Nanoscale Science and Technology
Room 114 - Session NS1-MoM

Water and Ionic Transport at the Nanoscale

Moderator: Mark Hersam, Northwestern University

8:45am **NS1-MoM-3 Phase Separation and Oxygen Diffusion in Resistive Memory*, Yiyang Li,*** University of Michigan **INVITED**

Resistive memory is a promising technology that conducts memory and computation through the migration of oxygen in transition metal oxide. Understanding the mechanisms of oxygen migraiton is critical towards understanding the functionality of resistive memory devices. In this talk, we present our recent research on the materials thermodynamics and kinetics principles that govern ion motion in oxide-based resistive memory. Using a combination of device measurements, materials characterization, and multiscale physical modeling, we find that oxygen vacancies do not obey Fick's First Law of diffusion as conventionally believed, but instead undergo composition phase separation, which enables diffusion against the concentration gradient. This work yields design rules for nonvolatile memory devices based on composition phase separation.

9:15am **NS1-MoM-5 Advanced Aqueous Separations Using Membranes with Tailored 1D and 2D Confinement*, Seth Darling,*** Argonne National Laboratory

Solute transport in confined environments is a subject of profound interest and ongoing exploration, with recent advancements pointing toward transformative possibilities. Traditional hindered transport theory, a longstanding framework for understanding solute movement through cylindrical pores, underscores the significance of convective and diffusive hindrance in impeding solute permeation, thereby limiting sharp solute separations by membranes. However, breakthroughs in membrane technology, particularly the utilization of near-perfect isoporous membranes, offer new avenues to surmount these limitations. By leveraging nanofabricated, defect-free silicon nitride membranes and employing recirculated feed strategies to enhance solute-membrane interactions, we have achieved encouraging solute rejections, effectively barring solutes larger than the pore size while facilitating the passage of smaller solutes. This advancement not only represents a departure from historical constraints but also holds promise for unprecedented membrane separations through meticulous process design and tight pore-size distributions. Concurrently, the integration of two-dimensional (2D) materials into membranes introduces a distinct paradigm for ion transport and separation applications. These membranes exploit interlayer galleries to drive separation and selectivity, with specific transport properties shaped by chemical and structural modifications within the interlayers. A novel approach involving exfoliated and restacked phyllosilicate minerals with molecular crosslinkers allows precise control over interlayer spacing, influencing ion diffusivities in the resulting crosslinked 2D membranes. These membranes, characterized by tunable ion diffusivities, provide a platform for systematic studies of confined ionic transport, offering insights into fundamental mechanisms governing solute movement in nanoconfinement and paving the way for synergistic advancements in membrane-based separation technologies.

9:30am **NS1-MoM-6 Radioactive Tracer Diffusion through TPT-CNMs*, Andre Beyer,*** *N. Khayya, A. Gölzhäuser,* Bielefeld University, Germany

In recent years, Carbon Nanomembranes (CNMs) have emerged as an innovative class of 2D materials known for their exceptional combination of high selectivity and permeation properties, with a particular emphasis on p-[1,1',4',1"]-terphenyl-4-thiol (TPT)-CNM with a thickness of about 1 nm. Such CNMs combine a rapid water permeation with ultrahigh ionic exclusion in aqueous solution [1]. Here, we report permeation measurements of carbon dioxide and water through TPT-CNMs, which were conducted with concentration-gradient-driven transport of radioactive tracer molecules, specifically [3H] H_2O, [14C] $NaHCO_3$, and [32P] H_3PO_4. Our investigation explores the impact of the pH value on the diffusion process. The equilibrium ratio of carbon dioxide and the corresponding anions change with the pH value. Therewith, an independent characterisation of the diffusion of carbon dioxide and its anionic forms appears to be feasible. Considering concentration polarization and outgassing effects, our results align with previously obtained radioactive diffusion data for a neutral pH value of 7 [2]. Interestingly, despite the electrostatic barrier associated with TPT-CNM in neutral aqueous environments, our observations indicate enhanced permeation of anions in the basic range of pH.

[1] Y. Yang et al., ACS Nano 12, 4695 (2018); Y. Yang et al., Adv. Mater. 32, 1907850 (2020).

[2] R. Dalpke, A. Dreyer, R. Korzetz, K. J. Dietz, and A. Beyer, J. Phys. Chem. Lett. 11, 6737 (2020).

9:45am **NS1-MoM-7 Molecular-Resolution Elucidation of Ice Defects Formed by Liquid Water Crystallization*, Jingshan Du,*** Pacific Northwest National Laboratory*; S. Banik,* University of Illinois - Chicago*; H. Chan,* Argonne National Laboratory*; B. Fritsch,* Helmholtz Institute Erlangen-Nürnberg for Renewable Energy, Germany*; Y. Xia,* University of Washington*; A. Hutzler,* Helmholtz Institute Erlangen-Nürnberg for Renewable Energy, Germany*; S. Sankaranarayanan,* University of Illinois - Chicago*; J. De Yoreo,* Pacific Northwest National Laboratory

Despite the ubiquity of ice, defects in ice formed by water crystallization have never been directly observed on the molecular scale. Here, we report the stabilization and Ångstrom-resolution electron imaging of ice I_h crystallized from liquid water. Combining lattice mapping and molecular dynamics simulations, we show that ice is highly tolerant to nanoscale defects such as misoriented subdomains and trapped gas bubbles, which are stabilized by molecular-scale structural motifs. We discovered subdomain-rich regions near the defective crystal edges despite the structure appearing single-crystalline according to diffraction criteria. These subdomains connect via low-angle grain boundaries with flat energy landscapes as a function of tilt angles, showing the high tolerance of ice to defect structures. Furthermore, bubble surfaces adopt low-energy nanofacets and create negligible strain fields in the surrounding crystal. These bubbles can dynamically nucleate, grow, migrate, dissolve, and coalesce under electron irradiation and be monitored in situ near a steady state. This work opens the door to understanding water crystallization behaviors at an unprecedented spatial resolution and provides new research paradigms to the theory, modeling, and forecasting of ice crystallization and melting in environmental, biological, and material systems.

10:00am **NS1-MoM-8 Reduce Liquid Waste and Improve Throughput in CVD and ALD Processing*, Kathleen Erickson,*** MSP - A Division of TSI

Liquid Flow Controllers (LFCs) are routinely used with vaporizers in gas-phase processing to improve process repeatability, precision and adjustability. In the microelectronic industry LFCs are used to vaporize liquid precursors for Chemical Vapor Deposition (CVD) or Atomic Layer Deposition (ALD). Due to high precision requirements and long LFC flow control response/stabilization times (time to reach and maintain ±1% of set-point), liquid or vapor divert schemes are often used in CVD and ALD. In this scenario, the liquid or vapor is diverted to exhaust until the LFC is able to reach and stably maintain the setpoint. Legacy LFCs can have response times on the order of 4-9 seconds. These long stabilization times, result in significant time sending vapor/liquid to the diverter line – meaning more liquid waste. More liquid is consumed, pumps are exposed to more liquid, and remediation systems have a higher load. Increased liquid waste negatively impacts cost of ownership in increased liquid precursor source cost, reduced pump lifetime, and increased maintenance requirements.Additionally, it worsens the environmental impact of these semiconductor processes. As more and more semi processes run short processing times, this long response time is becoming increasing problematic to throughput as well. For long processes, LFC response times have a relatively small impact on throughput; in a 150 second process, a 3 second liquid flow stabilization time only adds 2% to the processing time - still significant, but perhaps not intolerable. However, for short process times, like short pulse CVD or ALD, the stabilization time of the LFC can become a much larger percentage of the processing time.For example, in a 6 second short pulse CVD process, a 3 second response time increases deposition times by 50%.

This presentation will introduce a new Liquid Flow Controller (Turbo LFC) designed specifically for semiconductor processing. The Turbo LFC can reach ±1% of set-point within 0.3s – a significant improvement over conventional LFCs. The fast response time is a result of a high-speed sensor, which can also provide advantages in process control. A fast sensor enables a faster feedback control loop which can result in tighter control. This high-speed LFC also opens the possibility for measuring the delivered mass of vapor pulses (0.05 – 1+s), providing an alternative to ALD valves, which provide no feedback on mass flow rates and are affected by line pressure and temperature. Details on Turbo LFC design and performance data will be reviewed, including the accuracy, repeatability and response time, and the impact of ambient temperature and line pressure on performance.

Nanoscale Science and Technology
Room 114 - Session NS2-MoM

Imaging at the Nanoscale including Focused Ion Beam and Electron Microscopy

Moderator: Alec Talin, Sandia National Lab

10:30am **NS2-MoM-10 3D Reconstructions of Dislocation Networks via Focused Ion Beam Electron Channeling Contrast Imaging***, **Julia Deitz**, A. Polonsky, T. Ruggles, L. Jauregui, A. Allerman,* Sandia National Laboratories **INVITED**

Electron Channeling Contrast Imaging (ECCI) performed in a scanning electron microscope (SEM) serves as a rapid method for structural defect characterization in a wide array of crystalline materials such as metals and semiconductor materials/devices. Largely due to ease associated with sample preparation, much of this characterization has been performed in plan-view. The ability to instead perform ECCI cross-sectionally is advantageous for characterization of defects below 150 nm of the surface, sometimes at buried interfaces. Additionally, cross-sectional ECCI would demonstrate the potential to achieve three-dimensional (3D) analysis of dislocation networks, giving more detail on dislocation-dislocation interactions. With increases in serial sectioning implementations in the focused ion beam (FIB) SEM, automation routines are well positioned to obtain 3D dislocation network reconstructions. In this contribution, we demonstrate a 3D reconstruction of a dislocation network in an AlGaN via ECCI performed in the FIB-SEM and discuss practical challenges to collecting this data.

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11:00am **NS2-MoM-12 Transmission Electron Microscopy Investigation of Carbon Nanotube Growth on Stainless Steel Substrates***, **Joshua Hancock**, R. Vanfleet,* Brigham Young University

Carbon nanotubes (CNT) are a unique nanomaterial with a wide variety of research applications. One of the most common chemical vapor deposition (CVD) methods to grow CNTs is via the thermal decomposition of a hydrocarbon precursor gas by a metallic catalyst. This is usually done using an iron catalyst deposited on a “noble” or nonreactive substrate, such as alumina. In this setup, the foreign iron is treated to form nanoparticles on the substrate that act as the active sites for CNT growth. Stainless steel is a novel substrate because it does not require the addition of a foreign catalyst to grow carbon nanotubes. Instead, the substrate itself can be treated to become catalytic for CNT growth.

We investigate the effects of CNT growth on stainless steel and form a model for how nanotube growth occurs on the substrate. 316 stainless steel chips are first exposed to a flow of air at high temperature to oxidize the surface. The gas flow is then switched to ethylene to reduce the oxide layer and initialize CNT growth. The active temperature and exposure time of these steps are varied between samples to understand how each affects the growth and how the growth process evolves over time.

To better understand the growth mechanisms, we investigate the CNT-substrate interface at the base of the nanotubes. Focused ion beam (FIB) processing was used to create cross-sections of the CNTs and substrate. To protect the original material from ion beam damage and to contrast redeposited material, samples were coated in a thin layer of alumina using atomic layer deposition (ALD) prior to FIB processing. The final cross-sections were analyzed in a scanning transmission electron microscope (STEM) to allow for high resolution imaging and energy dispersive x-ray spectroscopy (EDX).

Results from STEM and EDX analysis have allowed us to form a basic model for CNT growth on stainless steel. CNTs were seen to grow out of iron-rich metallic nanoparticles embedded in the oxide layer. The oxide layer was also seen to lose iron over time, suggesting that these iron nanoparticles were reduced out of the oxide by the hydrocarbon precursor gas. The base particles were seen to sink into the oxide, leaving a hole when CNTs were removed. The diameters of the nanotubes were also seen to grow over time, suggesting that carbon infiltration is actively coating our CNTs. The effects of the sinking particle and infiltration quickly isolate the catalyst, leading to very short CNTs (1-10 microns).

