Tuesday Evening Poster Sessions, October 31, 2017

Surface Science Division
Room Central Hall - Session SS-TuP

Surface Science Poster Session

SS-TuP-1 Self-assembly of Organic Thin Films on Metal Surfaces, *David Wisman,* Indiana University, Department of Chemistry and NSWC Crane*; C Tempas, T Morris,* Indiana University*; S Kim, D Lee,* Seoul National University*; S Tait,* Indiana University Department of Chemistry

Organic semiconductors have the potential to replace silicon in some electronic devices because they require less stringent production environments and can offer new functionalities, such as flexible devices. One major drawback of organic semiconductor films is their inefficient charge transport. In traditional organic film materials, the molecules only lay flat in the first few molecular layers before transitioning to less favorable geometries for charge transport. Previous work published in our group has shown that the tris(N-phenyltriazole) (TPT) molecule on a Ag(111) surface shows planar stacking through more than 20 molecular layers due to the π-π donor-acceptor intermolecular contacts between the electron-deficient tris(triazole) core and electron-rich peripheral phenyl units. Scanning tunneling microscopy (STM) studies of this molecule show that it does not change its packing structure through the >20 molecular layers studied. Here, we present investigations of derivative molecules of TPT which have different electron distributions and structures to investigate their effect on stacking. We compare the monolayer packing of these derivative molecules to that of TPT by molecular-resolution STM analysis and then investigate multilayer stacking properties of these molecules. Comparison conductivity measurements for TPT, and the two derivative molecules are compared with pentacene using four-point conductivity measurements. These studies may lead to new organic semiconductor material designs that have well-controlled structure and increased charge transport, making them more competitive with traditional silicon devices.

SS-TuP-2 Periodic Modulation of Graphene by a 2D-FeO/Ir(111) Moiré Interlayer, *Yujing Ma,* M Batzill, University of South Florida

Ultrathin films of iron oxide form a two-dimensional (2D) FeO layer on Ir(111). Due to difference in lattice constant between 2D-FeO and Ir(111) a moiré superstructure is formed. We studied the 2D-FeO/Ir(111) structure by soft X-ray photoelectron spectroscopy(XPS), X-ray photoemission diffraction(XPD), and low energy electron diffraction(LEED), and scanning tunneling microscopy(STM). Moreover we show that this 2D-FeO layer may also be grown by iron intercalation and subsequent oxidation underneath a graphene layer on Ir(111). Thus the graphene can be decoupled from the metal by the 2D-FeO layer. Changes in the graphene C 1s binding energy can be attributed to shifts in the Fermi level of graphene as a consequence of interface band alignment for weak interactions between graphene and the substrate. A shift of C 1s to lower binding energy, for graphene supported on FeO/Ir(111), is a consequence of the dipole moment in the 2D-FeO layer normal to the Ir(111) surface. Broadening of the C 1s peak is consistent with a locally varying 2D-FeO dipole within the moiré structure and thus implies a modulated charge doping of the graphene.

SS-TuP-3 CO Oxidation on Single and Multiple Layer PdO(101) Structures Grown on Pd(100), *Vikram Mehar,* C Wu, University of Florida, Gainesville*; M Shipilin, E Lundgren,* Lund University, Sweden*; H Gronbeck,* Chalmers University of Technology, Sweden*; A Ashtagiri,* The Ohio State University*; J Weaver,* University of Florida, Gainesville

Palladium has been extensively used to promote the catalytic combustion of methane and CO oxidation in automotive exhausts. It has been observed that formation of metal oxide layers can significantly alter the surface reactivity due to differences in chemical properties of oxide and metal surface. Understanding the reactivity of oxides formed under different growth conditions could facilitate the design of catalysts attuned for specific applications. In this presentation, I will discuss results of our studies of Pd(100) oxidation by atomic oxygen as well as the adsorption and oxidation of CO on single-layer and multilayer PdO structures. We find that a well-ordered PdO(101) film of about 7 ML thickness develops on Pd(100) during oxidation with O-atoms at 500 K, whereas a thicker PdO structure (~14 ML) with an apparently more rough morphology grows at temperatures above 600 K. LEED shows that the multilayer PdO(101) structure adopts the same epitaxial relationship with the Pd(100) substrate as the so-called √5 surface oxide, which has been previously shown to correspond to a single layer of PdO(101).

