

Surface Science

Room 209 CDE W - Session SS-FrM

Surface Science of Reduced Dimensional Materials

Moderators: Moritz Eder, TU Wien, Naihao Chiang, University of Houston

8:15am SS-FrM-1 On-Surface Synthesis of Planar/Nonplanar Porous Graphene Nanoribbons and Nanosheets, Junfa Zhu, University of Science and Technology of China

INVITED

The low-dimensional porous graphene nanomaterials might have intriguing electronic properties and open exciting possibilities in the field of functional materials. By using rationally designed precursor molecules, on-surface synthesis (OSS) approach has emerged as a powerful platform for the synthesis of porous low-dimensional graphene-based nanostructures with atomic precision. In this presentation, we report our recent works on the synthesis of planar/nonplanar porous graphene nanoribbons and nanosheets on different metal surfaces. We have successfully synthesized the one-dimensional planar graphene nanoribbons (GNRs) containing periodic [14]annulene pores on Ag(111) and the two-dimensional non-planar graphene nanosheets containing periodic [30]annulene pores on Au(111), originating from a same precursor [1]. Two distinct reaction pathways on the two surfaces were regulated by different thermodynamic and kinetic mechanisms. In addition, a novel nonplanar porous [32]annulene graphene nanosheet that contains the narrowest periodic nanopores up to date was also successfully synthesized by the dissymmetrical debromination and regioselective coupling reactions of precursor molecules on an Au(111) surface [2]. With the combination of the scanning tunneling microscopy (STM), synchrotron radiation photoemission spectroscopy (SRPES) and density functional theory (DFT) calculations, we identified the reaction products, intermediates precisely, and obtained insights into the reaction mechanism. Moreover, the electronic properties of these porous graphene nanoribbons were also precisely characterized.

References:

[1] Qin T.; Guo D.; Xiong J.; Li X.; Hu L.; Yang W.; Chen Z.; Wu Y.; Ding H.; Hu J.; Xu Q.; Wang T.; Zhu J., Synthesis of a Porous [14]Annulene Graphene Nanoribbon and a Porous [30]Annulene Graphene Nanosheet on Metal Surfaces. *Angew. Chem., Int. Ed.*. 2023, 135: e202306368.

[2] Qin T.; Gao F.; Wu Y.; Liang B.; Hu L.; Yang W.; Ding H.; Hu J.; Xu Q.; Garcia-Lekue A.; Guo D.; Wang T.; Zhu J., Synthesis of Graphene Nanosheets Containing Ultra-Narrow Nonplanar Nanopores on Surfaces. *Submitted*.

8:45am SS-FrM-3 Deuterium Adsorption on N-Doped Carbon Materials, Buddhika Alupotha Gedara, Mi Yeon Byun, Zdenek Dohnalek, Zbynek Novotny, Tom Autrey, Pacific Northwest National Laboratory

Nitrogen-doped carbon materials have been identified as promising candidates for hydrogen activation and storage, however, there is little experimental insight into the nature of the hydrogen interaction in response to the changes in the physical behavior of these materials. In this study, we investigated deuterium adsorption on N-doped highly oriented pyrolytic graphite (N-HOPG) and porous, layered N-doped carbon (NC) materials using x-ray photoelectron spectroscopy (XPS) and scanning tunneling microscopy. N-dopants were introduced to HOPG through low-energy N_2^+ irradiation, while NC was obtained by pyrolysis of glucose and graphitic carbon nitride ($g-C_3N_4$). Nitrogen is embedded in the carbon materials in two predominant configurations: as graphitic N (N substituted in the hexagonal C lattice) and pyridinic N (substitutional N adjacent to a C vacancy). In both N-doped carbon materials the amount of graphitic N exceeds the amount of pyridinic N, at 6.9 and 4.2 atomic percent (at.%), and 4.3 and 2.7 at.%, in N-HOPG and NC respectively. Atomic deuterium (D) was generated by D_2 cracking over a hot tungsten (W) filament. XPS data showed that upon D exposure at 220 K, the pyridinic N peak (398.0 eV) shifted to a higher binding energy by +1.2 eV for HOPG and +1.1 eV for NC, while the graphitic N peak (400.7 eV) remained unchanged, indicating that the D atoms bound solely to pyridinic N. All the pyridinic N on HOPG could be saturated with D atoms, whereas only approximately 30% of the pyridinic N could bind D atoms in NC. This is attributed to the porosity of NC, which prevents atomic D from reaching some pyridinic N located within the pores. Deuterium fully desorbed from HOPG at 773 K, while complete desorption from NC was observed above 873 K. This study demonstrates a strong correlation between D adsorption on freestanding graphene and layered porous carbon materials, providing a comparative analysis of the N-doping effects on the surfaces and interfaces of carbon materials in both

idealized planar model systems (N-HOPG) and high surface area materials such as NC. The authors gratefully acknowledge the support of U.S. Department of Energy, Office of Science, Basic Energy Sciences, Physical Behavior of Materials under Award No. 80110.