11:15am **NS2-MoM-13 Focused Ion Beam Species Affect Beam Chemistry Applications***, **Gavin Mitchson**,* Thermo Fisher Scientific

Some modern plasma focused ion beam (PFIB) systems offer the capability to switch between multiple ion species. Each ion species (typically xenon, argon, oxygen, or nitrogen) can exhibit unique strengths and weaknesses for specific materials or applications. For example, oxygen often performs exceptionally well for cross sectioning resin-embedded samples and polymeric materials [1-3]. Low energy argon polishing can provide enhanced TEM image quality [4, 5]. However, signification portions of the parameter space remain unexplored and fundamental questions around limits of performance remain unanswered.

Beam chemistry is a critical component for many applications and to-date the effect of ion species in this space remains unexplored. For example, high quality protective caps are critical for most cross-sectioning and TEM lamella preparation processes. These protective caps are typically formed using at least one of a metallic precursor, a non-metallic carbonaceous precursor, or an insulating silicate-based precursor. In other situations, etchant precursors provide either selective material removal, enhanced material removal rates, or improved surface finish quality [6]. Since the initial PFIB system development and release, most workflows have relied on the Xe^+ ion species for beam chemistry processes. Critical knowledge around how other ion species perform with different beam chemistry precursors is missing.

We report the results of an extensive survey of different ion species and beam chemistry precursor combinations. All the ion species are capable of depositing various materials, although there are differences in the density of the deposited material and typical growth rates. Materials deposited using lighter ion species (argon, oxygen, and nitrogen) typically exhibit significant porosity and bubble formation when the ion beam energy exceeds some threshold, typically between 2-5 keV, which can obfuscate the relative yield comparisons (see Figure 1). For xenon, higher beam energies (below about 12 keV) are typically adequate for dense non-porous depositions. We also observed some interesting differences in etch rate as a function of halogenated etchant gas flow rate for argon and xenon ion species when milling silicon and aluminum substrates. For aluminum, the etch rate suppression or enhancement depends on the ion species, beam energy, and etchant precursor (Figure 2). We speculate that the different interaction volume sizes affect the relative kinetics of the possible gas-sample interaction pathways.

11:30am **NS2-MoM-14 Detection Efficiency Enhancement for Deterministic Single Ion Implantation***, **Kristian Stockbridge**,* Ionoptika, Ltd., UK*; D. Cox,* University of Surrey, UK*; G. Aresta,* Ionoptika, Ltd., UK*; R. Webb, S. Clowes, B. Murdin,* University of Surrey, UK

Techniques for deterministic implantation of single ions are currently of high interest for quantum tech-nology applications such as single photon emitters[1,2] and solid-state qubits[3]. Here we present our capabilities for single ion implantation over a range of ion species into different target materials at different implant energies (<100keV) using a liquid metal alloy ion source (LMAIS) Ionoptika Q-ONE single ion implanter.

For some systems their low secondary electron (SE) yield can limit our ability to efficiently detect single ion implantation, and this therefore limits the number of error-free deterministic implants we can ex-pect to achieve. We present on-chip ion beam induced charge (IBIC) detection for 25keV Bi+ and 50keV Bi2+ implantation into a Si device. The detection efficiency using IBIC is increased close to 100%. Alt-hough coincident SE detection was performed, the active substrate suppressed the emission of SEs such that the on-chip detection dominated.

SiO2 appears to be the target which gives consistently the best secondary electron detection efficiency. We therefore also investigate implantation through thin films of atomic layer deposited (ALD) SiO2 to enhance the detection efficiency of targets where on-chip detection may be incompatible.

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[2] K. Groot-Berning et al., ‘Deterministic Single-Ion Implantation of Rare-Earth Ions for Nanometer-Resolution Color-Center Generation’, Phys. Rev. Lett., vol. 123, p. 106802 (2019).

[3] M.T. Mądzik et al. ‘Conditional quantum operation of two exchange-coupled single-donor spin qubits in a MOS-compatible silicon device’, Nat Commun, vol. 12, p. 181 (2021).

Nanoscale Science and Technology
Room 114 - Session NS1+2D+QS-MoA

Functionality in 2D Nanostructures and Devices

Moderators: Nikolai Klimov, NIST, **Andy Mannix**, Stanford University

1:30pm **NS1+2D+QS-MoA-1 Low-Dimensional Neuromorphic Electronic Materials and Applications***, Mark Hersam,* Northwestern University **INVITED**

The exponentially improving performance of digital computers has recently slowed due to the speed and power consumption issues resulting from the von Neumann bottleneck. In contrast, neuromorphic computing aims to circumvent these limitations by spatially co-locating logic and memory in a manner analogous to biological neuronal networks [1]. Beyond reducing power consumption, neuromorphic devices provide efficient architectures for image recognition, machine learning, and artificial intelligence [2]. This talk will explore how low-dimensional nanoelectronic materials enable gate-tunable neuromorphic devices [3]. For example, by utilizing self-aligned, atomically thin heterojunctions, dual-gated Gaussian transistors have been realized, which show tunable anti-ambipolarity for artificial neurons, competitive learning, spiking circuits, and mixed-kernel support vector machines [4,5]. In addition, field-driven defect motion in polycrystalline monolayer MoS$_2$ enables gate-tunable memristive phenomena that serve as the basis of hybrid memristor/transistor devices (i.e., 'memtransistors') that concurrently provide logic and data storage functions [6]. The planar geometry of memtransistors further allows multiple contacts and dual gating that mimic the behavior of biological systems such as heterosynaptic responses [7]. Moreover, control over polycrystalline grain structure enhances the tunability of potentiation and depression, which enables unsupervised continuous learning in spiking neural networks [8]. Finally, the moiré potential in asymmetric twisted bilayer graphene/hexagonal boron nitride heterostructures gives rise to robust electronic ratchet states. The resulting hysteretic, non-volatile injection of charge carriers enables room-temperature operation of moiré synaptic transistors with diverse bio-realistic neuromorphic functionalities and efficient compute-in-memory designs for low-power artificial intelligence and machine learning hardware [9].

[1] V. K. Sangwan, *et al.*, *Matter*, **5**, 4133 (2022).

[2] V. K. Sangwan, *et al.*, *Nature Nanotechnology,* **15**, 517 (2020).

[3] M. E. Beck, *et al.*, *ACS Nano*, **14**, 6498 (2020).

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[6] X. Yan, *et al.*, *Advanced Materials*, **34**, 2108025 (2022).

[7] H.-S. Lee, *et al.*, *Advanced Functional Materials*, **30**, 2003683 (2020).

[8] J. Yuan, *et al.*, *Nano Letters*, **21**, 6432 (2021).

[9] X. Yan, *et al.*, *Nature*, **624**, 551 (2023).

2:00pm **NS1+2D+QS-MoA-3 Defect Manipulation in van der Waals Heterostructures and its Applications***, Son Le,* Laboratory for Physical Sciences*; T. Mai, M. Munoz, A. Hight Walker, C. Richter,* 100 Bureau Dr.*; A. Hanbicki, A. Friedman,* 8050 Greenmead Dr. **INVITED**

Reliable and accurate spatial doping of 2-dimensional (2D) materials is important for future applications using this novel class of materials. Here, we present our work on photo-doping of an h-BN/Graphene/h-BN heterostructure. Natural defect states in bulk h-BN can remotely dope graphene and can be optically activated or deactivated. In this way, we can modify both the carrier density and type in graphene accurately and reversibly by several orders of magnitude. Using a spatially-resolved light source, we can activate photo-dopants in selected areas of the sample, and by laterally modulating the doping, we have created PNP junction (PNPJ) devices. *In-situ* quantum Hall measurements were used to demonstrate the effectiveness of this doping technique and characterize the electrostatic profile of the PNPJ. Doping and undoping the heterostructure in a specific sequence, we were able to introduce and destroy correlation among the dopants. Defect correlation greatly enhances carrier mobility while the destruction of this correlated state significantly degrades the carrier mobility in the graphene, effectively creating a mobility switch. An elegant demonstration of this mobility switch is the observation of spin and valley-resolved Landau levels of the graphene in the quantum Hall regime with high-mobility, dopant correlated states, and spin and valley -degenerate Landau levels in the low-mobility, dopant uncorrelated states. I will discuss ongoing studies to better understanding the nature of these defects with photo-doping measurements of different hBN thicknesses as well as hBN from different sources. This doping technique opens up the possibility to engineer novel device and expand the applications of 2D heterostructures.

2:30pm **NS1+2D+QS-MoA-5 Extraordinary Tunnel Electroresistance in Layer-by-Layer Engineered Van Der Waals Ferroelectric Tunnel Junctions***, Qinqin Wang,* Department of Electrical and Computer Engineering and Quantum Technology Center, University of Maryland, College Park

The ability to engineer potential profiles of multilayered materials is critical for designing high-performance tunneling devices such as ferroelectric tunnel junctions (FTJs). FTJs comprise asymmetric electrodes and a ferroelectric spacer, promising semiconductor platform-compatible logic and memory devices. However, the traditional FTJs consisting of metal/oxide/metal multilayer heterostructures can only exhibit modest tunneling electroresistance (TER, usually $<10^6$), which is fundamentally undermined by the unavoidable defect states and interfacial trap states. Here, we constructed van der Waals (vdW) FTJs by a layered ferroelectric CuInP$_2$S$_6$ (CIPS) and graphene. Owing to the gigantic ferroelectric modulation of the chemical potentials in graphene by as large as ~1 eV, we demonstrated a giant TER of 10^9. While inserting just a monolayer MoS$_2$ between CIPS/graphene, the off state is further suppressed, leading to $>10^{10}$ TER. Our discovery opens a new solid-state paradigm where potential profiles can be unprecedentedly engineered in a layer-by-layer fashion, fundamentally strengthening the ability to manipulate electrons' tunneling behaviors and design advanced tunneling devices.

Keywords: 2d materials, ferroelectric tunnel junctions, tunneling electroresistance

2:45pm **NS1+2D+QS-MoA-6 Scanning Tunneling Microscopy Studies of Twisted Transition Metal Dichalcogenides***, Adina Luican-Mayer,* STEM 150 Louis Pasteur Private, Canada

Material systems, devices, and circuits, based on the manipulation of individual charges, spins, and photons in solid-state platforms are key for quantum technologies. Two-dimensional (2D) materials present an emerging opportunity for the development of novel quantum technologies, while also pushing the boundaries of fundamental understanding of materials. Our laboratory aims to create quantum functionality in 2D systems by combining fabrication and assembly techniques of 2D layers with atomically precise microscopy.

In this talk, I will focus on experimental observations of novel phenomena in moiré structures created by twisting 2D layers using scanning tunnelling microscopy and spectroscopy. I will discuss the demonstration of reversible local response of domain wall networks using scanning tunneling microscopy in ferroelectric interfaces of marginally twisted WS$_2$ bilayers. Moreover, in the case of twisted WS$_2$ bilayers close to 60°, we observe signatures of flat bands and study the influence of atomic relaxation on their band structure.

Nanoscale Science and Technology
Room 114 - Session NS2-MoA

Light-Matter Interactions at the Nanoscale

Moderator: Nikolai Klimov, NIST

4:00pm **NS2-MoA-11 Time-Resolved Photoemission Electron Microscopy Imaging of the Near-Field Dynamics in Silver Nanowires Excited by Few-Cycle Short-Wave Infrared Pulses***, Nelia Zaiats,* Lund University, Sweden

Ultrafast photoemission electron microscopy (PEEM) is a powerful technique for studying the dynamics of surface plasmons and other electronic excitations, allowing space and time coherent imaging of plasmonic phenomena at the sample, irradiated by pulsed light with high temporal and spatial resolution [1].

Short-wave infrared (SWIR) light is used for optical communication as well as in a wide range of sensor applications [2]. Femtosecond SWIR sources can also be used for generating high-order harmonics in the water window [3]. Silver nanowires, which can be synthesized with high crystal perfection [4], present a pronounced plasmonic resonance in the SWIR range, allowing them to act as efficient light concentrators. While detailed interferometric time-resolved photoemission electron microscopy (ITR-PEEM) measurements have been carried out on Ag nanoparticles in the visible and near-infrared range [5,6], few such PEEM studies have been reported in the SWIR range [1].