Although the surface structures are nominally identical, temperature-programmed reaction spectroscopy (TPRS) and reflection-absorption infrared spectroscopy (RAIRS) measurements show that the single layer and multilayer PdO(101) structures exhibit distinct reactivity toward CO. TPRS shows that the multilayer PdO(101) structure is more reactive toward CO compared with the single layer PdO(101). Also, RAIRS shows that CO binds only in an atop configuration on the coordinatively-unsaturated (cus) Pd atoms of the multilayer PdO(101) structure, whereas CO initially populates bridging Pd sites on the single PdO (101) layer before also occupying atop Pd sites at higher CO coverages. I will also present results of density functional theory calculations that clarify the origins the different chemical reactivity of the single and multiple layer PdO(101) structures toward CO.

SS-TuP-4 Evaluation of Dynamic Wettability on 2D Inverse Opal Structure, *Naoya Yoshida,* T Genma, K Fukasawa, T Okura, Kogakuin University, Japan

Static wettability of smooth solid surface is determined by balance of surface energy of each solid-liquid, liquid-gas, and solid-gas interface, and surface roughness. However, dynamic wettability is thought to be involved by various factors, *e. g.*, the non-uniformity of composition and surface structure. We fabricated inorganic oxide surface with 2D inverse opal structure and homogeneity of the composition and surface structure, and evaluated their static and dynamic wettabilities.

At first, uniformed 2D colloidal particle film was fabricated on a glass substrate in the similar manner to LB method. SiO_2 thin films with 2D inverse opal structure were fabricated by using a precursor solution (tetraethoxysilane in ethanol and 2-ethoxyethanol) by spin coating and heating at 500°C. The prepared samples were stored in a constant temperature and humidity chamber. These samples were characterized by scanning electron microscope (SEM), optical microscope, and contact angle meter.

SEM images of fabricated samples are shown in figure 1. Film thickness and structure were different depending on the concentration of the precursor solution. Wettability seemed to depend on the film structure, especially in terms of dynamic wetting process; on several samples, thin water film was obviously observed at the outside of a droplet edge. Furthermore, pinning of a water droplet by large defects was also observed.

We fabricated SiO_2 thin films with various 2D inverse opal structures, and their static and dynamic wettabilities were evaluated. In this presentation, details of them and wetting processes will be discussed.

SS-TuP-5 Direct Attachment and *In Situ* Metalation of 29,31-H Phthalocyanine on Chlorine-terminated Si(111) Surface, *Chuan He,* A Teplyakov, University of Delaware

The attachment of phthalocyanine molecules to solid surfaces has attracted substantial attention due to the unique physical and chemical properties of the resulting interfaces, which can be further tuned by designing chemical schemes to tether the phthalocyanine moieties by strong covalent bonds. Furthermore, the properties of these interfaces can be tuned and controlled even further by varying the coordinated metalation, thus providing a wide variety of platforms for many applications, including catalysis and sensing. In this work, an efficient wet chemistry method is developed to achieve the covalent attachment of phthalocyanine molecules directly to a chlorine terminated Si(111) surface without any additional linkers or functional groups. The possibility of cobalt metalation of this modified surface is also tested. X-ray photoelectron spectroscopy (XPS), time-of-flight secondary ion mass spectrometry (TOF-SIMS), and atomic-force microscopy (AFM) are utilized to study the phthalocyanine attachment and metalation processes. The theoretical predictions of core-level N 1s binding energies are investigated by density functional theory (DFT) calculations to compare with experimental results obtained by XPS and to infer the possible mechanisms of surface reactions.

