# Wednesday Afternoon, October 24, 2018

### Surface Science Division Room 203C - Session SS+AS+EM-WeA

#### **Semiconducting Surfaces**

**Moderators:** Melissa Hines, Cornell University, Ludo Juurlink, Leiden University

# 2:20pm SS+AS+EM-WeA-1 Functionalizing Semiconductor Surfaces and Interfaces, Stacey Bent, Stanford University INVITED

Semiconductor surface and interface science serves as the foundation for applications ranging from microelectronics to optoelectronics to biosensing. Given the importance of semiconductor surface chemistry in important technologies of today, understanding the fundamental chemistry at a molecular level is key to future advances. This talk will describe studies on the adsorption of organic molecules at semiconductor surfaces, aimed at the ultimate goal of controlling the chemical and electrical properties of these hybrid systems. The presentation will examine model systems of molecular adsorption on the Ge(100)-2×1 surface using a combination of experimental and theoretical methods. The reactivity of different functional groups will be described, with particular focus on reactions of bi- and trifunctional molecules. The results help elucidate the way in which the molecular structure as well as the identity of the reactive moieties affect the product distribution of the molecules upon adsorption. Monolayer surface chemistry is also extended to the formation of covalently-bound organic multilayers through the process of molecular layer deposition (MLD). Results will be presented of our studies to understand MLD mechanisms and bonding. We show that chain termination events during MLD can be counteracted by absorption of precursors into the MLD film, which reintroduces reactive sites that lead to continuation of film growth. We will also describe new MLD chemistries, including photo-initiated MLD that forms new carbon-carbon bonds at the surface.

#### 3:00pm SS+AS+EM-WeA-3 Atomic Structure of UHV-prepared GaP(111)A Surface and its Reactivity Towards Simple Molecules, *Denis Potapenko*, *X Yang*, *B Koel*, Princeton University

Effective pyridine-catalyzed photo-reduction of CO<sub>2</sub> to methanol has been demonstrated on p-type GaP(111)A photocathode. Despite the considerable effort there is still no consensus about either the molecular mechanisms of this process or the role of electrode's surface. As a part of larger effort aimed at elucidation of the mechanisms of CO<sub>2</sub> photoreduction we have conducted a UHV study of the atomic-scale structure and reactivity of the single-crystal GaP(111)A surface. Atomicresolution STM images supported by LEED data show that ion sputtered and annealed surface exhibit both 2 x 2 Ga-vacancy reconstruction and relatively disordered P-trimer formations. The stoichiometric excess of Ga leads to formation of large droplets on the surface. TPD experiments and IR spectroscopy with water and methanol demonstrate dissociative adsorption of these molecules that leads to recombinative desorption around 300 K.

3:20pm SS+AS+EM-WeA-4 Stabilization Mechanism of the Se- or S-treated GaAs(111)B Surface, Shunji Goto, The University of Electro-Communications (UEC-Tokyo), Japan; A Ohtake, National Institute for Materials Science (NIMS), Japan; J Nakamura, The University of Electro-Communications (UEC-Tokyo), Japan

GaAs surfaces are stabilized by surface treatments with S or Se because of the reduction of the dangling bond density [1,2]. Recently, it has been reported that when the GaAs(111)B-(2 x 2) As trimer surface is treated with Se, the diffraction pattern changes from (2 x 2) to (1 x 1) [3]. The Se-treated GaAs(111)B-(1 x 1) surface was considered to have a simple structure; all As atoms of the ideal (111)B surface are replaced by Se atoms. However, a scanning tunneling microscope (STM) image shows highly disordered array of bright features with a density of 0.25 per (2 x 2) unit [4,5]. This is incompatible with the simple structure model for (1 x 1). Very recently, another structure model has been proposed for the Se-treated surface, where three As atoms on the topmost surface are replaced by Se atoms per (2 x 2) unit. This As / Se terminated model is electronically stable because the so-called electron counting rule is satisfied and the STM simulations for this model reproduces the most of features of STM experiments. In this study, we discuss the structural stability of the As / Se termination model from the viewpoint of the formation energy. We depict a phase diagram as functions of chemical potentials of Se ( $\mu$ (Se)) and As ( $\mu$ (As)) using the firstprinciples calculations within the density functional theory.