Here, we investigate the near-field dynamics in Ag nanowires excited by SWIR pulses with a duration of ~17 fs (2.3 optical cycles) and wavelengths in the 1600-2400 nm range using ITR-PEEM. We study the nanowire response for different light polarizations and intensities as well as for different orientations and geometrical shapes of the wires. We excite the dipolar longitudinal plasmon mode of the nanowire and investigate the near-field dynamics on the femtosecond time scale using two SWIR pump-probe pulses separated in time. The study shows the use of ITR-PEEM in the SWIR wavelength regime, revealing the dynamics of a model plasmonic system.

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4:15pm **NS2-MoA-12 AFM-IR of EHD-Printed PbS Quantum Dots: Quantifying Ligand Exchange at the Nanoscale**, *L. Ferraresi, G. Kara,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland; ***Nancy Burnham,*** Worcester Polytechnic Institute; *R. Furrer, D. Dirin, F. La Mattinia, M. Kovalenko, M. Calame, I. Shorubalko,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

Colloidal quantum dots (cQDs) recently emerged as building blocks for semiconductor materials with tuneable properties. Electro-hydrodynamic printing can be used to obtain sub-micrometre patterns of cQDs without elaborate and aggressive photolithography steps. Post-deposition ligand exchange is necessary for the introduction of new functionalities into cQD solids. However, achieving a complete bulk exchange is challenging and conventional infrared spectroscopy lacks the required spatial resolution for its analysis. Infrared nanospectroscopy (AFM-IR) enables quantitative analysis of the evolution of vibrational signals and structural topography on the nanometre scale upon ligand substitution on lead sulphide (PbS) cQDs. A solution of ethane-dithiol in acetonitrile demonstrated rapid (~60 s) and controllable exchange of more than 80% of the ligands, encompassing structures up to ~800 nm in thickness. Prolonged exposures (>1 h) led to the degradation of the microstructures, with a systematic removal of cQDs regulated by surface-to-bulk ratios and solvent interactions. This study establishes a method for the development of devices through a combination of tuneable photoactive materials, additive manufacturing of microstructures, and their quantitative nanometre-scale analysis.

4:30pm **NS2-MoA-13 AVS National Student Awardee Talk: Slow and Fast Timescale Effects of Photoinduced Surface Oxygen Vacancies on the Charge Carrier Dynamics of TiO_2**, ***Bugrahan Guner***[1], *O. Dagdeviren,* École de technologie supérieure, University of Quebec, Canada

The migration of charge carriers (*e.g.*, electrons, holes) in metal oxides, such as TiO_2, plays a vital role in (photo)catalytic applications [1]. Nevertheless, photoinduced surface oxygen vacancies (PI-SOV) scan significantly alter the dynamics of charge carriers [2-4]. Here, we study the effect of PI-SOVs (prompted by high-energy ultraviolet irradiation) on fast (*i.e.*, electrons) and slow (*i.e.*, holes) charge carriers via time-resolved atomic force microscopy (TR-AFM) measurements, while simultaneously exploring the effect of gold nanoparticles (Au-NPs). We conducted our measurements on Au-NP-deposited titanium dioxide, *i.e.*, TiO_2. Our measurements illustrate that the induced oxygen vacancy (V_O) defects result in a decrease in time constants associated with the migration of electrons. In addition, we quantified the effect of induced defectson the migration barrier of slow charge carriers, *i.e.*, holes. Our respective measurements show that PI-SOVs lower the migration barrier of holes for both the TiO_2 and TiO_2/Au-NP interface. We believe that the observed statistical difference is caused by the effect of defects over the recombination and trapping mechanisms of fast and slow charge carriers. Our results express the important effect of V_O on charge migration dynamics, which underlines the need for further studies of defects under realistic conditions.

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Funding information:

This work was supported by the Canada Economic Development Fund, Natural Sciences and Engineering Research Council of Canada, and Le Fonds de Recherche du Québec - Nature et Technologies.

4:45pm **NS2-MoA-14 Improving the Shape and Optical Stabilities of Plasmonic Au Nanobipyramids by Metal Shell Deposition***, **Thomas Egan**, G. Chen,* University of Central Florida

Anisotropic plasmonic metal nanoparticles show significant promise as light-sensitizing components in the design of advanced optical materials. Their plasmon resonances can be tuned across the visible and near-IR regions by adjusting their aspect ratio, allowing them to harvest solar energy well below the bandgaps of conventional semiconductors. However, their processing and applications under environmental conditions often involve elevated temperatures, which may cause restructuring and shape change of the anisotropic plasmonic metal nanoparticles and lead to a shift of their plasmon resonances. Therefore, it is necessary to improve their thermal stability for robust applications. Here, using anisotropic Au nanobipyramids (NBPs) immobilized on a TiO_2 support as a model system, we demonstrate that the deposition of thin shells of different metals (Au@M NBPs, M = Ag, Pd, Pt) can alter and drastically increase their thermal stability. For pure Au NBPs and Au@Ag NBPs, their tips are observed to progressively blunt and their plasmon resonances begin to blue-shift when annealed at 100 ℃ for 1 hour, while the shapes and plasmon resonances of Au@Pd and Au@Pt NBPs are well-maintained up to 200 ℃ and 250 ℃, respectively. Notably, we also find that the thermal stability of Au@Pd and Au@Pt NBPs is unaffected by annealing up to 4 hours, suggesting that this method can be used to protect metal nanoparticles under various operating conditions. We further use energy-dispersive X-ray (EDX) spectroscopy to map the alloying processes of these core@shell NBPs, which reveal that the onset temperature of alloying increases as the lattice mismatch between core and shell metals increases.

5:00pm **NS2-MoA-15 Photonic Chip Packaging for Extreme Environments***, **Nikolai N Klimov**, S. Robinson,* National Institute for Science and Technology (NIST)*; A. Rao,* National Institute for Science and Technology (NIST); University of Maryland*; D. Barker, F. Bateman, G. Holland, D. Westly,* National Institute for Science and Technology (NIST)

Integrated photonic sensors have advanced significantly in the past decade, driven by a combination of the inherent scalability of integrated photonics combined with precision nanofabrication as well as an ever-increasing range of applications. Such integrated sensors offer advantages in size, weight, and power compared to traditional sensor form-factors. While photonic packaging is well established for many applications, robust and ruggedized photonic packaging is indispensable to field-deployable photonic sensors that must operate under extreme environments. Here, we demonstrate a method for photonic packaging that can operate at cryogenic temperatures as well as in high radiation environments. We also assess the feasibility of the bonding method for high-temperature applications. Using low temperature hydroxide catalysis bonding and a custom packaging station to actively align and attach optical fiber v-groove arrays to a silicon photonic chip, we characterize the packaged chip across a wide temperature range from 360 K down to 3.8 K, confirming suitability for cryogenic operation, and observe < 1 dB variation per grating coupler across a 50 nm wavelength range. Moreover, we expose our packaged chip to an electron beam corresponding to an accumulative radiation dose of 1.1 MGray and observe no degradation in insertion loss across 1510 nm to 1630 nm wavelength range. Finally, we bond several dies and confirm the

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[1] **AVS National Student Awardee**

high temperature compatibility of our bonding approach by observing no change in mechanical bond strength before and after annealing at 973 K. Our packaging methodology can be readily adapted to different photonics applications, ranging from cryogenic circuits to deployable extreme-environment sensors.

5:15pm **NS2-MoA-16 Periodic and Quasi-Periodic Plasmonic Architectures for Strong Light-Matter Interaction***, M. Ferrera, V. Aglieri, J. Pelli Cresi,* Istituto Italiano di Tecnologia, Italy*; E. Ghidorsi,* Istituto Italiano di Tecnologia, Dipartimento di Fisica, Università degli Studi di Genova, Italy*;* ***Andrea Toma,*** Istituto Italiano di Tecnologia, Italy

Light-matter interaction pervades our everyday life and typically involves an energy exchange between an external electromagnetic field and a photon emitter (dye molecules, quantum dots, etc.). Usually, this interaction is rather weak and such that only the spontaneous emission rate is modified. However, the engineering of nanohybrid architectures can promote an energy exchange rate between light and matter faster than any relaxation process. Under these conditions, the system can enter the strong-coupling regime and new hybrid light-matter states, called polaritons, are formed, with potential interest in various fields spanning from photocatalysis to optoelectronic and quantum technologies [1].

For the realization of functional polaritonic devices, particular interest has been devoted towards the integration of quantum emitters with periodic structures supporting collective modes, such as surface plasmon polariton Bloch waves or surface lattice resonances. These resonance features can be properly controlled by acting on the design of the photonic architecture. Among the various possibilities, the exploitation of photonic metamaterials lacking periodic translational order (e.g. quasicrystals) can significantly increase the design degrees of freedom of the photonic component thus offering interesting perspectives in the development of novel hybrid platforms [2]. Here, the focus is on strongly-coupled systems in which quantum emitters are integrated with both periodic and quasi-periodic two-dimensional plasmonic crystals. The radiative properties of the heterostructures along with their intrinsic photophysics were investigated by means of both steady-state and time-resolved optical spectroscopies [3,4]. This work is stimulating toward a further exploration on the symmetry-dependent properties of long-range ordered 2D arrays, with a particular focus on their peculiar near-field distribution. More in general, the presented results can set the stage for the realization of novel polaritonic devices with tailored properties for specific applications, such as photocatalysis.

Acknowledgement

This work is part of the REPLY project that received funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation program (grant agreement No 101002422).

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Nanoscale Science and Technology
Room 114 - Session NS1-TuM

Synthesis and Visualization of Nanostructures I

Moderator: Aubrey Todd Hanibiki, Laboratory for Physical Sciences

8:00am **NS1-TuM-1 Fabrication of High Aspect Ratio Gan and AlN Nanopillar Arrays with M-Oriented Sidewalls by Combining Dry and Wet Processes for the Next Generation of Deep Ultraviolet Light-Emitting Diodes**, ***Lucas Jaloustre***, *S. Sales de mello,* CNRS-LTM, France; *L. Valera,* CEA, France; *S. Labau, C. Petit-Etienne,* CNRS-LTM, France; *G. Jacopin,* CNRS, France; *C. Durand,* CEA, France; *E. Pargon,* CNRS-LTM, France

III-N materials have emerged as promising candidates for ultraviolet (UV) light-emitting diodes (LEDs) due to their wide band gap [1]. However, the current planar architecture of III-N LEDs has demonstrated unsatisfactory efficiency, particularly for wavelengths below 250 nm where the external quantum efficiency drops below 0.1%. One strategy to address this challenge is the transition from a planar to a core-shell geometry [2], which offers many advantages (reduced defects, larger emissive area, better light extraction). This approach involves patterning organized arrays of AlN or GaN nanowires with a high aspect ratio (AR>10) and ideally *m*-plane sidewalls using plasma etching. Metalorganic Chemical Vapor Deposition (MOCVD) is then used to radially grow emissive quantum wells.

In this work, the authors developed an Inductively Coupled Plasma etching process using a Cl_2 chemistry and a hard mark, suitable for the realization of highly anisotropic and smooth GaN and AlN pillars with an aspect ratio up to 16. They demonstrate that the III-N etching using Cl_2 plasma is driven by crystallographic orientation preferential etching and ultimately leads to the formation of *a*-plane sidewalls (Fig. 1-a), and not to the desired *m*-faceting [3].

Post-etching chemical treatments have been explored to improve sidewall roughness and to favour the appearance of the *m*-planes. The authors demonstrate the feasibility of such *m*-faceting on III-N pillars with KOH wet etching and highlight two key parameters for this process[4]: 1) The presence of a HM with a suitable shape during the wet to prevent top pillar erosion and drives the etching, allowing the transfer of the HM shape onto the pillars (Fig. 1-b and 1-c). 2) A tapered pillar profile, which is mandatory to ensure the HM shape transfer.

The wet mechanisms leading to this faceting, and the significant impact of KOH concentration on the etching dynamics (Fig. 2), will be discussed.