SS-TuP-6 Structural Growth and Oxidation of TbO$_x$ Thin Films on Pt(111), *Christopher Lee,* V Mehar, University of Florida*; S Keil, V Zielasek, M Bäumer,* University of Bremen, Germany*; J Weaver,* University of Florida

Within the family of rare earth oxides (REOs), the terbium oxides exhibit favorable properties in selective oxidation catalysis due to the flexibility in the storage and release of oxygen within the lattice, specifically through structural rearrangement into well-ordered intermediates between the Tb_2O_3 and TbO_2 stoichiometries. We investigated the growth and structures of TbO_x films grown on Pt(111) in ultra-high vacuum (UHV) as well as the oxidation of the films by plasma-generated gaseous atomic oxygen. LEED and STM show that the deposition produces crystalline Tb_2O_3 films that adopt an oxygen deficient cubic fluorite structure where the film conforms

to the hexagonal registry of the Pt(111) substrate. This is characterized by initial surface wetting up to 2 ML of Tb_2O_3 followed by 3D Stranski-Krastanov island growth at higher coverages.

We also find that the terbia film undergoes isomeric reorganization into the longer-order bixbyite Tb_2O_3 conformation when subject to a combination of atomic oxygen exposure along with subsequent annealing at 1000 K. LEED and TPD show that coexisting, ordered intermediates between Tb_2O_3 and TbO_2 may then be created by further oxidizing the bixbyite Tb_2O_3 film via atomic oxygen beam exposure. In particular, two distinct O_2 desorption peaks in TPD spectra provide evidence of the sequential phase stabilization of Tb_7O_{12} (ι-phase) and $Tb_{11}O_{20}$ (δ-phase) along with lower temperature peaks corresponding with more weakly bound surface oxygen. The rapid reorganization of oxygen and oxygen vacancies within this intermediate regime is promising in that it suggests that specific structural arrangements of the terbia lattice may readily adjust to accommodate dissimilar metal cations into the metallic lattice to stabilize ordered, substitutionally doped films. The future outlook is to characterize structure and promoted surface chemistry of doped terbium oxide films, particularly the changes in oxidation induced by the incorporation of high valence dopants and low valence dopants into the metallic framework of the oxide. The larger implication is that the substitutional doping of terbium oxides can provide fundamental insight into doped oxide catalysis, which can provide the additional degree of tuneability towards increased reactivity or selectivity towards partial oxidation pathways required for an effective oxidative coupling of methane (OCM) catalyst.

SS-TuP-7 Surface Spectroscopy and Thermal Desorption Studies of Sulfur-Doped Tungsten Oxide, *Anthony Babore, J Langford, J Hemminger,* University of California Irvine

Mixed sulfide/oxide films were prepared on W (100) as a model system for sulfur-doped tungsten trioxide which has shown potential for enhanced visible light absorption for photocatalysis. In this study we sought to gain a fundamental understanding of the effects of incorporating sulfur atoms into a tungsten oxide system. This was accomplished using a combination of temperature programmed desorption (TPD), Auger electron spectroscopy (AES) and high-resolution electron energy loss spectroscopy (HREELS). D_2O was used as a probe molecule in the TPD experiments and yielded pronounced effects on the desorption spectra for pure oxide, sulfide and mixed sulfide/oxide films on W (100). In addition coverage dependent desorption energies were calculated and compared. AES spectra displayed the relative amounts of sulfur and oxygen on the surface and HREELS experiments confirmed the presence and thermal stability of tungsten sulfide on the surface.

SS-TuP-8 Preparation and Characterization of Metal-doped Calcium Phosphate, *Yuki Iwai, N Yoshida, T Okura,* Kogakuin University, Japan

Ti-doped Hydroxyapatite (TiHAp) partially substituted Ca^{2+} with Ti^{4+} exhibits excellent photocatalytic decomposition of organic compounds, as TiO_2 shows. Although the mechanisms of phtocatalytic activities of TiHAp are still unclear, Ti-doping and/or defects derived from Ti-doping are thought to be important in photocatalytic reactions. Here, we tried to fabricate beta-tricalcium phosphate (β-TCP), with substitution of Ca sites or PO_4 sites with M or MO_4 (M = Ge, Zr, Nb, Mo, Sn, W), respectively, and to study the effect on crystal structure and photocatalytic activity.