9:00am SS-FrM-4 Ion Beam-induced MoS₂ Surface Modification: An XPS Study of Ar⁺ and Ar Gas Cluster Ion Beam Treatments, Francesco Laudani, Markus Sauer, Dmitry Polyushkin, Technische Universität Wien, Austria; Lorenzo Pettorosso, Technische Universität Vienna, Austria; Jakob Rath, Jakob Hemetsberger, Annette Foelske, Technische Universität Wien, Austria

Two-dimensional (2D) materials are a class of crystals structured as sub-nanometer sheets with no dangling bonds on the basal plane. Molybdenum Disulfide MoS₂ is the second most studied 2D material after graphene [1]. The material's electrical and chemical properties can be tuned by creating defects. The most studied type of defect due to its ease of formation is the sulfur vacancy, usually found to be a p-dopant [2] and to increase of chemical reactivity of the surface as the defect presents increased chemical reactivity [3]. One way of creating such vacancies is ion beam exposure. It provides a precise and spatially resolved possibility of creating defects on the material's surface due to preferential sputtering of sulfur [4]. XPS studies of ion beam exposure effects on MoS₂ have previously been featured in several publications which showed molybdenum (IV) to reduce to an oxidation state whose nature is not yet precisely defined [5,6,7]. As the nature of the treated surface is still not fully understood our study aims to gain further understanding through a more in-depth investigation of ion beam treatment. We compare freshly cleaved surfaces of MoS₂ crystals (molybdenite) and CVD-deposited MoS₂ monolayers using Ar⁺ ion beam at 500 eV and 1 keV to induce preferential sputtering of sulfur and follow the evolution of molybdenum's chemical environment by recording the Mo 3d region using XPS. In addition, we investigate the effects of Gas Cluster Ion Beams with clusters Ar⁺₂₅₀₀ at 20 keV energy and Ar⁺₁₀₀₀ at 2.5 keV energy, respectively, to understand how a different energy transfer may affect the surface transformation for monomer compared to gas cluster ion beams [8]. The surface properties are then investigated further by monitoring the reactivity by exposing the sample to ambient conditions and following the re-oxidation process in air. The results allow for a better understanding of the surface modifications and a preliminary model for the effects of Ar ions on MoS₂ is proposed which might contribute to a more precise tailoring of MoS₂ (electronic) properties in the future.

References

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9:15am SS-FrM-5 Model Studies of the Surface Structure and Stability of Metal Nanoparticles on Oxide Surfaces Under Catalytic Conditions, Ravi Ranjan, Francisco Zaera, University of California - Riverside

The surface structure and properties of copper (Cu) and platinum (Pt) nanoparticles (NPs) supported on tantalum oxide films (Cu/TaO_x/Ta and Pt/TaO_x/Ta) have been investigated under ultrahigh vacuum (UHV) and high-pressure conditions using reflection absorption infrared spectroscopy (RAIRS) and temperature-programmed desorption (TPD), together with carbon monoxide (CO) as a probe molecule. After oxidizing the surface of the tantalum disc in situ under UHV, both metals were vapor-deposited onto tantalum at room temperature, and the resulting surface sites were titrated with CO at 77 K for the Cu-deposited surface and 300 K for the Pt-deposited surface. Sequential growth of Cu led to the formation of distinct adsorption sites, including atoms at the metal-oxide interface and on (100) and (111) facets. These Cu NPs remained stable under UHV up to at least 500 K. However, under atmospheric CO pressures, they exhibited reduced thermal stability, remaining intact only between 300 and 450 K. Furthermore, CO adsorption was found to be significantly more exothermic under UHV conditions compared to ambient CO environments, indicating a notable pressure dependence in adsorption energetics.

In contrast, Pt deposition at room temperature resulted in a less dramatic development, manifested by a slight shift in the a-top CO adsorption

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frequency, the only feature observed in the RAIRS data, to higher wavenumbers with increasing deposition time. No bridging CO features were detected, suggesting the formation of relatively small and isolated NPs. Pt NPs were observed to diffuse into the subsurface upon heating above around 600 K, suggesting limited thermal stability on the TaOx support. These studies provide insights into the structural evolution and dynamic behavior of metal NPs on oxide supports under conditions relevant to catalysis.