This work provides a promising path for achieving pillar arrays that serve as the first building block for highly efficient UV core-shell LEDs. The fabrication of straight and smooth GaN and AlN pillar arrays with the desired crystalline *m*-planes sidewalls (Fig 1-d) was achieved through a two-step process combining Cl_2 plasma etching and KOH wet etching. Finally, successful MOCVD growth of AlGaN/AlN MQWs has been achieved through a collaboration with CEA-PHELIQS, demonstrating UV-C emission on such structures (Fig. 3).

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8:15am **NS1-TuM-2 Exploring Large PAH in "Stardust" by HR-AFM**, ***Percy Zahl,*** Brookhaven National Laboratory; *M. Chacon-Patino, J. Frye-Jones,* National High Magnetic Field Laboratory

Meteorites provide a glimpse into the past of the solar system by preserving snapshots of the universe for millions to billions of years. (1) Organic material, i.e. Polycyclic Aromatic Hydrocarbons (PAH), forms over this time, creating common molecules seen on Earth such as carboxylic acids, aromatic and aliphatic hydrocarbons, and most importantly for life, amino acids. (1–4) Organic material formed in such a way is often revealed as ultra-complex mixtures, similar to fossil fuels and natural dissolved organic matter seen on Earth.

However, these ultra-complex organic mixtures require ultrahigh-resolution analytical techniques to achieve comprehensive molecular-level characterization.

Very little work has been done so far using High Resolution non-contact Atomic Force Microscopy (HR-AFM) (5). Here we analyze highly enriched PAH meteorite sample extracts using our preliminary extraction method similar to that used for fossil fuels. Acetone assists in the extraction of highly aromatic compounds out of a powdered form of meteorite specimens that have already been extracted in a polarity gradient from methanol to toluene. The resulting fraction is enriched in highly aromatic/hydrogen-deficient compounds, as suggested by ultrahigh resolution FT-ICR mass spectrometry.

Comparison with a fraction extracted with chloroform demonstrates a drop in the H/C ratio (a measure of aromaticity) from an average 1.6 for chloroform species down to 0.6 for acetone-extracted compounds. This correlates with the images observed in HR-AFM, where more aliphatic species were observed in the chloroform extract compared to the highly aromatic compounds observed in the acetone fraction. The selected motifs observed highlight the highly aromatic composition of species from the acetone fraction, containing complex ring systems.

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8:30am **NS1-TuM-3 Direct Atomic Manipulation of a Buried Graphene Interface**, ***Marek Kolmer***, Ames National Laboratory; *J. Hall,* Iowa State University; *S. Chen,* Ames National Laboratory; *S. Roberts, Z. Fei,* Iowa State University; *Y. Han,* Ames National Laboratory; *M. Tringides,* Iowa State University

Controlling the two-dimensional (2-d) materials via interlayer interactions is a promising strategy for synthesizing novel electronic and topological phases. These interactions may be tuned locally via atomic-scale manipulations using scanning tunneling microscopes (STMs). However, the practical application of STM manipulation encounters fundamental problems in protecting the designer structures formed atop the surface. We will present a material platform and experimental protocol for precisely manipulating the buried epitaxial graphene interface, which can serve as a template for further targeted synthesis. The covalent bonds between a graphene buffer layer and the silicon carbide substrate may be reversibly broken and restored using the electric field from the STM tip. The charge separation resulting from covalent bond redistribution is used to pull or push the interface atoms by controlling the polarity of the tip-sample bias voltage. The reported effect propagates to the top graphene layer to form a pattern of nanoprotrusions with lateral precision, reaching the dimension of a single unit cell of the interface moiré lattice (~1.8 nm).

Local and reversible electric-field-induced patterning of the epitaxial graphene heterostructures from the bottom interface presents an alternative architecture to synthesize metastable configurations at the interface. The decoupled interface could be locally arranged in ordered superstructures, creating a new 2-d phase with long-range periodic potential experienced by the Dirac electrons in the top graphene layer(s). The location of the voltage pulses controls the period of these superstructures. It allows exploring novel, rich 2-d quantum phases with low-energy electronic excitations of tailored superlattice minibands. This band structure control is realized directly within the epitaxial graphene-based tunable heterostructures on the technologically relevant silicon carbide substrate.

Acknowledgements This work was supported by the U.S. Department of Energy (DOE), Office of Science, Basic Energy Sciences, Materials Science and Engineering Division. The research was performed at the Ames National Laboratory, which is operated for the U.S. DOE by Iowa State University under contract # DE-AC02-07CH11358.

8:45am **NS1-TuM-4 Effect of Methanol and Photoinduced Surface Oxygen Vacancies on the Charge Carrier Dynamics in TiO_2**, *B. Guner,* ***Omur E. Dagdeviren,*** École de technologie supérieure, University of Quebec, Canada

The migration of holes in metal oxides such as TiO_2 plays a vital role in (photo)catalytic applications. The dynamics of charge carriers under operation conditions can be influenced by both methanol addition and photoinduced surface oxygen vacancies (PI-SOVs) [1]. Nevertheless, the existing knowledge of the effect of methanol as a function of PI-SOVs solely concentrates on the chemical reduction process. For this reason, the fundamental understanding of the time-dependent charge carrier-vacancy interactions with the presence of methanol is impaired. To overcome this challenge, we performed time-resolved atomic force microscopy measurements to quantitatively disclose the dynamics of charge carriers in a rutile-terminated, single-crystal TiO_2 (100) sample under the influence of methanol as a function of high-energy ultraviolet (UV) surface irradiation [2-4]. We have three major experimental findings: (I) The addition of methanol decreased the time constant, and hence, the mobility of the charge carriers. (II) The energy barrier within the system was decreased by UV irradiation; however, the presence of methanol did not alter the corresponding barrier. (III) The reversibility of the charge carrier dynamics was observed upon the termination of UV irradiation. Our analysis of methanol's role in hole mobility as a function of surface irradiation contributes to the understanding of surface interactions and charge carrier dynamics, offering valuable insights for (photo)catalytic applications.

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Funding information:

This work was supported by the Canada Economic Development Fund, Natural Sciences and Engineering Research Council of Canada, and Le Fonds de Recherche du Québec - Nature et Technologies.

9:00am **NS1-TuM-5 Single Nanofabrication Step of Low Series Resistance Silicon Nanowire-Based Devices for Giant Piezoresistance Characterization***, K. Shao Chi, L. Spejo,* University of Campinas (UNICAMP), Brazil*; R. Minamisawa,* Institut für Mathematik und Naturwissenschaften Fachhochschule, Switzerland*; J. Diniz,* ***Marcos Puydinger,*** University of Campinas (UNICAMP), Brazil

Strained silicon nanowires (NWs) are essential in sensor applications and in the microelectronics industry, as they exhibit giant piezoresistance and variable carrier mobility as a function of stress. In this work, individual NWs were fabricated from strained silicon-on-insulator (sSOI) thin films with 0.8% biaxial strain. After a lithographic step, the buried oxide (BOX) was removed from the SOI film, thus suspending the NWs. The new boundary condition induces mechanical stress amplification, now uniaxial in the NW longitudinal direction (Fig. 1a). Finite element method simulations show that before the removal of SiO2, the nanowires exhibit predominantly uniaxial strain of around 0.8%, while the peripheral pads show a 0.8% biaxial strain. In this sense, the proposal is to stress the NWs to levels higher than those employed in industry.

The solutions proposed in the literature usually require external actuators or complex multi-step nanofabrication infrastructure [1], [2]. Here, we present a method for prototyping sSiNWs without external actuators and measuring their piezoresistance directly in a 2-probe setup (Figs. 1b and 1c) without further lithographic or metal deposition steps for the electrical contacts. We were able to simplify the steps involved in the fabrication, thus making fast prototyping of strained NWs possible using a single-step protocol and electrical characterization with optimized contact resistance.

In addition, Raman spectroscopy was used to measure the NW uniaxial stress. Figure 2 shows an example of the fabricated suspended sSiNW and its spectrum. We have measured the uniaxial mechanical stresses of around 2.9 GPa for 200 nm-wide sSiNWs, which vary depending on the NW dimension. Parameter optimization can further result in the fabrication of MOSFETs based on a single ultra-strained NW in gate-all-around (GAA) topology, as well as chemical and physical sensors for various technological applications.

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9:15am **NS1-TuM-6 Size-Dependent Oxidation of Supported Pd and Pd-Pt Nano-Catalysts Under Methane Oxidation Conditions***, A. Large,* Diamond Light Source, UK*; H. Hoddinott, R. Palmer,* University of Swansea, UK*;* ***Georg Held,*** Diamond Light Source, UK

The importance of cluster size effects in heterogeneous catalysis has been recognized for some time and numerous studies have addressed this issue. XPS is awell-established technique to study changes in the chemical composition and electronic structure of catalyst nanoparticles. However, as XPS is an averaging technique and based on the detection of electrons, experiments require a narrow distribution of cluster size and a conducting homogeneous support in order to avoid sample charging, which would prevent accurate measurements of chemical shifts. Traditional methods of catalyst synthesis by impregnation/calcination of support powders lead to very large particle size distributions (typically ±50 %) and insulating samples, which fail on both the above criteria. Here we present an operando XPS study of Al_2O_3-supported Pd and Pd-Pt nanoparticles under various dry and wet reaction conditions for methane oxidation ($CH_4 + O_2$ [+ H_2O]). In order to create a system as close to industrial catalysts as possible, the support consists of 5-10 nm thick alumina films electrochemically grown on Al foil. The nanoparticles (1 to 10,000 atoms) were produced by gas condensation and size-selected within 4-5%. TEM confirms the narrow size distribution. The XPS spectra of the clusters before the reaction show clear chemical shifts as function of particle size. Under dry reaction conditions larger clusters tend to oxidize fully (Pd II) whereas smaller particles show a mix of oxidation states (Pd 0-II).The situation is less clear under wet conditions, which could be due to sintering.

9:30am **NS1-TuM-7 Evidence from Molecular Force Microscopy of Magnetic Monopole-Like Behavior in Chiral Molecule-Coated Superparamagnetic Nanoparticles***,* ***Sidney Cohen****, Q. Zhu,* Weizmann Institute of Science, Israel*; J. Fransson,* Uppsala University, Sweden*; O. Brontvein, R. Naaman,* Weizmann Institute of Science, Israel

Ever since Dirac's proposition[1] nearly a century ago that magnetic monopoles should exist, experimentalists have searched for them without success. A flurry of work starting about 50 years ago eventually showed magnetic *monopole-like* behavior in very limited and extreme conditions. The potential for tunable energy storage and stable information storage makes such structures interesting technologically. Here, we use atomic force microscope (AFM)-based molecular force microscopy to show that superparamagnetic iron oxide nanoparticles (SPIONs) coated with an amino acids of opposing chirality (D and L-cysteine) exhibit monopole magnetic behavior whose polarity depends on the chirality of the molecular entity. The effect is governed by the chiral-induced spin selectivity effect (see for instance references 2-4). Attractive magnetic forces were measured between a magnetized Ni film and an SPION attached to the AFM tip through a polyethylene glycol linker. Experiments were performed in a phosphate-buffered saline solution. A thin Ti (oxide) layer covered the Ni to prevent chemical interaction between the cysteine thiol and Ni. The direction of the field was changed between up and down by flipping a magnet held under the substrate. The magnetic force is comprised of spin-exchange interactions (short range) and magnetic monopole interactions (longer range). The interaction force was observed to vary with chirality of the molecule and magnetic field direction. A significant difference was measured in the pulling force for the D vs. L cysteine and up/down magnet orientations when the Ti (oxide) layer was about 2 nm thick. Thicker layers (of Ti oxide or aluminum oxide) result in significant reduction of the magnetic force, allowing estimation of the range of the force interaction.