M-doped β-TCPs were prepared by solid-state or coprecipitation methods. By solid-state method, $CaCO_3$ and $CaHPO_4 \cdot 2H_2O$ with GeO_2, Nb_2O_5, or SnO_2 (Ca / (P + M) = 1.50, M / (P + M) = 0.01~0.03) were mixed with an alumina mortar. Samples were obtained after calcination at 1000°C for 10 h. By coprecipitation method, $Ca(NO_3)_2 \cdot 4H_2O$ and H_3PO_4 with $ZrCl_2O \cdot 8H_2O$, $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$, or $(NH_4)_{10}W_{12}O_{41} \cdot 5H_2O$ (Ca + M) / P or Ca / (P + M) = 1.50, M / (Ca or P + M) = 0.01~0.10) were dissolved in ion-exchanged water, and stirred for a while. After that, dil. NH_3 aq. was slowly added to the solution until pH became 9.5. The precipitation was filtered, dried at 100°C, and calcined at 1000°C for 24 h. The obtained powders were characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), and scanning electron microscope (SEM) measurements.

XRD measurements indicated that the samples were mainly β-TCP. In the case of Zr, Mo, and W doping, the obtained samples exhibited β-TCP single phase at low doping concentrations. Against doping of Ge, Nb, and Sn, β-TCP was obtained with other phases such as hydroxyapatite (HAp). In this presentation, structure, adsorption of organic/inorganic compounds, photocatalytic activity will be also discussed.

SS-TuP-9 Multiscale Investigation of Catalytic Activity of Ultra-Thin Molybdenum Nitride for Hydrogendenitrogenation Process, *W Kaden, Asim Khaniya,* University of Central Florida

Hydrogendenitrogenation (HDN), a process of removing organonitrogen compounds from petroleum or coal-derived feedstocks, is one of the hydro treating processes in the oil refinery industry. Carefully executed studies on transition metal nitrides, mainly in molybdenum nitride, show that it can be an excellent potential substitute for hydrotreating catalysis. But till to this date, the majority of applied catalysis research for this system has been conducted over Mo2N. This proposed research is intended to explore the activity of recently developed lamellar MoN sheets for catalytic hydrogendenitrogenation (HDN) associated with larger organics, relevant to oil refinery applications (i.e. pyridine and carbazole conversion).

Delta phase MoN (δ-MoN) is preferable for this process due to the expected weakening of the Mo-N bonds within MoN relative to Mo2N due to higher nitrogen coverage in MoN and its catalytic activity is expected to be enhanced when lamellar sheets of MoN are used due to the higher surface area to volume ratio. In addition to δ-MoN, we will explore and compare the catalytic activity of different forms of molybdenum nitrides.

The lamellar sheet of the δ-MoN will be grown by using method established elsewhere and the conventional surface science techniques, like scanning-tunneling microscopy (STM), atomic force microscopy (AFM), and kelvin probe force microscopy (KPFM) will be used to make the molecularly, atomically and in some cases sub-atomically localized measurements of surface structures.

SS-TuP-11 Infrared Analysis of Competitive Surface Adsorption in Superconformal Chemical Vapor Deposition, *Zhejun Zhang,* University of Illinois at Urbana-Champaign, US*; E Mohimi, T Talukdar, G Girolami, J Abelson,* University of Illinois at Urbana-Champaign

A major challenge in nanoscale device fabrication is to fill a deep via or trench with material without leaving a low density 'seam' along the centerline. We previously demonstrated two variants of low temperature chemical vapor deposition (CVD) that afford *superconformal* coating, in which film growth is faster deep in the feature that near to the opening. This affords a 'V' shaped coating profile, and as deposition proceeds, the apex of the V moves upwards until the feature is completely filled. Our first method, suitable for the growth of metallic films such as CrB_2, involves the use of a highly reactive inhibitor, such as H atoms, in conjunction with the CVD precursor to suppress the growth rate in the upper portion of the feature. Our second method, suitable for dielectric deposition such as MgO, takes advantage of the intrinsic competition between two reactants, such as the precursor and water, on the film growth surface for adsorption sites. In both cases, the kinetic results are successfully modeled using Langmuir-Hinshelwood (LH) adsorption theory in combination with molecular transport of the reactants inside the feature.