9:30am **SS-FrM-6 Exploring the Catalytic Potential of Supported MgO Nanostructures for CH₄ Conversion, Arephin Islam¹**, Brookhaven National Laboratory; *Jose Rodriguez*, Brookhaven National Laboratory and State University of New York at Stony Brook

Natural gas, primarily composed of methane, is a versatile energy vector with significant potential for efficient energy utilization. Converting methane into valuable hydrocarbons, such as ethane and ethylene, at low temperatures without deactivation challenges remains a critical objective. MgO nanostructures have emerged as promising candidates for methane activation due to their unique surface properties, while Cu-based catalysts demonstrate potential for selective methane oxidation at reduced temperatures. This study examines the growth and reactivity of MgO nanostructures on Cu₂O/Cu(111) and Au(111) substrate using scanning tunneling microscopy (STM) and synchrotron-based ambient-pressure X-ray photoelectron spectroscopy (AP-XPS). Mg deposition onto the "29" structured copper oxide film promotes oxygen transfer from the Cu₂O/Cu(111) substrate to Mg, forming MgO and CuO_x phases. The resulting structures exhibit diverse morphologies, including embedded MgO nanostructures (1–3 Mg atoms) and randomly dispersed MgO nanoparticles. AP-XPS and STM analyses reveal that MgO nanostructures (0.2–0.5 nm wide, 0.4–0.6 Å high) embedded in Cu₂O/Cu(111) substrates activate methane at room temperature, dissociating it primarily into CH_x (x = 2 or 3) and H adatoms with minimal C adatom formation. At 500 K, these structures facilitate C–C coupling into ethane and ethylene with negligible carbon deposition and no catalyst deactivation, significantly outperforming bulk MgO catalysts, which require temperatures exceeding 700 K. Density functional theory (DFT) calculations support these experimental findings, showing that methane activation is a downhill process on MgO/Cu₂O/Cu(111) surfaces. Methane dissociation is driven by electron transfer from copper to MgO and the presence of under-coordinated Mg and O atoms. The formation of O–CH₃ and O–H bonds lowers the energy barrier for C–H bond cleavage in methane. Furthermore, DFT studies indicate that smaller Mg₂O₂ clusters exhibit stronger binding and lower activation barriers for C–H dissociation, while larger Mg₃O₃ clusters enhance C–C coupling due to weaker *CH₃ binding. To understand the role of Cu, MgO was also deposited on inert Au(111) surface followed by similar XPS and STM experiments. These results highlight the critical role of size in optimizing the catalytic performance of MgO nanostructures for selective methane conversion.

9:45am **SS-FrM-7 Automated Matter Manipulation to Create Artificial Lattice Structures, Ganesh Narasimha²**, *Mykola Telychko, Woon Yang, Arthur Baddorf, An-Ping Li, Rama Vasudevan*, Oak Ridge National Laboratory

The precise arrangement of matter using scanning tunneling microscopy (STM) presents a controlled route for engineering structures that exhibits designer quantum states. Nonetheless, the sensitive nature of STM tip poses significant operational challenges in assembling diverse lattice geometries with tailored functionalities. In this work, we introduce a reinforcement learning (RL) driven experimental framework to construct artificial nanostructures via spatial manipulation carbon monoxide (CO) molecules on a copper substrate. The pipeline integrates deep learning-based image analysis for molecule recognition that is coupled with an RL agent that predicts optimized parameters for molecule manipulation. Initial manipulation strategies are generated through stochastic sampling of tip parameters—bias voltage, current setpoint, and tip speed—which are compiled into action sequences serving as training data for the RL agent. Upon training, the agent is deployed on the STM for construction of artificial structures. The workflow uses additional techniques such as data augmentation, active drift correction, and high-precision controls, thereby facilitating the creation of artificial lattice structures. Acknowledgement: Research was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Scientific User

Facilities Division as part of the QIS Infrastructure Project, "Precision Atomic Assembly for Quantum Information Science" and performed at the Center for Nanophase Materials Sciences (CNMS), which is a US Department of Energy, Office of Science User Facility at Oak Ridge National Laboratory.

(Acknowledgement: This research was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Scientific User Facilities Division as part of the QIS Infrastructure Project, "Precision Atomic Assembly for Quantum Information Science" and performed at the Center for Nanophase Materials Sciences (CNMS), which is a US Department of Energy, Office of Science User Facility at Oak Ridge National Laboratory).