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9:45am **NS1-TuM-8 Mapping the Slow-Decay of End States in a Laterally Extended Graphene Nanoribbon***,* ***Umamahesh Thupakula****,* CEMES-CNRS, France

End states (ESs) of quasi one-dimensional (1D) graphene nanoribbons (GNRs) have become a central focus in recent years because of their inclusion into the classification of topological tailor-made quantum systems.[1] So far, the topological phases of the GNRs are solely characterized by an integer parameter, called Z_2 invariant originating from the Zak phase, which takes values of 0 or 1 corresponding to topologically trivial or nontrivial configurations, respectively.[1] In these calculations, an infinite nanoribbon is always considered after defining the unit cells structure long the ribbon.[2] However, finite length nanoribbons are mainly observed and characterized experimentally. The tracking of the step-by-step building-up of ESs from a few unit cells to the formation of desired

1D/quasi-1D GNR molecular wire electronic band structure remains elusive. Combining picometer scale precision scanning tunneling microscopy, dI/dV spectroscopy mapping (STM and STS) with topological Hückel molecular orbital (HMO) calculations, we explain how topological quantum states are emerging or not with respect to the length 'N' (number of repeating molecular units) of the GNR starting from the basic polyene to the 1D polyacetylene chain and by a step-by-step enlargement of the nanoribbon width.

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Nanoscale Science and Technology
Room 114 - Session NS2-TuM

Novel Imaging Techniques at the Nanoscale

Moderator: Adina Luican-Mayer, University of Ottawa, Canada

11:30am **NS2-TuM-15 Silicon-Containing Poly(Phthalaldehyde) Resists for Nanofrazor Applications – Direct Patterning of Hard Mask Materials by Thermal Scanning Probe Lithography***, **Nicholas Hendricks**, E. Çağin,* Heidelberg Instruments Nano AG, Switzerland

Thermal scanning probe lithography (t-SPL), enabled by the NanoFrazor technology, is establishing itself as a mature and reliable direct-write nanolithography technique for generating nanoscale structures [1-5]. The NanoFrazor technology offers an alternative or complementary process for conventional lithography techniques of electron-beam lithography (EBL) or focused-ion beam (FIB). t-SPL generates patterns by scanning an ultrasharp tip over a sample surface to induce local changes with a thermal stimulus. By using thermal energy as the stimulus, it is possible to perform various modifications to the sample via removal, conversion, or addition of/to the sample surface. Along with an ultrasharp tip, the t-SPL cantilever contains several other important functions such as an integrated thermal height sensor and an integrated heating element both of which are advantageous for fabricating devices for nanoelectronics, photonics, molecular sensing, and quantum computing.

The main thermal imaging resists used in t-SPL are poly(phthalaldehyde) (PPA) based materials that are commercially available from Allresist or Polymer Solutions. PPA is an all-organic based resist capable of undergoing direct sublimation when exposed to temperatures greater than the decomposition temperature, ~180°C, by a localized endothermic depolymerization reaction. With such characteristics, PPA has been able to produce sub-10 nm lateral dimensions while providing sub-nm vertical resolution and having an etch selectivity of 3 to 1 for a trifluoromethane-based reactive ion etch of silicon dioxide, a standard hard mask material. With a flexible synthesis, PPA can undergo an efficient and effective copolymerization to allow for the direct incorporation of silicon-containing functionalities into the thermal imaging resist. Such a strategy allows for the direct patterning of the silicon-containing hard mask for high-resolution and grayscale patterning with a simplified film stack.

Within this presentation, the background and workings of t-SPL will be introduced as well as the nanolithography and processing capabilities of silicon-containing PPA will be shown. There will be a focus on patterning high-resolution, e.g. sub-40 nm features, from a bi-layer stack of silicon-containing PPA and organic transfer layer (OTL) into a silicon substrate.

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11:45am **NS2-TuM-16 Atomic Force Microscopy-Based Nanoscale Mechanics as a Function of Temperature***, **Gheorghe Stan,*** National Institute for Science and Technology (NIST)*; C. Ciobanu,* Colorado School of Mines

In the last decades, Atomic Force Microscopy (AFM)-based nanoscale mechanics underwent significant developments in terms of measurements and analytics.The measured relative contrast between stiff and compliant materials can now be resolved quantitatively through either relevant models or numerical modeling to reveal the true mechanical properties of materials and structures tested. In comparison, the temperature dependence of the mechanical response has not been sufficiently investigated. The AFM modules for thermomechanical applications are just starting to be added on commercial instruments and the theoretical framework for such measurements is still in its infancy. In this work, we analyze AFM-based quasistatic and dynamic measurements from room temperature up to 250 C on few test materials to probe the reliability of these measurements for nanoscale mechanical properties. The temperature dependence of the tip-sample contact mechanics in these measurements was analyzed both analytically and numerically. This analysis consistently reproduced the experimental response and was used to extract the temperature dependence of the elastic modulus of materials tested.

12:00pm **NS2-TuM-17 Cryogenic Scattering Near-Field Optical Microscopy for Probing Optical Properties with 20nm Spatial Resolution at Temperatures < 10k***, **Tobias Gokus,*** attocube systems AG, Germany*; A. Danilov,* attocube systems AG*; R. Hentrich, A. Huber,* attocube systems AG, Germany

Near-field microscopy and spectroscopy has matured as a key technology for modern optics, combining the resolving power of atomic force (AFM) based measurements with the analytical capabilities of optical microscopy and spectroscopy.

Scattering-type near-field microscopy (s-SNOM) has already proven itself vital for probing local optical material properties of modern nanomaterials by enabling applications such as chemical identification [1], free-carrier profiling [2], or the direct mapping and measurement of the dispersion relation of propagating plasmon [3,4], phonon [5], and exciton polaritons [6] in layered materials.

Transferring these near-field measurement capabilities to cryogenic temperatures opens up new avenues for nanoscale-resolved optical characterization of novel materials and their fundamental properties. Until recently, cryogenic s-SNOM measurements were only available to a few experts utilizing home-built microscopes [7].

By integrating a scattering-type near-field optical microscope (s-SNOM) into an ultra-stable, vibrationally damped and automated closed-cycle cryostat system we developed the first commercial cryogenic s-SNOM microscope. It uniquely supports near-field amplitude and phase resolved imaging and spectroscopy in the visible to THZ spectral range and operates in a variable temperature range between < 10K to room temperature. We will demonstrate infrared and visiblenear-fieldimaging with deep subwavelength spatial resolution, visualizing phonon polariton modes in hBNand exciton polariton waveguide modes in MoS_2 at temperatures < 10K.

Furthermore, we will present recent researchresults onspatially resolved measurements of the spectral response of a2D electron gas and its impact on the surface phonon polariton of $LaAlO_3/SrTiO_3$ heterostructure systems by utilizing s-SNOM based mid-infrared spectroscopy at variable temperatures [8,9].

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2D Materials
Room 122 - Session 2D+LS+NS+SS-TuA
Electronics Properties

Moderators: Masa Ishigami, University of Central Florida, **Slavomir Nemsak**, Advanced Light Source, Lawrence Berkeley National Laboratory

2:15pm **2D+LS+NS+SS-TuA-1 NanoARPES for the Study of 2D Materials***,* ***Aaron Bostwick,*** Advanced Light Source, Lawrence Berkeley National Laboratory **INVITED**

Angle-resolved photoemission spectroscopy (ARPES) is the premier technique for the determination of the electronic bandstructure of solids, and has found wide application for manyclasses of materials, such as oxides, semiconductors, metals, and low-dimensional materials and surfaces. Among the important topics it addresses are the underlying many-body interactions that determine the ground and excited state functionalities of all materials. Recently the development of nanoARPES using microfocused x-ray beams has opened ARPES to a wider class of samples and enabled the measurement of 2D devices *in-situ* with applied electric fields, currents, strain and femtosecond laser pulses to the samples. In this talk I will give an introduction to the ARPES technique, the MAESTRO facility and share some our recent work on the bandstructure and many-body interactions in 2D heterostructuresof chalcogenides, graphene, and boron nitride and light induced metastable phases in 1T-TaS_2.

2:45pm **2D+LS+NS+SS-TuA-3 Observation of Interlayer Plasmon Polaron in Graphene/WS_2 Heterostructures***, S. Ulstrup,* Aarhus University, Denmark*; Y. Veld,* Radboud University, Netherlands*; J. Miwa, A. ones,* Aarhus University, Denmark*; K. McCreary, J. Robinson, B. Jonker,* Naval Research Laboratory*; S. Singh,* Carnegie Mellon University, USA*; R. Koch, E. Rotenberg, A. Bostwick, C. Jozwiak,* Advanced Light Source, Lawrence Berkeley National Laboratory*; M. Rosner,* Radboud University, Netherlands*;* ***Jyoti Katoch,*** Carnegie Mellon University, USA

Van der Waals heterostructures offer us exciting opportunity to create materials with novel properties and exotic phenomena such as superconductivity, bound quasiparticles, topological states as well as magnetic phases. In this talk, I will present our work on directly visualizing the electronic structure of graphene/WS_2/hBN heterostructure using micro-focused angle-resolved photoemission spectroscopy (microARPES). Upon electron doping via potassium deposition, we observe the formation of quasiparticle interlayer plasmon polarons in graphene/WS_2 heterostructure due to many-body interactions. I will discuss that such low-energy quasiparticle excitation is important to consider as they can have huge implications on the electronic and optical properties of heterostructures based on 2D transition metal dichalcogenides.

3:00pm **2D+LS+NS+SS-TuA-4 Harnessing the Synergy of X-ray Photoelectron Spectroscopy (XPS) and Argon Cluster Etching for Profound Analysis of MoS_2 and Graphene***,* ***Jonathan Counsell,*** Kratos Analytical Limited, UK*; C. Moffitt, D. Surman,* Kratos Analytical Inc.*; L. Soomary, K. Zahra,* Kratos Analytical Limited, UK

Understanding the intricate properties of two-dimensional (2D) materials such as MoS2 and graphene is pivotal for advancing their applications across diverse fields. However, achieving comprehensive characterization at the nanoscale requires advanced analytical techniques. This study explores the synergistic potential of X-ray Photoelectron Spectroscopy (XPS) coupled with Gas Cluster Ion Source (GCIS) etching and depth profiling to delve deeper into the structural and electronic intricacies of MoS2 and graphene.

By integrating XPS with GCIS etching, we not only discern the elemental composition, chemical bonding, and electronic states of these materials with exceptional precision but also unravel their depth-dependent characteristics. The incorporation of GCIS etching facilitates controlled removal of surface layers, enabling depth profiling to uncover buried interfaces, defects, and contamination effects that influence spectral results.

The combined approach allows for the characterization of MoS2-graphene heterostructures, providing insights into interfacial interactions and electronic coupling mechanisms. Through systematic analysis, we demonstrate the complementary advantages of XPS and GCIS etching in elucidating the structural and electronic complexities of 2D materials.

The integration of GCIS etching with XPS not only enhances the depth resolution and sensitivity of the analysis but also offers a deeper understanding of the nanoscale landscape of MoS2, graphene, and their heterostructures. This multidimensional approach accelerates the development of tailored devices and applications based on 2D materials, propelling advancements in nanotechnology and beyond.

3:15pm **2D+LS+NS+SS-TuA-5 Monark Quantum Foundry: Advancing 2d Quantum Materials Through Automated Pipelines***,* ***Amirhossein Hasani****, N. Borys,* Montana State University, USA*; H. Churchill,* University of Arkansas

At the MonArk Quantum Foundry, located at Montana State University and the University of Arkansas, we are advancing the field of 2D quantum materials through the development of 2D Quantum Materials Pipelines (2D-QMaPs). These pipelines automate and streamline the critical stages of 2D quantum material sample preparation and device fabrication.

Robotic Exfoliation and Flake Hunting: Our process begins with an automated robotic exfoliator designed to produce up to 50 chips per hour, each measuring 1 cm by 1 cm. This machine carefully peels off thin layers from bulk materials, allowing precise control over factors such as temperature, pressure, and applied force, which is crucial for producing high-quality thin flakes, including single-layered crystals. After exfoliation, an automated optical cataloger identifies and catalogs the thin flakes. This system scans the entire chip, locates these flakes, and stores their information in a database. The cataloger uses advanced machine learning and AI algorithms for quick and accurate detection and features a motorized stage with photoluminescence and Raman spectroscopy capabilities, completing a full scan at 20x magnification in approximately 5 minutes.

