Nanoscale Science and Technology Division
Room B113 - Session NS+EM+MN-MoA

Nanoscale Devices, Structures and Materials

Moderators: Aubrey Hanbicki, Laboratory for Physical Sciences, **Deep Jariwala**, University of Pennsylvania

1:40pm **NS+EM+MN-MoA-1 Integrated Nanophotonics Temperature Metrology Platform**, ***Nikolai N. Klimov***, *K. Douglass, D. Barker, T. Bui, S. Robinson, T. Herman, K. Quelhas,* National Institute of Standards and Technology (NIST)

Temperature, being, perhaps, the second most measured physical property after time and frequency plays a crucial role in various aspects of modern technology ranging from medicine and Earth's climate to semiconductor industry and advanced manufacturing process control. While there have been great strides in developing novel thermometry approaches, resistance-based thermometry remains the standard method for disseminating the SI unit of temperature at the highest level of precision. The fundamental limitations of resistance thermometry, as well as the desire to reduce sensor ownership cost, have ignited a substantial interest in the development of alternative technologies such as photonics-based temperature sensors.

At the National Institute of Standards and Technology (NIST), we are developing a new photonics-based temperature measurement solution that has the potential to revolutionize how temperature is realized and disseminated to customers. One of the key elements of our Photonic Thermometry program is an ultra-Sensitive Photonic Thermometer (SPoT) – an on-chip integrated silicon nanophotonic resonator, whose optical resonance frequency shifts with temperature due to high thermo-optic coefficient of silicon and can be used to trace temperature variations with high precision. Our goal is to evolve SPoT into a robust, field-deployable device that is on par or better than the state-of-the-art resistance thermometer.

In this work, we describe the performance of SPoT thermometer, as well as a new photonic readout for SPoT. In our new read-out scheme, we employ a novel offset-locking technique for reading out the resonance wavelength of the SPoT. This method provides extremely high accuracy for relative temperature changes on a short time scale (<< 1s).Our results indicated that the packaged on-chip integrated SPoT can detect temperature fluctuations as small 2 µK over 200 ms integration time. This methodology as well as other proposed methods will be discussed.We also show a benchmark comparison of SPoT thermometer to Standard Platinum Resistance Thermometer (SPRT) – the best-in-class resistance thermometer, in various fixed-point cells of ITS-90, evaluating temperature resolution and repeatability

2:00pm **NS+EM+MN-MoA-2 AVS Dorothy M. and Earl S. Hoffman Scholarship Recipient Talk: Breaking the Efficiency Bottleneck of Micro-LEDs Through Nanoscale and Excitonic Engineering**, ***Yixin Xiao***[1], *R. maddaka, Y. Wu, Y. Malholtra, Y. Guo, S. Yang, K. Sun, A. Pandey, J. Min, Z. Mi,* University of Michigan, Ann Arbor

The performance of conventional optoelectronic devices, such as LEDs and laser diodes, is extremely sensitive to the presence of defects and dislocations. For these reasons, it has remained challenging to achieve high efficiency nanoscale LEDs and laser diodes. For example, while conventional broad area LEDs can exhibit external quantum efficiency (EQE) in the range of 80-100%, the EQE of submicron scale LEDs is often <1%, due to the dominant nonradiative surface recombination. The operation of conventional LEDs involves the radiative recombination of free electrons and holes in the active region. It is known that an exciton, a bound state of an electron and hole through strong Coulomb interaction, can drastically enhance the radiative recombination efficiency, which can be potentially exploited to make micro and nanoscale LEDs relatively immune from the presence of defects/traps. Recent studies have shown that the exciton oscillator strength can be increased by nearly two orders of magnitude in small size InGaN nanowires, due to efficient strain relaxation. Here we demonstrate that the efficiency bottleneck of µLEDs can be fundamentally addressed by utilizing bottom-up III-nitride nanostructures. We report on the demonstration of micrometer scale green and red LEDs with an external quantum efficiency of 25% and 8%, respectively, which are the highest values ever reported to the best of our knowledge. We employ selective area plasma-assisted molecular beam epitaxy as the material synthesis platform. Due to efficient strain relaxation, such bottom-up nanostructures are largely free of dislocations. By exploiting the large exciton binding energy and oscillator strength of quantum-confined InGaN nanostructures, we show that the external quantum efficiency of a green-emitting micrometer scale LED can be dramatically improved from ~4% to >25%. The dramatically improved efficiency is attributed to the utilization of semipolar planes in strain-relaxed nanostructures to minimize polarization and quantum-confined Stark effect and the formation of nanoscale quantum-confinement to enhance electron-hole wavefunction overlap. We have further developed a new approach that included an InGaN/GaN short period superlattice together with an InGaN quantum dot active region to achieve high efficiency red emission. A maximum quantum efficiency of >7% was measured. Our studies offer a viable path to achieve high efficiency micrometer scale LEDs for a broad range of applications including mobile displays, virtual/augmented reality, biomedical sensing, and high-speed optical interconnects, that were difficult for conventional quantum well based LEDs.