10:00am **SS-FrM-8 Morton Traum Student Award Announcement,**

10:30am **SS-FrM-10 Atomic-Scale Exploration of Low-Dimensional Materials, Nathan Guisinger**, Argonne National Laboratory, USA **INVITED** Low-dimensional materials functioning at the nanoscale are a critical component for a variety of current and future technologies. From the optimization of light harvesting solar technologies to novel electronic and magnetic device architectures, key physical phenomena are occurring at the nanometer and atomic length-scales and predominately at interfaces. This talk will cover research and review capabilities within the Center for Nanoscale Materials (CNM) at Argonne National Laboratory, which is one of the five Department of Energy Nanoscale Research Centers. In this presentation, I will discuss low-dimensional material research occurring in the Nanoscale Synthesis and Characterization (NSC) Group at the (CNM). I will discuss the synthesis and characterization of advanced material platforms, such as graphene and borophene, and a more recent focus on artificial lattices. Specifically, the synthesis of artificial graphene nanoribbons by positioning carbon monoxide molecules on a copper surface to confine its surface state electrons into artificial atoms positioned to emulate the low-energy electronic structure of graphene derivatives. We demonstrate that the dimensionality of artificial graphene can be reduced to one dimension with proper "edge" passivation, with the emergence of an effectively gapped one-dimensional nanoribbon structure. Remarkably, these one-dimensional structures show evidence of topological effects analogous to graphene nanoribbons. Guided by first-principles calculations, we spatially explore robust, zero-dimensional topological states by altering the topological invariants of quasi-one-dimensional artificial graphene nanostructures. The robustness and flexibility of our platform allows us to toggle the topological invariants between trivial and non-trivial on the same nanostructure. Our atomic synthesis gives access to nanoribbon geometries beyond the current reach of synthetic chemistry and thus provides an ideal platform for the design and study of novel topological and quantum states of matter.

11:00am **SS-FrM-12 Using Two-Dimensional Covalent Organic Frameworks to Stabilize Single-Atom Catalysts on Model Surfaces, Yufei Bai**, Indiana University Bloomington; *David Wisman*, NAVSEA Crane; *Steven Tait*, Indiana University Bloomington

Single-atom catalysts (SACs) combine the advantages of homogeneous and heterogeneous catalysts by limiting the reaction sites to isolated single metal atoms with well-defined chemical properties. A metal-ligand coordination method to stabilize SACs has been previously developed by our group, in which 1,10-phenanthroline-5,6-dione (PDO) was used as the ligand to coordinate with metals such as Pt, Fe, and Cr to form stable metal single sites on the gold surface.¹ To further enhance the metal loading per unit surface area while preserving catalyst stability, we explored utilizing the uniform pores of single-layered two-dimensional covalent organic frameworks (2D COFs) for the stabilization of these SACs. Highly stable on-surface 2D COFs with well-defined pore sizes were synthesized on model surfaces under ultra-high vacuum (UHV) or ambient conditions, subsequently serving as templates to host ligand-coordinated Pt SACs. Under UHV conditions, the formation of 2D COF with hexagonal symmetry on the Au(111) surface was achieved by surface-mediated Ullmann-type radical coupling of COF precursor 1,3,5-tris-(4-bromophenyl)benzene (TBB).² Subsequent deposition of PDO and Pt on the COF surface allowed the formation of single-site Pt catalysts via coordination interaction. Surface characterization, including scanning tunneling microscopy (STM) and X-ray photoelectron spectroscopy (XPS), demonstrated the confinement of PDO in the COF pores and the existence of oxidized Pt, indicating its single-atom nature. Under ambient conditions, an imine-linked 2D COF was formed on the highly oriented pyrolytic graphite (HOPG) surface via a solid-vapor interface mechanism, resulting in a high-quality network with long-range order.³ Subsequently, PDO and PDO-coordinated Pt were deposited onto

¹ **JVST Highlighted Talk**

² **JVST Highlighted Talk**

the COF surface, and their surface distribution and interactions with the COF network at the liquid/solid interface were characterized by STM. Those results were compared to the self-assembly behavior of PDO and PDO-coordinated Pt complexes on graphite without 2D COF. STM analysis provides insights into the intermolecular interactions that determine the supramolecular structure and patterning on the surface. In both systems studied, the 2D COF has a significant impact on the distribution of Pt-PDO complexes. These systems which combine COF confinement and metal-ligand coordination strategy to stabilize SACs offer the possibility to achieve higher stability and greater metal loading.