Under the As-rich and Se-poor conditions, the  $(2 \times 2)$  As-trimer surface is the most stable phase. The As / Se terminated surface appears in the phase diagram under more As (Se) poor (rich) conditions, being consistent with the recent experiment [5]. It is noted that the fully-Se-terminated  $(1 \times 1)$ surface can exist as a stable phase in the limit of Se-rich condition, whereas this surface does not satisfy the electron counting rule and has a metallic

surface state attributed to surplus electrons of Se. At the fully-Seterminated surface, a lone pair forms at each Se site and resultant excess electrons occupy the anti-bonding orbital of the outermost bilayer. As a result, the surface Se atoms hardly leave any dangling bond. We have also found that the S-treated surface is passivated by a similar mechanism.

[1] J. Fan, H. Oigawa and Y. Nannichi, Jpn. J. Appl. Phys. 27 L2125 (1988).

[2] V. N. Bessolov and M. V. Lebedev, Fiz. Tekh. Poluprovodn 46, 10201 (1998).

[3] D. A. Woolf, Z. Sobiesierski, D. I. Westwood, and R. H. Williams, J. Appl. Phys. **71**, 4908 (1992)

[4] A. Ohtake and Y. Sakuma, Cryst. Growth Des. 17, 363 (2017).

[5] A. Ohtake, S. Goto, and J. Nakamura, Sci. Rep., 8, 1220 (2018).

4:20pm SS+AS+EM-WeA-7 Novel Pathways in Reaching Buried Interfaces of Organic/Inorganic Hybrid Systems: A Mechanistic Understanding of Polymer Adsorption on Passivated Metal Oxide Surfaces, *Tom Hauffman*, *S Pletincx, K Marcoen, F Cavezza*, Vrije Universiteit Brussel, Belgium; *L Fockaert, J Mol,* Technical University Delft, Netherlands; *H Terryn*, Vrije Universiteit Brussel, Belgium

The interface between a metal oxide and its coating is of great importance for the durability and efficiency of the entire organic/inorganic hybrid system. However, the analysis of such solid/solid interfaces is challenging, as they are masked by a µm-thick polymer layer on one side and a thick metal oxide matrix on the other side. The main difficulty will be to investigate buried interfaces non-destructively with conventional surface analysis techniques.<sup>1,2</sup>

In this work, we characterize interfacial interactions of several ultrathin polymeric films with aluminum and zinc oxides by utilizing novel methodologies, leading to the characterization of a realistic model interface. The adsorption mechanisms are established by using XPS for chemical state information and ToF-SIMS for direct molecular information of these ultrathin organic overlayers.

The use of these nanometer thin films opens up the possibility of an in situ characterization of environmental influences such as water ingress. The use of Ambient Pressure X-ray Photoelectron Spectroscopy (APXPS) is employed to describe the adsorption mechanism in the presence of water vapor. Furthermore, ATR-FTIR Kretschmann is utilized to obtain a near-interface infrared spectrum while simultaneously, the influence of an above-the-polymer electrolyte (such as water) on the interface is characterized.<sup>3</sup>

The homogeneity of these interfacial interactions is characterized on the nanoscale using nano-infrared spectroscopy. This novel technique allows to conduct infrared spectroscopy on the nanoscale by using an AFM tip, which probes the expansion of the material due to IR absorbance. This results in a local vibrational spectrum.

This work comprises novel synthesis routes and methodologies, which enable to probe interfacial changes in hybrid systems non-destructively and in situ. As such, coatings systems can be optimized with respect to the bare substrate.

 van den Brand, J.; Blajiev, O.; Beentjes, P. C. J.; Terryn, H.; de Wit, J. H. W. Interaction of Anhydride and Carboxylic Acid Compounds with Aluminum Oxide Surfaces Studied Using Infrared Reflection Absorption

Spectroscopy. *Langmuir***2004**,*20*(15), 6308–6317.

(2) Hinder, S. J.; Lowe, C.; Maxted, J. T.; Watts, J. F. A ToF-SIMS Investigation of a Buried Polymer/polymer Interface Exposed by Ultra-Low-Angle Microtomy. *Surf. Interface Anal.***2004**, *36*(12), 1575–1581.

(3) Pletincx, S.; Trotochaud, L.; Fockaert, L.-L.; Mol, J. M. C.; Head, A.; Karslioğlu, O.; Bluhm, H.; Terryn, H.; Hauffman, T. In Situ Characterization of the Initial Effect of Water on Molecular Interactions at the Interface of Organic/Inorganic Hybrid Systems. *Sci. Rep.***2017**, *7*(45123).