Device Fabrication and Packaging: The identified flakes are then transferred to a specialized environment for device fabrication and packaging. This stage employs high-resolution thermal scanning probe lithography, laser writing systems, and electron beam evaporators to create devices. The entire process is conducted in an inert atmosphere (glove box), preserving the material's integrity by preventing exposure to air and moisture.

Characterization: The final stage integrates quantum device characterization tools, including photoluminescence (PL), Raman spectroscopy, magneto-optical probes, nano-optical spectroscopy, bulk and nanoscale cryogenic magnetometry, and magneto-transport measurements. These tools provide a comprehensive analysis of material and device properties, including mobilities, transition temperatures, photon autocorrelations, coherence times, and more.

Through the development of the 2D-QMaPs, the MonArk Quantum Foundry is revolutionizing the study of 2D quantum materials, paving the way for significant advancements in quantum computing, sensing, and other technologies.

4:00pm **2D+LS+NS+SS-TuA-8 Manipulation of Chiral Interface States in a Moiré Quantum Anomalous Hall Insulator***,* ***Tiancong Zhu,*** Purdue University **INVITED**

Quantum anomalous Hall (QAH) effect reflects the interplay between magnetism and non-trivial topology characterized by integer Chern numbers, which is expressed by chiral edge states that carry dissipationless current along sample boundaries. The recent discovery of QAH effect in van der Waals moiré heterostructures provides new opportunities in studying this exotic two-dimensional state of matter. Specifically, magnetism in these moiré QAH systems is induced by orbital motion of electrons, which allows full electrical control of the magnetic and the corresponding topological state. In this talk, I will discuss our recent scanning tunneling microscopy and spectroscopy (STM/STS) measurements on twisted monolayer-bilayer graphene (tMBLG), where QAH effect with gate-switchable Chern numbers have been observed previously in transport measurement. First I will discuss local scanning tunneling spectroscopy measurements on the correlated insulating states at $v = 2$and $v = 3$electrons per moiré unit cell, where the $v = 3$state shows a total Chern number of ±2. Under a small magnetic field, the sign of Chern number at the $v = 3$states can be switched by changing the local carrier concentration with gating, which is a result of competition between bulk and edge orbital magnetization of the QAH state.[1] The observation of a gate-switchable Chern number provides us an opportunity to directly visualize the chiral edge state in a moiré QAH insulator for the first time, which I will show through gate-dependent STS measurement and d*I*/d*V* mappings.[2] I will also demonstrate the capability to manipulate the spatial location and chirality of the QAH edge state through controlling the local carrier concentration with the STM tip.

[1] C. Zhang, T. Zhu et al,. Nature Communications, 14, 3595 (2023)

[2] C. Zhang, T. Zhu et al., Nature Physics (2024)

4:30pm **2D+LS+NS+SS-TuA-10 Scanning Tunneling Microscopy and Spectroscopy of Single Layer $NiTe_2$ on Au***, **Stephanie Lough,*** University of Central Florida*; M. Ishigami,* University of Central Florida

Previous angle-resolved photoemission studies [1, 2] have shown that $NiTe_2$ is a type II Dirac material that possesses a Dirac point very close to the Fermi level. In addition, the material has a topological surface state which can be valley spin-polarized.A recent study [3] has shown that this state can be exploited to develop Josephson diodes with potential applications as memory devices which can be coupled to qubits. There has been a significant interest [3-5] in studying properties of single layer $NiTe_2$ due to its strong interlayer coupling in bulk via Te Pz orbitals.

In this talk, we will discuss the properties of single layer $NiTe_2$ on Au generated by gold-assisted exfoliation measured using low temperature scanning tunneling microscopy and spectroscopy. We find that this interface possesses rectangular lattice with periodicities that are different from bulk $NiTe_2$ or Au (111) by over 30%.Tunneling spectra reveals strong coupling between $NiTe_2$ and Au (111).We compare these results recent theoretical calculations [6] on strained $NiTe_2$ and its impact on topological surface states.

1.Ghosh, S., et al., *Observation of bulk states and spin-polarized topological surface states in transition metal dichalcogenide Dirac semimetal candidate NiTe2.* Physical Review B, 2019. **100**.

2.Mukherjee, S., et al., *Fermi-crossing Type-II Dirac fermions and topological surface states in NiTe.* Scientific Reports, 2020. **10**(1).

3.Pal, B., et al., *Josephson diode effect from Cooper pair momentum in a topological semimetal.* Nature Physics, 2022. **18**(10): p. 1228-+.

4.Zhao, B., et al., *Synthetic Control of Two-Dimensional NiTeSingle Crystals with Highly Uniform Thickness Distributions.* Journal of the American Chemical Society, 2018. **140**(43): p. 14217-14223.

5.Zheng, F.P., et al., *Emergent superconductivity in two-dimensional NiTe crystals.* Physical Review B, 2020. **101**(10).

6.Ferreira, P.P., et al., *Strain engineering the topological type-II Dirac semimetal NiTe.* Physical Review B, 2021. **103**(12).

4:45pm **2D+LS+NS+SS-TuA-11 Nanoscale heterogeneities at Transition Metal Dichalcogenide-Au Interfaces***, **Taisuke Ohta,** A. Boehm, A. Kim, C. Spataru, K. Thuermer, J. Sugar,* Sandia National Laboratories*; J. Fonseca Vega, J. Robinson,* Naval Research Laboratory

Two-dimensional geometry renders unique screening properties in transition metal dichalcogenides (TMDs). Consequently, the electronic properties of TMDs are susceptible to extrinsic factors (*e.g.*, substrate, strains, and charge transfer), and display spatial nonuniformities. Thus, material combinations (*i.e.*, TMD, dielectrics, and metals) and their nuanced interactions need to be considered when designing TMD-based devices. Of particular importance are the interfaces with metallic contacts. Uncovering the origin of heterogeneities at TMD-metal interfaces and establishing strategies to control TMD-metal interfaces could enable engineering pathways for future applications. We show that the electronic structures of exfoliated WS_2-Au interfaces exhibit pronounced heterogeneity arising from the microstructure of the supporting metal. These electronic structure variations indicate spatially nonuniform doping levels and Schottky barrier height across the junction. Through examination using photoelectron emission microscopy, we reveal key differences in the work function and occupied states. With *ab initio* calculation, electron backscatter diffraction, and scanning tunneling microscopy, our measurements show distinct variations in excess of 100meV due to the crystal facets of Au. Additionally, when multilayer WS_2 and Au(111) facets are azimuthally aligned, strong interactions induce mechanical slippage of the interfacing WS_2 layer, with respect to the rest of the WS_2 layers, resulting in local stacking variations with an occupied state energy shift of 20-50meV. Finally, we employed oxygen plasma treatment of Au to fabricate homogenous TMD-Au interfaces while also tuning the electronic properties of the TMDs. Our findings illustrate that the electronic properties of TMDs are greatly impacted by the interface interactions at energy and length scales pertinent to electronics and optoelectronics.

The work at Sandia National Laboratories (SNL) was supported by LDRD program and the US Department of Energy (DOE), Office of Basic Energy Sciences, Division of Materials Sciences and Engineering (BES 20-017574). The work at the US Naval Research Laboratory was funded by the Office of Naval Research. SNL is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly-owned subsidiary of Honeywell International, Inc., for the US DOE's National Nuclear Security Administration under contract DE-NA0003525. This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the US DOE or the US Government.

5:00pm **2D+LS+NS+SS-TuA-12 Xenon Trapping in Silica Nanocages Supported on Metal Powder***, **Laiba Bilal,*** SBU*; A. Boscoboinik,* Brookhaven National Laboratory

Trapping of Xenon gas atoms in silica nanocages supported on metal powders (Ru and Co) is investigated by lab-based ambient pressure X-ray photoelectron spectroscopy (AP-XPS).Xenon, being a noble gas, has very low reactivity[1]. This makes it useful for applications where chemical reactions are unwanted. The first use for Xenon was in flash lamps used in photography[2], and it is still used for this purpose today. It has wide applications, from its use in the cells of plasma television as a propellant in spacecraft that use ion propulsion[3] to its various applications in the medical industry[4].

Xenon occurs in slight traces within Earth, 1 part in 10 million by volume of dry air[2]. Like several other noble gases, xenon is present in meteorites and manufactured on a small scale by the fractional distillation of liquid air. However, Xe's concentration in the earth's crust and the atmosphere are much lower than predicted, which is also known as the "missing Xenon paradox"[5].

The Discovery of an effective way to trap and separate Xenon from other gases can have significant advantages. The presence of Xenon in nuclear fuel rods was partially responsible for the Chernobyl accident[6]. Consequently, the nuclear energy industry is also trying to imprint a way to control the release of Xe, produced during the nuclear fission of uranium. Characteristics of chemical interactions between Xe and metal surfaces have been observed and explained, also several low-energy electron diffraction studies at cryogenic temperatures have experimentally demonstrated an on-top site adsorption preference for Xe adatoms on metal surfaces[7].

Prior studies on 2D silicate bilayers grown on metal supports at Brookhaven National Lab showed that these structures could irreversibly trap all noble gases larger than Ne[8] at room temperature. The noble gas atoms were trapped within hexagonal prism-shaped silicate nanocages-like structures and could then be released by heating the materials to different temperatures, i.e., Ar: 348 K, Kr: 498 K, Xe: 673 K, Rn: 775 K[8,9]. Figure 1A illustrates the potential energy diagram for a noble gas atom getting trapped in a silica nanocage. It can be seen that the activated physisorption mechanism that traps noble gas atoms has a high desorption energy barrier (E_{des}).

Figure 1B shows a silicate bilayer structure (side and top views), while Figure 1C shows a hexagonal prism nanocage, the building block of the bilayer structure. [8]. Since synthesizing such silicate bilayers is very expensive and time-consuming[10] for practical purposes, my project focuses on developing and testing scalable silicate nanocage[9] materials to trap noble gases, with especial focus on Xenon.

Nanoscale Science and Technology
Room Central Hall - Session NS-ThP

Nanoscale Science and Technology Poster Session

NS-ThP-1 Characterization of MOS Capacitors on 4H Silicon Carbide Substrate Submitted to Beta Ionizing Radiation*, E. Magalhaes,* Center for Semiconductor Components and Nanotechnology - CCNano, Brazil*; R. Reigota Cesar,* ***José Alexandre Diniz,*** Center for Semiconductor Components and Nanotechnology - CCSNano, Brazil

This work presents a study of the characterization of the MOS capacitors when subjected to ionizing radiation. Such experiments support analyzing the robustness of silicon carbide semiconductor devices, which has attracted increasing attention due to its robustness properties and tolerance to ionizing radiation. For these studies, beta radiation was applied as a source of ionizing radiation, and the Capacitance - Voltage and Current-Voltage characteristics were obtained for devices as-fabricated and post-irradiation. The extracted Flat-band Voltage (VFB) and Leakage Current (LC) parameters were compared to evaluate the irradiation robustness.

In our experiments, MOS capacitors were fabricated with 300 nm thick aluminum upper and down electrodes and 50 nm thick silicon oxide deposited on the N-type 4H-SiC wafers with a 30 µm thick epitaxial layer (3x1015 cm-3), and a 350 µm thick substrate (3x1018 cm-3). We carried out the beta irradiation experiment on one sample (post-irradiation). Another control sample (as-fabricated) without irradiation was used to compare the electrical values. To irradiate the sample, a Strontium 90 was a beta source with an energy of 1955.7 KeV, a dose rate of 566 Rad/hand, and an exposure time of 72 hours.