References

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11:15am **SS-FrM-13 Probing the Promotion or Inhibition of TiO₂ Atomic Layer Deposition on Si(111) Surfaces Modified by Basic Amines**, *Tyler Parke, Andrew Teplyakov*, University of Delaware

The use of small molecule inhibitors (SMI) in area selective atomic layer deposition (AS-ALD) processes has gained widespread attention for their capability to confer selectivity onto ALD substrates with near-atomic scale resolution. In TiO₂ AS-ALD processes using hydrogen-terminated silicon (H-Si) and oxidized silicon (HO-Si), some nitrogen-containing compounds have been shown to selectively block titanium precursors from the H-Si non-growth surface and prevent selectivity loss.

In this study, an array of basic amines, such as aniline, parafluoroaniline (pFA), pyridine, and trifluoroethylamine (TFEA) were investigated for their use as inhibitors or promoters of TiO₂ thermal ALD processes on the (111) single-crystal surface of H-Si and HO-Si. While some compounds act as a small molecule inhibitor (SMI) on these surfaces during the TiO₂ ALD processes, blocking precursor adsorption, some, such as para-fluoroaniline (pFA), act as a promoter of TiO₂ deposition, enhancing growth rate well beyond what is expected for the unmodified HO-Si growth surface.

Nucleation patterns during the first few cycles of ALD on amine-modified silicon surfaces were probed by atomic force microscopy (AFM) to determine the cause of inhibition or promotion. To further understand these patterns, surface topography was compared between surfaces reacted with basic amines -in vapor-phase and in solution. The bonding of the amines to each surface was confirmed with X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FT-IR), which were correlated with density functional theory (DFT) simulations to model the resulting surface structures. Understanding the amine-modified surface structures and what drives them to promote or inhibit ALD processes will allow for a greater capability to tune surface selectivity, and thus a wider scope of 3D architectures to be formed and integrated into nanoelectronic devices.

11:30am **SS-FrM-14 Oxides Formed on Multi-Phase Complex Concentrated Alloys: Nanoscale Spectroscopic Imaging with XPEEM and ToF-SIMS**, *Keithen Orson, Samuel Inman*, University of Virginia; *Jerzy Sadowski*, Brookhaven National Laboratory; *Derk Rading, Julia Zakel*, ION-TOF USA, Inc.; *John Scully, Petra Reinke*, University of Virginia

Complex concentrated alloys (CCAs) present an opportunity to design new alloys with tunable mechanical and corrosion properties. The inclusion of second phases for strengthening is desirable, but second phases can be detrimental to localized corrosion resistance even if both phases individually have good corrosion resistance. The chemistry and structure of the corrosion-protectant passive layer is not well understood for these complex alloys. The air-formed native oxide of a two-phase CCA with the composition Al_{0.3}Cr_{0.5}Fe₂Mn_{0.25}Mo_{0.15}Ni_{1.5}Ti_{0.3} is characterized with X-ray photoemission electron microscopy (XPEEM) and time-of-flight secondary ion mass spectroscopy (ToF-SIMS). These hyperspectral imaging techniques combine nanoscale spatial resolution with spectroscopic information and produce >10⁶ spectra. There are sharp differences in oxide character between the FCC and L2₁ phases of this nominally two-phase alloy. Clustering analysis of the XPEEM and ToF-SIMS images reveals that there are at least two additional phases present in the alloy which are identified by their distinct oxide contributions: nanoscale Ti-rich and Al-rich inclusions that comprise <1% of the total alloy, making them difficult to study without these techniques. Minor inclusions may nevertheless have large implications for local breakdown of corrosion resistance. XPEEM and complementary X-ray photoelectron spectroscopy (XPS) show that the primary elements in the passive film are Al, Cr, Fe, and Ti. Mo is present in the 4+, 5+, and 6+ oxidation states, acting as an aliovalent cation. Spectral

features observed in Cr spectra with XPEEM indicate that the Cr chemical environment varies between the passive film formed on the FCC matrix and the L2₁ second phase, and the minor inclusions have distinct passive films than the surrounding alloy. To further understand what oxide species form in the passive film, a controlled oxidation of the clean alloy surface was done in UHV conditions and combined with XPEEM and XPS to understand what oxides form during early oxidation. This controlled oxidation reveals that Cr, Al, and Ti oxidize in the first few monolayers of Oxygen exposure. Mo, Fe, and Mn oxidize to a smaller degree, and Ni does not contribute at all during the first 100 Langmuir of oxygen exposure. The composition and rate of oxide formation also varies based on the underlying phase. Direct observations of the passive film over the different phases provide insight into the local corrosion resistance CCAs at phase boundaries, aiding in the future design of corrosion resistant multi-phase CCAs.

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