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4:40pm SS+AS+EM-WeA-8 Surface Modification of Metal Oxide Surfaces with Gas-Phase Propiolic Acid for Dye Sensitization by Click Reaction, *Chuan He, A Teplyakov, B Abraham, M Konh, Z Li, L Gundlach, S Bai,* University of Delaware; *E Galoppini, H Fan, R Harmer,* Rutgers, the State University of New Jersey

The dye sensitization on metal oxide (ZnO, TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, CuO) surfaces has attracted plenteous attention due to their numerous applications in solar energy conversion, photo-catalysis, sensing, etc. Substantial research efforts focused on designing different dyes and surface nanostructuring. However, the actual chemical attachment schemes still heavily rely on the use of sensitizer molecules functionalized with carboxylic (COOH) or phosphonic (P(O)(OH)<sub>2</sub>) anchor groups. Despite consequential progress in chemical modification schemes, the very nature of these functional groups may cause very pronounced surface morphology change of the nanomaterials designed because of etching process in liquid phase. In addition, these acidic groups often lead to the formation of multiple binding configurations (such as monodentate or bidentate miixtures) on the surface. To address both issues, we developed a two-step preparation procedure to achieve the morphology preservation of metal oxide surfaces and well-defined covalent binding of the intersurface functionality. The first step requires exposing clean metal oxide nanostructures to a gas-phase prop-2-ynoic acid (propiolic acid) in medium or high vacuum, which results in the formation of a robust and stable surface-bound carboxylate with ethynyl groups available for further modification. For the second step, we applied copper catalyzed click reaction on this preserved surface to covalently bind dye molecules functionalized with azido groups to these pre-functionalized surfaces. For example, a suitable organic dye (9-(4azidophenyl)-2,5-di-tert-butylperylene (DTBPe-Ph-N<sub>3</sub>)) was used to investigate a distinct signature of covalently-bound dye molecules on ZnO nanorods surface. In this study, we have confirmed the morphology preservation and selective covalent attachment of the carboxylic acid onto the metal oxide surface by the combination of measurements including scanning electron microscopy (SEM), infrared, X-ray absorption photoelectron spectroscopy (XPS), solid-state 13C NMR, transient absorption spectroscopy (TA) and density functional theory (DFT) calculations.

5:00pm SS+AS+EM-WeA-9 Solar Energy Storage in the Norbornadienequadricyclane System: From Surface Science to In-situ Photochemistry and photospectroelectrochemistry, *M Schwarz, F Waidhas, C Schuschke,* Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany; *S Mohr,* Friedrich-Alexander-Universität Erlangen-Nürnberg; *O Brummel, T Döpper, C Weiss, K Civale,* Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany; *M Jevric,* Chalmers University of Technology, Gothenburg, Sweden; *J Bachmann,* Friedrich-Alexander-Universität Erlangen-Nürnberg. Germany, Germany; *A Görling, A Hirsch,* Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany; *K Moth-Poulsen,* Chalmers University of Technology, Gothenburg, Sweden; Jörg Libuda, Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany

Photochemical isomerization of small organic molecules enables chemical energy storage via a single-photon-single-molecule process. A prototypical example is the conversion of norbornadiene (NBD) to its strained metastable valence isomer quadricyclane (QC), which releases up to 100 kJ/mol upon cycloreversion. This makes the NBD-QC system a solar fuel with an energy density comparable to state-of-the-art batteries.

In order to obtain a better understanding of NBD-based energy storage systems, we studied both the photochemical conversion and the catalytically and electrochemically triggered back-conversion at atomically defined interfaces. We combined vibrational spectroscopy with in-situ photochemistry in ultrahigh vacuum (UHV) and in electrochemical (EC) environments. In UHV, NBD and QC films were grown by physical vapor deposition (PVD) on Pt(111). By infrared refection absorption spectroscopy (IRAS), we observe spontaneous cycloreversion in QC monolayers even at 130 K, while QC multilayers are stable. Adsorbed NBD adopts a  $\eta_2:\eta_1$  geometry which involves an agostic C-H-Pt interaction. At 300 K, this species undergoes dehydrogenation by splitting off the agostic H.