Table 1 presents the average values of VFB and hysteresis, which were extracted from C-V curves, and leakage current for accumulation region, extracted from I-V curves. From Table 1, the average values of VFB of -2.01 and -2.33 for as-fabricated and post-irradiation devices were obtained, respectively. If these values are more negative, an increase in effective charge densities at SiO2/SiC structures can be expected after beta irradiation. The difference between them of 0.32 V indicates a 16% charge density increase. The hysteresis values also increased to 0.05 V for 0.36 V, seven times higher. This agrees with the VFB results. Therefore, MOS capacitors are damaged after beta irradiation. However, it can be expected that this damage was not high due to the extracted average values of leakage currents of 297 µA and 201 µA for as-fabricated and post-irradiation devices were obtained, respectively. These values are the same level of magnitude order with a reduction of 32% in the comparison. Thus, our results indicate that the irradiation damage at MOS capacitors on the SiC substrate was not severe. So, SiC devices can be used for applications with irradiation exposure, such as in space aircraft.

NS-ThP-2 Multidimensional Contact Potential Difference Measurements at the Nanoscale in Inorganic Oxides*, B. Guner,* ***Omur E. Dagdeviren,*** École de technologie supérieure, University of Quebec, Canada

Inorganic oxide-based sample systems are popular for applications in catalysis, sensing, renewable energy, and fuel cells in which electronic properties play important roles. Environmental conditions, e.g., temperature, can greatly impact the electronic properties and thereby the performance. The lack of basic knowledge of the local variation of electronic properties as a function of temperature limits the fundamental understanding of systems and hampers their robustness. Here, we demonstrate the multidimensionality of contact potential difference (CPD, i.e., the difference in the work functions of the gold-coated probe and the sample when they are in proximity and under thermodynamic equilibrium, a.k.a., volta potential) at the nanoscale in inorganic perovskites and metal-oxides with scanning probe microscopy (SPM) measurements [1, 2]. We concentrated on single-crystal, inorganic perovskites (*e.g.*, strontium titanate, $SrTiO_3$) and metal-oxides (*e.g.*, titanium dioxide, TiO_2) to have the least amount of uncertainty of sample properties. We employed an undoped $SrTiO_3$ and TiO_2, as they are vastly utilized due to their ideal lattice match for similar systems, cost efficiency, stability, and technological and scientific importance. Our experiments reveal three important results: (I) the CPD of both $SrTiO_3$ and TiO_2 evolve with temperature, (II) the measured CPD is dominated by the local surface state at small tip-sample separations (*i.e.*, tip-sample distance < 10 nm), and (III) the thermodynamically driven intrinsic doping of the material is the governing mechanism of the variation of the CPD for these sample systems. These results clearly show that care must be given to identify the temperature-dependent change of electronic properties to attain and preserve the desired performance of inorganic oxide-based sample systems.

[1] Bugrahan Guner and Omur E. Dagdeviren, ACS Applied Electronic Materials **4** (8), 4085 (2022).

[2] Bugrahan Guner, Simon Laflamme, and Omur E. Dagdeviren, Review of Scientific Instruments **94** (6) (2023).

Funding information:

This work was supported by the Canada Economic Development Fund, Natural Sciences and Engineering Research Council of Canada, and Le Fonds de Recherche du Québec - Nature et Technologies.

NS-ThP-3 Distributed Injection into a 1D Ballistic Channel*,* ***Bert Voigtländer,*** *K. Moors,* Forschungszentrum Juelich GmbH, Germany*; C. Wagner,* Forschungszentrum Jülich GmbH, Germany*; H. Soltner, F. Lüpke, S. Tautz,* Forschungszentrum Juelich GmbH, Germany

The injection of charge carriers into a 1D ballistic channel is usually considered as local injection at opposite ends of the channel. However, in important cases like edge channels of 2D materials, the injection can happen in a distributed manner along the 1D channel. We calculate the potential in the 2D half-plane, terminated by a 1D ballistic channel using proper boundary conditions for the injection of charge carriers into a ballistic channel. We identify hallmark potential-signatures for an 1D ballistic channel and compare them to the behavior of an ohmic conductive channel.

NS-ThP-4 Atomic Force Microscope Customization for Multidimensional Measurements*,* ***Bugrahan Guner,*** *O. Dagdeviren,* École de technologie supérieure, University of Quebec, Canada

Atomic force microscopy (AFM) is an analytical surface characterization tool that reveals the surface topography at a nanometer-length scale while probing local sample properties. Advanced imaging techniques, such as frequency modulation, to achieve high resolution and quantitative surface properties are not implemented in many commercial systems. In this presentation, we illustrate the step-by-step customization of a commercial atomic force microscope [1]. The original instrument was capable of surface topography and basic force spectroscopy measurements while employing environmental control, such as temperature variation of the sample/tip, etc. We demonstrate the capabilities of the customized system with (automated) frequency modulation-based experiments, *e.g.*, voltage and/or distance spectroscopy [2], time-resolved AFM, and two-dimensional force spectroscopy measurements under ambient conditions. We also illustrate the enhanced stability of the setup with active topography and frequency drift corrections. We think that our methodology can be useful for the customization and automation of other scanning probe systems.

[1] Bugrahan Guner, Simon Laflamme, and Omur E. Dagdeviren, Review of Scientific Instruments **94** (6) (2023).

[2] Bugrahan Guner and Omur E. Dagdeviren, ACS Applied Electronic Materials **4** (8), 4085 (2022).

Funding information:

This work was supported by the Canada Economic Development Fund, Natural Sciences and Engineering Research Council of Canada, and Le Fonds de Recherche du Québec - Nature et Technologies.

NS-ThP-5 Improving Leakage Current from a Super Clean STI Technology of DRAM*,* ***Hyojin Park,*** *B. Choi,* Sungkyunkwan University, Korea

With the arrival of the fourth industrial era represented by AI, the Internet on things (IoT), demands for diverse products and characteristics are increasing in the semiconductor market, and the difficulty of developing semiconductor processes to satisfy these demands is rapidly increasing. Especially, under the extreme scaling, even small parameters can easily change the characteristics or structures unlike before, and new failure situations that did not exist in the past are continuously occurring. For example, in the case of DRAM, making a right size of active layer was also hard with a previous way, but it is successful making the structure as we designed with silicone liner process called thin poly. With this result, we thought there will be no difficulties with the Si liner, but unfortunately extreme size shrinkage revealed a problem of Si liner scheme. It is the increasing of interfacial leakage current in STI because of the defects in Si liner itself. Fortunately, we were able to develop the new Si liner process that can form liner like epitaxial Si, and with this super clean STI technology we were able to improve the defects of Si liner. The reduction in defects led to an enhancement in interfacial leakage current, and through this, the problems encountered with STI appeared to be resolved. However, the introduction of this super clean STI technology has also caused new problems that we don’t expect before.Due to abnormal growth in some STI top regions, a bridge failure occurred between adjacent STI, making it impossible to properly isolate the STI and causing interference between adjacent cell transistors. We conducted various evaluations to solve this problem, and as a result, we were able to provide a truly novel STI technology that improved bridge defects through Si liner thickness control. In the future, scaling will continue, and new and unprecedented problems will continue to arise. However, this super-clean STI technology has become a reassuring cornerstone that supports the STI development even in the middle of difficult hardships and will become a major force in DRAM development beyond D1a node.

NS-ThP-6 Surface Assembly of Tetraphenylporphyrin Mediated by Reaction with Tin Tetrachloride Pentahydrate Investigated with Atomic Force Microscopy*, **Quynh Do**, J. Garno,* Louisiana State University

Free-base tetraphenylporphyrin (H_2TPP) was linked to a Si(111) substrate by reaction with tin tetrachloride pentahydrate ($SnCl_4 \cdot 5H_2O$). Introduction of $SnCl_4{\cdot}5H_2O$ into a solution containing free-base porphyrin resulted in the coordination of a tin atom at the center of the H_2TPP macrocycle. The resulting metalated molecules (Sn-TPP) are skewered together to produce a cofacial arrangement. The metal Sn atom is coordinated to the center nitrogen atoms of the porphyrin macrocycle with O-Sn-O bonding to adjacent molecules. Nanopatterns were prepared by colloidal lithography for subsequent characterizations with atomic force microscopy (AFM). Unsubstituted porphyrin molecules typically attach randomly to surfaces through physisorption. However for Sn-TPP, covalent bonds to the surface and linked individual Sn-TPP molecules was facilitated by O-Sn-O bonds.

The coordination of Sn atoms within the macrocycles of H_2TPP was tracked with UV-Vis spectral analysis, exhibiting a red shift in the Soret band and changes in the relative intensities of the four Q-bands of the porphyrin. The red shift in the Soret band is attributable to the expansion of the porphyrin macrocycle to accommodate the larger Sn atom. Nanostructures were produced using colloidal lithography to facilitate AFM characterizations and to evaluate the influence of experimental parameters of concentration and reaction time for producing stacked Sn-TPP assemblies. A surface mask of silica mesospheres was used to guide the adsorption of Sn-TPP molecules to form defined geometries. After removal of the silica particle mask by steps of rinsing and sonication, nanorings of Sn-TPP were observed. Periodic arrangements of nanorings over broad areas of Si(111) were evident for topography images captured with tapping-mode AFM. Precise control of material properties can be gained by directing the assembly of porphyrins to form nanostructures, and colloidal lithography enables high-throughput at the nanoscale. The conjugated electronic structure of porphyrins leads to thermal stability and distinct electronic and optical properties which are tunable by the choice of functional substituents. Nanomaterials of porphyrins have promising applications in molecular electronics, solar cells, and sensors.

NS-ThP-7 Probing Isotopic Effects on Hyperbolic Phonon Polaritons in MoO_3 with Nanoscale IR Imaging*, **Jeremy Schultz**, S. Krylyuk,* National Institute of Standards and Technology (NIST)*; J. Schwartz,* University of Maryland, College Park*; A. Davydov, A. Centrone,* National Institute of Standards and Technology (NIST)

Hyperbolic phonon polaritons (HPhPs), hybrids of light and lattice vibrations in polar dielectric crystals, enable nanophotonic applications by providing a method to confine and manipulate light at the nanoscale. Molybdenum trioxide (α-MoO_3) is a naturally hyperbolic material, meaning that anisotropy in its dielectric function determines the directional propagation of in-plane HPhPs within its reststrahlen bands. A range of strategies are being developed to alter the intrinsic dielectric functions of natural hyperbolic materials and control the confinement and propagation of HPhPs. Since isotopic disorder can limit phonon-based processes such as HPhPs, here we synthesize isotopically enriched $^{92}MoO_3$ (^{92}Mo: 99.93 %) and $^{100}MoO3$ (^{100}Mo: 99.01%) crystals to tune the properties and dispersion of HPhPs with respect to natural α-MoO_3, which is composed of seven stable Mo isotopes. Real-space, near-field maps measured with the photothermal induced resonance (PTIR) technique were used to compare in-plane HPhPs in α-MoO_3 and isotopically enriched analogues within a reststrahlen band (≈ 820 cm^{-1} to ≈ 972 cm^{-1}). We found that isotopic enrichment (e.g., $^{92}MoO_3$ and $^{100}MoO_3$) alters the dielectric function and shifts the HPhP dispersion (HPhP angular wavenumber × thickness vs IR frequency) by ≈ -7 % and ≈ +9 %, respectively, and changes the HPhP group velocities by ≈ ±12 %, while the lifetimes (≈ 3 ps) in $^{92}MoO_3$ were found to be slightly improved, by ≈ 20 %. The latter improvement was attributed to a decrease in isotopic disorder. Altogether, isotopic enrichment was found to offer fine control over the properties that determine the anisotropic in-plane propagation of HPhPs in α-MoO_3, which is essential to its implementation in nanophotonic applications.

NS-ThP-9 Effect of Thermal Annealing on Electrical Property of Platinum Nanowires Deposited by Focused Electron Beam Induced Deposition*, **Rajendra Rai**, U. Dhakal, B. D.C., Y. Miyahara,* Texas State University

Focused Electron-Beam Induced deposition (FEBID) is a direct writing technique which can be used for fabrication of nanoelectronic devices such as single-electron transistor. However, FEBID-deposited nanoelectrodes exhibit high resistance because the as-deposited structures contain unwanted precursor elements like carbon due to its precursor Me3CpMePt (IV), (Me: methyl, Cp: cyclopentadienyl). We report the electrical properties of Pt nanowires with thickness ranging from 2 nm to 200 nm deposited by FEBID technique. We investigated the post-deposition processing techniques to turn the deposited nanowires into electrically conducting as pure Pt metals. We found that the thermal annealing of the as-deposited Pt nanowires can increase in electrical conductance by five orders of magnitude. The resulting annealed wire shows metallic electrical conduction down to 100 mK. We will present the length and width dependence of the electrical conductance and their correlation with the structure of the nanowires which are measured by atomic force microscopy and Kelvin probe force microscopy.