In this interpretation, the suppression of film growth rate corresponds to a situation in which a significant fraction of the surface binding sites are occupied (blocked) by a species that does not lead to film growth, either the inhibitor in the first case or an excess of one reactant in the second case. However, we have not had any direct proof of this assumed mechanism. To obtain direct insight, we have implemented reflection FTIR spectroscopy in order to measure the surface coverage in adsorbates in real time during film growth. This is very challenging experimentally, however, the use of a thin dielectric on metal substrate at high angle of incidence provides enough signal enhancement in p-polarization to observe sub-monolayer coverages. In addition, the intrinsic cancellation of the absorption signal in s-polarization means that the contribution of gas phase absorption can be cancelled out by subtraction of the p and s signals.

We report the absorption competition for surface sites for the growth of MgO and HfO_2 by low temperature CVD, and we compare the measurements with the range of values estimated from LH theory in view of the molecular fluxes to the surface and the measured film growth rates. As expected, the data reveal surprises such as a low total surface coverage in adsorbates even under competitive kinetics, indicating that the density of binding sites is significantly smaller than the geometric density of sites for these oxides.

SS-TuP-15 Universal Calibration of Computationally Predicted N 1s Binding Energies for Interpretation of XPS Experimental Measurements, *Jing Zhao*[1], *A Teplyakov,* University of Delaware

An independent approach was obtained to calibrate simulated N 1s core level energies by density functional theory to interpret XPS experimental measurements. In this work, we compare the application of two basis sets, LANL2DZ and 6-311G+(d,p) and B3LYP functional and analyse the correlation factors contributing to theoretical with experimental results. The correlation is first examined with the known nitrogen-containing functional groups on well-characterized and well-understood organic monolayers and then applied to a number of nitrogen-containing chemical systems where the nature of the chemical environment of nitrogen atoms had only been proposed based on a number of analytical techniques. Based on this independent calibration approach, a high degree of correlation is obtained and the basic strategies for comparing computationally predicted and experimentally obtained values are proposed.

SS-TuP-18 Vibrational Spectroscopy of Hydrogen Sulfide Adsorbed on Metallic W (100) and Oxygen Adsorbed W (100), *Joel Langford, A Babore, J Hemminger,* University of California Irvine

High resolution electron energy loss spectroscopy, Auger electron spectroscopy, and temperature-programmed desorption has been used to study hydrogen sulfide adsorbed on two systems; metallic W (100) and oxygen adsorbed W (100). At exposure temperatures of 150 K hydrogen sulfide dissociates on both systems. However, for metallic W (100) and oxygen adsorbed W (100) the dissociation products are different. On metallic W (100) hydrogen sulfide dissociates to form W-SH and W-H. On oxygen adsorbed W (100) hydrogen sulfide dissociates to produce a combination of W-SH, W-OH, and W-H. These species were confirmed with both D_2O and D_2S experiments. When metallic W (100) is annealed SH groups recombine to desorb as molecular hydrogen leaving behind the W-S species. Therefore, adsorption of hydrogen sulfide on W (100) is irreversible. Both the vibrational spectroscopy and thermal desorption lack evidence of molecularly adsorbed H_2S. The desorption peak for molecular H_2S is therefore below the 150 K exposure temperature. On oxygen adsorbed W (100) both water and hydrogen desorption is observed. The intensity ratio of W-OH to W-SH vibrational modes and the water to hydrogen desorption ratio as a function of surface oxygen to tungsten was measured. Both the intensity ratio of W-OH to W-SH vibrational modes and water to hydrogen desorption peak is proportional to the oxygen concentration. A descriptive interpretation of hydrogen sulfide W (100) chemistry will be provided in this talk

SS-TuP-19 Lubricity of Gold Nanocrystals on Graphene Measured using Quartz Crystal Microbalance, *M Lodge,* University of Central Florida*; C Tang,* University of California Merced*; Brandon Blue,* University of Central Florida*; W Hubbard,* University of California at Los Angeles*; A Martini,* University of California Merced*; B Dawson, M Ishigami,* University of Central Florida

Recent simulations [Guerra et al, Nature Materials, 9, 634 (2010)] have predicted that, at high sliding speeds, gold nanocrystals slide with vanishing friction on graphite. Unlike structural or super-lubricity observed previously in nanotribology experiments, the predicted reduction of friction by two orders of magnitude was found to be independent of the commensurability between the two sliding surfaces. As such, the predicted phenomenon, named ballistic nanofriction, is novel and different from any phenomena previously observed in nanotribology.