In UHV environments, the conversion from NBD to QC can be triggered insitu by UV light and a co-deposited photosensitizer (PS). In electrochemical environments, back-conversion can be triggered by the electrode. On Pt(111) electrodes, we monitored the back-conversion in-situ by electrochemical infrared reflection absorption spectroscopy (EC-IRRAS). The corresponding spectra were analyzed based on simulated spectra from density functional theory (DFT). Using a new photoelectrochemical IRRAS setup (PEC-IRRAS), we monitored the complete storage and release cycle by in-situ vibrational spectroscopy. Selectivities were determined both for the conversion and the back-conversion, showing that the stability of the PS is a critical step in the storage cycle. Much higher reversibility could be obtained using new NBD derivatives which avoid an additional PS.

To better control the energy transfer via the electrode interface, we investigated NBD derivatives anchored via a carboxylic-acid function to atomically defined oxide surfaces. In UHV, dense monolayers of surfaceanchored NBD derivatives were prepared by PVD onto  $Co_3O_4(111)$  films on Ir(100). The molecules adopt an upright-standing orientation with the NBD units pointing away from the interface. Finally, we explored the behavior of these films in (photo)electrochemical environments.

[1] O. Brummel et al., ChemSusChem 9, 1424 (2016).

[2] U. Bauer at al., Chem. Eur. J. 23, 1613 (2017).

[3] O. Brummel et al., J. Phys. Chem. Lett., 8, 2819 (2017).

5:20pm SS+AS+EM-WeA-10 In-situ Characterization of Photon induced Chemistries in Organotin Clusters with Ambient Pressure XPS, J. Trey Diulus<sup>1</sup>, R Frederick, Oregon State University; M Li, Rutgers, the State University of New Jersey; D Hutchison, I Lyubinetsky, L Árnadóttir, M Olsen, Oregon State University; E Garfunkel, Rutgers, the State University of New Jersey; M Nyman, Oregon State University; H Ogasawara, SLAC National Accelerator Laboratory; G Herman, Oregon State University Solution-based organometallic nanoclusters are unique nanoscale material precursors due to the precise control over their size, shape, and structure. The interaction of photons with these clusters can result in the preferential removal of organic ligands at room temperature. This process leads to a change in solubility, which makes these materials prime candidates for next-generation photoresists for extreme ultraviolet (EUV) nanolithography. In this study, we are investigating the interaction of soft X-ray radiation with organotin based clusters to better understand the transition from organometallic to metal oxide nanoclusters. As a model system we are studying the charge neutral, sodium templated, butyl-tin Keggin (β-NaSn<sub>13</sub>) cluster [NaO<sub>4</sub>(BuSn)<sub>12</sub>(OH)<sub>3</sub>(O)<sub>9</sub>(OCH<sub>3</sub>)<sub>12</sub>(Sn(H<sub>2</sub>O)<sub>2</sub>)]. For analysis, the β-NaSn<sub>13</sub> clusters were dissolved in 2-heptanone and spincoated to form uniform thin films (~20 nm thick). Ambient pressure X-ray photoelectron spectroscopy (AP-XPS) was used to measure the Sn 3d, O 1s, and C 1s core-levels, using photon energies above and below key absorption edges. These measurements allowed us to characterize changes in the both the atomic concentrations and chemical states of  $\beta$ -NaSn<sub>13</sub> under a range of ambient conditions, including ultrahigh vacuum and multiple partial pressures (between 0.01 to 1 Torr) of oxygen, water, methanol, nitrogen, and hydrogen. Successive C 1s scans were collected during exposure to measure the change in intensity with respect to exposure time, ambient, and photon energy. A significant reduction in the C 1s signal was observed for oxygen ambients, suggesting the oxygen partial pressure was able to increase the rate of butyl group desorption from the B-NaSn13 clusters. Furthermore, a photon dose array was collected in each condition to determine whether ambient conditions have an effect on the photon induced chemistries leading to a solubility transition. The dose array was plotted as contrast curves, which indicate that an oxygen ambient significantly lowers the required photon dose relative to UHV. We also found that a water ambient significantly increases the required photon dose compared to an oxygen or UHV ambient. These studies ultimately improve the understanding of photon induced chemistries in the  $\beta$ -NaSn<sub>13</sub> based thin films and provide mechanistic insights for the formation of tin oxide nanomaterials at surfaces.