NS-ThP-10 Enhancing Ferroelectricity and Controlling Defects in Aluminum Nitride Thin Films through Ion Bombardment*, **Bogdan Dryzhakov**,* Oak Ridge National Laboratory*; K. Kelley,* Oak Ridge Natinal Laboratory

The wurtzite ferroelectric aluminum nitride (AlN) is a promising multifunctional semiconductor in memory applications. However, its inherent structural factors raise coercive often even above breakdown voltage fields, making reducing the domain switching energy barrier is a key focus. Defects, such as Al and N vacancies and their complexes with native impurities like oxygen and carbon, play a crucial role in determining the dynamics of ferroelectric switching through the interaction with the lattice leading to domain nucleation, pinning, lattice softening, and polarization screening. In this study, we leverage the optical signatures of defect chemistries, probed by cathodoluminescence (CL), to correlate with the ferroelectric behavior of AlN. CL overcomes the wide bandgap of AlN (>6 eV) to excite color centers through ionization processes that exhibit nanoscale heterogeneity and small optical interaction cross-sections. With this understanding in hand, we employ helium ion irradiation to induce local structure changes and tune defect density and type by modulating the dose. CL spectra demonstrate irradiation-dose-dependent altered defect chemistry, suggesting the irradiation dose gradually evolves existing defects to favor the formation of nitrogen vacancies and diminishing oxide complexes. From functional atomic force microscopy measurements, the relative increase in dose correlates to a steep drop off in ferroelectric coercive field, which is accompanied by a significant enhancement of piezoelectric coefficient. By leveraging the method of He-ion irradiation to enhance potential nucleation sites, we reduce the barrier to switching by >40% and postulate the type and functionality of defect states crucial for engineering ferroelectric properties in AlN thin films.

NS-ThP-11 Delamination of Epitaxially-Grown Single-Crystalline GeTe Films for Flexible Phase Change Memory*, **Seohui Lee**, C. Yoo,* University of Central Florida*; H. Shin,* University of Central Florida, Dong-A University, Republic of Korea*; S. Han, Y. Jung,* University of Central Florida

Integrating phase change materials with flexible substrates is crucial for advancing technologies like phase change memory (PCM). This study focuses on GeTe as a fundamental PCM, synthesized over large areas using Chemical Vapor Deposition (CVD). Our objective is to develop thin, flexible GeTe-based materials for PCM applications.

To achieve this, we employ a Ni-electroplating-assisted spalling technique to separate GeTe films from Ge wafers. We confirm the successful delamination of GeTe through Raman spectroscopy and X-Ray Diffraction (XRD).

This work focuses on the development of thin, flexible GeTe-based materials to enhance phase change memory applications by integrating them with flexible substrates. By exploring it, we aim to explore new possibilities for future technologies.

NS-ThP-12 The Incorporation of Nanohelical Metamaterial Into 1D Photonic Topological Insulator System: A Route to the Generation of Strong Chiral Response*, **Sema Guvenc Kilic,*** University of Nebraska-Lincoln*; U. Kilic,* University of Nebraska - Lincoln*; M. Schubert, E. Schubert,* University of Nebraska-Lincoln*; C. Argyropoulos,* Penn State University

During the last two decades, 1D and 2D photonic topological insulators (PTI) have gained great interest due to their potential applications in quantum information technologies, waveguide systems, and next-generation photonic integrated circuits [1-4]. In this study, we propose to combine a 1D photonic crystal system with a 3D nanohelical metamaterial platform that can potentially achieve large chiral response. The optical manifestation of chirality known as circular dichroism (CD) is the differential absorption (A) of left circularly polarized (LCP) light from that of right circularly polarized (RCP) light case ($CD=A_{LCP}-A_{RCP}$). In the proposed system, we employ one-turn nanohelical structures comprising gold (Au) and silicon (Si) helical subsegments. This helical structure is integrated into a 1D photonic topological structure consisting of alternating TiO_2 and SiO_2 flat thin film layers. Based on our systematic finite element modeling studies, we observe a strong leak mode that emerges in the photonic gap region with perfect transmissivity and strong electric field confinement at the central two layers of 1D-PTI. The spectral location of the leak mode is in excellent agreement with the topological edge mode frequency in the superlattice energy dispersion diagram of the proposed PTI system. As a last step of this study, Si-Au plasmonic nanohelical structure with the highest chiroptical response was placed at the center of the topological interface where the electric field coupling is maximum. By engineering the thickness of individual layers in 1D photonic crystal system, we successfully spectrally matched the chirality of the nanohelical structure with the topological leak mode. We observed that the integration of nanohelical structure into the PTI system provided a solid route to generate a strong chirality response.

NS-ThP-14 $Ge(CH_2I)_4$ on Si(100): Matching the Molecule to the Surface and the Science*, **Brandon Blue**, R. Addou, D. Allis, J. Barton, N. Culum, M. Drew, T. Enright, A. Hill, T. Huff, O. MacLean, T. McCallum, M. Morin, M. Moses, R. Plumadore, M. Taucer, D. Therien, D. Vobornik,* CBN Nano Technologies Inc., Canada

As we approach smaller silicon device sizes and search for new functionalities, a promising avenue of research is the combination of silicon with organic molecules. Despite 50 years of research into this field, a concerted multi-factor effort for targeted molecule reactivity has yet to be applied. Herein, we present one such effort with the on-surface, multi-technique analysis of a newly synthesized, highly symmetrical molecule, **t**etrakis(**i**odo**me**thyl)**ge**rmane ($Ge(CH_2I)_4$; TIMe-Ge), consisting of four iodomethyl (CH_2I) “legs” bound to a germanium atom “core” in a tetrahedral geometry. TIMe-Ge was selected for this study based on its expected reactivity and surface behavior with Si(100) 2x1, which was supported by simulations and literature precedent. TIMe-Ge’s properties enable three of its legs to covalently bond with Si(100) 2x1 in a limited number of configurations via C-I dissociative addition, with the fourth CH_2I pointing near-normal to the surface. The unbound CH_2I group was expected to exhibit a distinctive scanning tunneling microscopy (STM) signature due to its low, but non-zero, rotational barrier. Additionally, the iodine atoms on all legs provide an x-ray photoelectron spectroscopy (XPS) signal that is both straightforward to fit, and predictably high in signal intensity: a crucial factor for quantitative analysis in sub-monolayer depositions. Leveraging these characterization features, we examined the impact of sample temperature on TIMe-Ge surface binding configurations, demonstrating a temperature dependence that changed the number of bound legs. For three-leg-bound TIMe-Ge, ultraviolet irradiation and STM tip-mediated bias-pulse dehalogenation were performed on the remaining CH_2I at 77 K, alongside STM tip-mediated rotation of the remaining CH_2I at 4 K. The dehalogenation processes produced carbon-centered radicals potentially suitable for on-surface synthesis, donation of functional groups, and/or the attachment of new functional groups.

NS-ThP-15 Percolation Electronic Transport in 2D Nanowire Networks Containing Void Type Defects*, **Andrew Qiu**, D. Kumar Gorle, S. Alzahrani, A. Ural,* University of Florida

Two-dimensional (2D) networks consisting of one-dimensional (1D) wires, such as carbon nanotubes, metal nanowires, and graphene nanoribbons, are promising candidates for next-generation flexible transparent conducting electrodes in devices such as organic light-emitting diodes (OLEDs), solar cells, touch screens, smart windows, transparent heaters, and liquid crystal displays. 2D nanowire networks also have functionality in device applications such as flexible electronics, thin film transistors, wearables, electronic skin, biosensors, and neuromorphic computation. Electronic transport in 2D nanowire networks is governed by percolation, which deals with the formation of long-range connectivity in random networks. Understanding the impact of void type defects, which could be present due to lack of control in the deposition/fabrication process or introduced intentionally, on percolation electronic transport in nanowire networks is critical for many applications.

In this work, we study the effect of voids on electronic transport in 2D nanowire networks using Monte Carlo simulations. We define the relative void size as the ratio of the length of the side of the void to the length of the side of the nanowire network. We first study the impact of voids on networks consisting of randomly oriented and straight nanowires. We compute the percolation probability in these networks as a function of nanowire density for different relative void sizes. We find that both the mean and standard deviation of the percolation probability density function increase with increasing relative void size. We then compute the relative conductivity change as a function of nanowire density for different relative void sizes and find that it increases approximately linearly with relative void size.The conductivity of 2D nanowire networks exhibits a power-law dependence on nanowire density as predicted by percolation theory. In order to study the effect of voids on this dependence, we extract the local power-law critical exponent as a function of nanowire density for different relative void sizes. We find that the critical exponent approaches 2 at high density for all relative void sizes, in agreement with networks without voids. Using the same procedure as randomly oriented and straight nanowires, we then investigate the impact of voids on the nanoscale charge transport through networks consisting of curvy and aligned nanowires. These results show that Monte Carlo simulations are an essential predictive tool for providing insights into the percolation electronic transport in 2D nanowire networks, which are promising candidates for a wide range of functional applications.

NS-ThP-16 Frequency Shift and Damping Noises of Mechanical Resonator with Quality Factor Modified by Optomechanical Force*, **Md Mahamudul Hasan**, N. Bingamon, B. D.C., Y. Miyahara,* Texas State University

In Frequency Modulation Atomic Force Microscopy (FM-AFM), several noise sources can affect the accuracy and resolution of measurements. Deflection sensing noise in an FM-AFM cantilever, combined with thermal noise, contributes to noise in the frequency shift (Δf) and damping (γ_{dis})1). Therefore, quantifying noise is essential for understanding tip-sample physics and achieving high-resolution imaging in FM-AFM. The optical excitation using laser sources to excite the cantilever not only removes the spurious mechanical crosstalk2,3) but also allows us to modify the Q for the optimized Δf and γ_{dis} measurements4). In our present setup, we developed a low-temperature FM-AFM integrated with a cryogen-free dilution refrigerator (LD250, Bluefors). Here, two lasers with different wavelengths i.e. 1310 nm for excitation and 1550 nm for interferometric detection were deployed. The intensity of 1310 nm was modulated to drive the cantilever at its resonance frequency (f_0) to get a cantilever resonance free from spurious mechanical resonances while the 1550 nm laser was used for deflection sensing. Besides, we also regulated the quality (Q) factor of the cantilever by adjusting the average intensity of the 1310 nm laser without changing the Fabry-Perot cavity length. We carefully observed the Δf and γ_{dis} noise for varying Q and our results showed these noises are independent of the modified Q. We will present the effect of modified Q on the bandwidth and noises of Δf and γ_{dis} measurements.

We sincerely acknowledge funding from NSF (DMR-2122041, DMR-2044920, DMR-2117438) and Texas State University.

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NS-ThP-17 Chemical Synthesis of Metal Nitride Nanoparticles for Electrocatalysis*, **Rihana Burciaga**,* Clark Atlanta University

Copper nitride (Cu_3N) is an intruiging material known for its diverse applications and unique properties. Cu_3N exhibits a range of physical characteristics including high thermal and chemical stability. The material has garnered interest for its potential in semiconductor devices, solar cells, and as a catalyst in various chemical reactions. Studies have demonstrated that Cu_3N can be synthesized through various methods, including reactive sputtering and chemical vapor deposition, and its properties can be tailored for specific applications. However, conventional methods lack of precise control on the size and morphologies of the nitride nanomaterials. Herein, we explore the chemical synthesis of metal nitride nanoparticles by leveraging colloidal chemistry to harness the synthetic control. Uniform size and morphology of pure phases of metal nitride nanoparticles have been successfully obtained. We further explore its electrocatalytic applications in key reactions for sustainable energy applications. Metal nitride nanoparticles demonstrated superior performance in electrocatalysis.

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