In this talk, we present experimental evidence of ballistic nanofriction for gold nanocrystals deposited onto graphene-coated quartz crystal microbalances (QCMs) under UHV. Lubricity exceeding ballistic nanofriction is observed down to surface velocities of 4.7 cm/sec: much lower than expected from the work of Guerra et al. The observed drag coefficients are $\sim 8.65 \times 10^{-14}$ kg/s. This is significantly smaller than the theoretically-predicted value of 2.0×10^{-13} kg/s, and suggests a much lower interaction strength between graphene and gold nanocrystals than previously assumed in contemporary theoretical models of gold nanocrystals on graphite.

SS-TuP-20 Controllable Synthesis of Ru/Pt Core Shell Nanoparticles with Bi-functional Interfaces towards PROX Reactions, *Yun Lang, J Yang,* Huazhong University of Science and Technology, PR China*; K Cao,* Huazhong University of Science and Technology, PR China, China*; M Gong,* Huazhong University of Science and Technology, PR China*; B Shan, R Chen,* Huazhong University of Science and Technology, PR China, China

Bimetallic nanoparticles (NPs) have attracted great attention due to their unique properties for catalytic applications. Compared with the physical mixture of monometallic NPs or alloyed bimetallic NPs, the formation of core shell structure could further enhance the activity, selectivity and stability. Synthesizing core shell NPs with well controlled shell thickness and composition is of great importance in optimizing their reactivity. Here we report synthesis of Ru/Pt core shell NPs using area-selective ALD techniques. The catalytic performance towards preferential oxidation of CO under excess H_2 (PROX) reaction is studied with different Pt shell thickness. The catalytic activity of hybrid nanostructure can be tuned continuously with shell thickness originated from electron structure modification. Utilizing the lattice mismatch between Pt(111)/Ru(101), Pt(111) layer can be selectively deposited on Ru (001) facet while leaving Ru (101) uncovered. The exposure facets demonstrate two functions in PROX reaction, Pt(111) on Ru(001) decrease CO reaction barrier and Ru (101) increase active oxygen adsorption and dissociation. Both activity and selectivity towards PROX reaction have been enhanced compared with fully covered Ru/Pt core shell nanoparticles or their alloy.

SS-TuP-21 Corrosion Resistance of Yttrium Trifluoride (YF3) and Yttrium Oxyfluoride (YOF) used in Plasma Process Chamber, *Yoshinobu Shiba, A Teramoto, T Goto,* Tohoku university, Japan*; Y Kishi,* Nippon Yttrium Co., Ltd, Japan*; Y Shirai, S Sugawa,* Tohoku university, Japan

Chemically stable materials that have corrosion resistance from reactive species enhanced by plasma have been required for an inner wall of semiconductor process chambers. Although the materials with low formation enthalpy (such as aluminum oxide (Al_2O_3) and yttrium oxide (Y_2O_3)) have been widely used for the protect material, it was revealed that yttrium oxyfluoride (YOF) was much stable than Y_2O_3 against the reactive species. [1] In this research, we compared the chemical stability of Yttrium trifluoride (YF_3) and that of YOF. The enthalpy of formation of YF_3 (-1719 kJ/mol) is close to Y_2O_3's (-1905 kJ/mol), and the enthalpy of formation of one metal-fluorine bond of YF_3 (-573 kJ/mol) is smaller than that of one metal-oxygen bond of Y_2O_3 (318 kJ/mol). YF_3 films and YOF films were exposed to N_2/Ar, H_2/Ar, NH_3/Ar, NF_3/Ar, and O_2/Ar plasmas using microwave-excited surface-wave high-density plasma equipment [2], and investigated their corrosion resistance. After plasma-irradiation, the YF_3 film's surfaces were damaged, and the fluorine content in YF_3 decreased in all condition. In contrast, the YOF's surfaces were not changed by these plasmas. These indicate that the YOF is much stable against the plasma bombardment than the YF_3.

[1] Shiba et al. J. Vac. Sci. Technol. A 35, 021405 (2017)

[2] T. Ohmi, et al. J. Phys. D: Appl. Phys., 39, R1 (2006)

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