#### 5:40pm SS+AS+EM-WeA-11 Integrated Photonics Driven Electron Emission from LaB<sub>6</sub> Nanoparticles, Fatemeh Rezaeifar, R Kapadia, University of Southern California

In this report, we demonstrate the novel approach of integrated photonics waveguide driven electron emission from Lanthanum hexaboride (LaB<sub>6</sub>) nanoparticles drop-casted over the surface of an optical waveguide. We use integrated waveguide under LaB<sub>6</sub> electron emitter as a mean to transport photons and evanescently couple them to emitter. This evanescent coupling occurs through longer interaction length and photons can be absorbed efficiently compared to free space laser illumination from top on a metallic emitter. Furthermore, nanoparticles with the average diameter of 4 nm are at the order of electron mean free path and electron emission occurs with fewer scattering compared to lectron emission from conventional bulky metallic emitters. As such, in our proposed device, the higher optical absorption along with fewer scattering inside nanoparticles enable us with larger quantum efficiency electron emitters beyond the available photon driven emitters.

<sup>1</sup> Morton S. Traum Award Finalist

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We experimentally studied the photon driven electron emission characteristics obtained by coupling of CW laser (wavelength = 405 nm) to LaB<sub>6</sub> nanoparticle emitters drop-casted on top surface of the silicon nitride (  $Si_3N_4$ ) integrated waveguide with height of 5  $\mu$ m and width of 50  $\mu$ m. Optical V-groove was fabricated for coupling 405 nm laser from 200 µm optical fiber in to Si<sub>3</sub>N<sub>4</sub> waveguide. The work function of the LaB<sub>6</sub> is only 2.69 eV and it requires very small E-field to collect the photon driven emitted electrons. We observed photocurrent at E-field as small as 0.3  $V/\mu m$  while the dark current detection from LaB<sub>6</sub> nanoparticles begins at higher E-field, 3.5 V/ $\mu$ m. In addition, we present the photocurrent dependency on the laser power. At small E-field in which no tunneling is possible, we observed a linear relation between the photocurrent and laser power indicating single photon ability for this emission process. This matches to the expected slope of 1 for single photon absorption process, given that the work function of LaB<sub>6</sub> is only 2.69 eV, and our photon energy at 405 nm is 3.1 eV. Finally, we present the comparative results for the emission properties from LaB<sub>6</sub> nanoparticles illuminated with free space laser to evaluate the performance of the proposed integrated photonics driven emitters. While free space illuminated method results in emission current as small as 1 pA with 390 mW of laser power, the integrated photonics driven emitter emits above 50 pA with much smaller laser power transporting inside the optical waveguide, 100  $\mu$ W. As such, we conclude integrated photonics driven emitter as a potential solution for increasing the quantum efficiency of the future electron emitters.

# 6:00pm SS+AS+EM-WeA-12 Photon Stimulated Desorption and Diffusion of CO on TiO<sub>2</sub>(110), Nikolay Petrik, R Mu, A Dahal, Z Wang, Z Dohnalek, I Lyubinetsky, G Kimmel, Pacific Northwest National Laboratory

Thermal diffusion of CO adsorbed on rutile TiO<sub>2</sub>(110) was studied in the 20 - 110 K range using photon-stimulated desorption (PSD), temperature programmed desorption (TPD) and scanning tunneling microscopy. During UV irradiation, CO desorbs from certain photoactive sites (e.g. oxygen vacancies). This phenomenon was exploited to study CO thermal diffusion in three steps: first empty these sites during a first irradiation cycle, then replenish them with CO during annealing, and finally probe the active site occupancy in the second PSD cycle. The PSD and TPD experiments show that the CO diffusion rate correlates with the CO adsorption energy stronger binding corresponds to slower diffusion. Increasing the CO coverage or hydroxylation of the surface decreases the CO binding and increases the CO diffusion rate. Relative to the reduced surface, the CO adsorption energy increases and the diffusion decreases on the oxidized surface. The CO diffusion kinetics can be modeled satisfactorily as an Arrhenius process with a "normal" prefactor (i.e.  $v = 10^{12} s^{-1}$ ) and a Gaussian distribution of activation energies where the peak of the distribution is ~0.28 eV and the full width at half maximum (FWHM) is ~0.1 eV at the lowest coverages. The observations are consistent with a significant electrostatic component of the CO binding energy on the TiO<sub>2</sub>(110) surface which is affected by changes in the surface dipole and dipole-dipole interactions.

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