-ordered monolayers. Terephthalic acid (TPA) is a versatile molecular linker and has, consequently, received considerable attention with regard to the assembly of three-dimensional self-assembling porous frameworks stabilized by metal-carboxylate bonds, as well as in two-dimensional supramolecular architectures at surfaces. In particular, TPA/ TiO_2 is a promising system to create an overlayer of upright oriented molecules exposing an organic surface terminated by carboxyl groups.

The coverage-dependent adsorption of terephthalic acid on rutile TiO_2 was investigated by means of non-contact atom force microscopy (NC-AFM) and near-edge x-ray absorption fine structure (NEXAFS) spectroscopy [1, 2] under ultra-high vacuum conditions at room temperature. Experiments were performed in two separate systems, one for performing the NC-AFM measurements and the second UHV system using synchrotron radiation for the NEXAFS experiments, which were carried out at the HE-SGM beamline of BESSY II.

Individual molecules are observed to adsorb in a flat-lying geometry at low coverages up to ~ 0.3 monolayer (ML). The molecules are immobile at room temperature, implying a diffusion barrier of larger than 0.8 eV. This rather high value might be explained by an anchoring to surface defect sites. A transition from flat-lying to upright-oriented molecules is revealed by NEXAFS when saturation coverage is achieved. High resolution NC-AFM images reveal two different structures at coverages between ~ 0.8 ML and 1 ML: (i) a well-ordered (2×1) structure and (ii) a mixed structure of molecular rows oriented along the [001] crystallographic direction. The latter structure might originate from a pairwise interaction of two neighboring molecules through the top carboxyl groups. Further increase of the exposure results in a saturation of the corresponding signal in the NEXAFS spectra revealing that the growth of TPA on TiO_2 at room temperature is self-limiting.

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SS-TuP19 Effect of Rapid Thermal Annealing on the Electrical and Optical Properties of Reactively Sputtered Ag_2O Thin Films. *C.C. Tseng*, National Chung Hsing University, Taiwan, *J.H. Hsieh*, Ming Chi University of Technology, Taiwan, *W. Wu*, National Chung Hsing University, Taiwan

Silver oxide films were deposited on glass substrates by magnetron sputtering of a silver target in various Ar-O₂ reactive mixtures with deposited temperature. After deposition, some of these films were annealed using a rapid thermal annealing (RTA) system, with the variation of temperature. A UV-VIS-NIR photometer and a Hall measurement system were used to characterize the optical and electrical properties of these films. The films show a systematic change from Ag_2O (hexagonal) phase to $\text{Ag}+\text{Ag}_2\text{O}$ (cubic) phases at various annealing temperatures. When the annealing temperature is higher than 300°C, the Ag_2O (hexagonal) phase transformed to $\text{Ag}+\text{Ag}_2\text{O}$ (cubic) composite phases. Accordingly, the band gap of these films will change, along with the optical and electrical properties.

Keywords: silver oxide, UV-VIS, rapid thermal annealing, optical property, electrical property.

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