2:20pm **NS+EM+MN-MoA-3 Modeling Gas Phase Etching in High Aspect Ratio Stacked Nanostructures for Semiconductor Processing: Stacked SiGe Layer Etching**, ***Zach Zajo***, Stanford University; *D. Mui, J. Zhu, M. Kawaguchi,* Lam Research Corp.; *E. Shaqfeh,* Stanford University

Gate all around (GAA) nano-transistors offer better channel control and increased current carrying capacity compared to FinFETs (Field Effect Transistor) which are currently the standard in the semiconductor industry. However, the need for precise control of their nanoscale features poses a challenge in manufacturing such GAA nano-transistors. The high material selectivity required in fabricating these transistors makes gas phase etching much more appealing in comparison to liquid phase and plasma-based etching techniques. An etching configuration that is of particular interest is one consisting of alternating layers of Si and SiGe from which the SiGe layers are selectively etched by fluorine gas. In the etching of these structures, it is important to have a uniform etch-rate for SiGe layers from top to bottom, to maintain consistency of the etched features. This consistency is essential for the superior performance of the GAA devices. The key gas phase processing challenge then is to determine and maximize the number of SiGe-Si layers that can be stacked in a single structure while still maintaining a nearly uniform etch-rate from top to bottom in the stack. While experiments have offered insights in terms of the effect of layer thickness, number of layers, gas pressure etc. on the viability of the process, such experiments are quite expensive and tedious. We propose and develop computer simulations as a tool to predict the etch profile evolution over time in a gas phase etching process. The tool is based on a mathematical model which considers the transport processes and surface interactions involved in the gas phase etching process – which at the nanoscale is primarily governed by Knudsen diffusion in the free molecular flow regime. Thus, the transport model is formulated as a boundary integral equation which takes into account the direct flux of etchant molecules that any given point on the exposed surface receives from the bulk gas phase as well as the re-emission flux from other parts of the structure itself. We compared the applicability of two different surface reaction models - a model where the local etch rate is linear in the flux at a point and a Langmuir adsorption/reaction model- to connect the net flux received at a point on the surface to the local etch rate. Our results show that the re-emission of etchants at the etching interface plays a vital role in determining the differential etch rates observed across layers at different depths in the stacked feature. In addition, we have characterized the effect of layer thickness and the spacing between adjacent stacks as these impact the etch rates observed from layer to layer.

2:40pm **NS+EM+MN-MoA-4 Fabrication of Silicon Microfluidic Gratings for Neutron Imaging**, *S. Robinson, R. Murphy,* National Institute of Standards and Technology (NIST); *Y. Kim,* National Institute of Standards and Technology (NIST)/ University of Maryland, College Park; *J. LaManna, C. Wolf, K. Weigandt, D. Hussey,* ***Nikolai N. Klimov***, National Institute of Standards and Technology (NIST)

In this work, we describe the development of a spatial modulator for x-ray and neutron beams. Current technology for x-ray and neutron phase imaging uses individual source gratings with a fixed period to modulate the beams. Each fixed-period, or "static" source grating, has a limited range in spatial resolution at a specific length scale, thus, a relatively larger set of individual static source gratings is required to achieve high-resolution imaging. Our team is developing a neutron imaging technique that will probe heterogeneous samples across multiple length scales. To address the need for a large variability of source grating periods, we are building a

[1] **AVS Dorothy M. and Earl S. Hoffman Scholarship Recipient**

silicon microfluidic-based device, DynAmic ReconflgUrable Source grating (DARIUS), that itself is capable to adjust, with high resolution, the grating period from 20 µm to 20,000 µm. With such on-demand tunability, a single DARIUS has the potential to replace more than 500 static source gratings, minimizing the need of fabricating, installing, and aligning a new source grating for the required period. To achieve such functionality, DARIUS features over 5,000 microfluidic grating channels that are etched on both the front and back sides of a 100 mm silicon wafer. Each of these grating channels can be selectively infilled with a neutron and/or x-ray absorbing fluid, and provide real-time reconfigurable spatial modulations for neutron and x-ray beams. In this work, we present updates on the double-sided deep silicon etch (on the front and the back of the wafer), as well as the front-to-back alignment of the silicon channels. We also describe the progress towards wafer bonding to seal the front and back sides of DARIUS device. Due to the challenges of both the microfluidic control and tight fabrication tolerances, we are evaluating the design specifics to address larger grating periods.

3:00pm **NS+EM+MN-MoA-5 The Small Shift Matters – Submilliradian Tilt Goniometry in Scanning Electron Microscopy**, ***Andrew Madison***, *J. Villarrubia, D. Westly, R. Dixson, C. Copeland,* National Institute of Standards and Technology (NIST)*; J. Gerling, K. Cochrane, A. Brodie, L. Muray,* KLA-Tencor*; J. Liddle, S. Stavis,* National Institute of Standards and Technology (NIST)

Electron optical aberrations degrade the accuracy and reliability of scanning electron microscopy. Among multiple aberrations of potential concern, an axial tilt of the electron beam shifts the apparent positions and deforms the intensity profiles of features in scanning electron micrographs. Measurement of the beam tilt can enable either a physical correction of beam deflection or an analytical correction in a measurement function. In this study, we report a novel reference structure and image analysis method to measure such shifts, among other key effects. Our new concept has the potential to improve accuracy in scanning electron microscopy, with multifunctional standards enabling integrative calibrations of beam tilt and beyond. Such advances will be of particular interest in semiconductor manufacturing metrology, where even the small shift matters.

We explore conical frustum arrays as multifunctional reference structures, using physical theory to guide ongoing experiments. For a tilt inclination ϑ, a centroid shift s between the top and bottom edges of a conical frustum shows the effect of tilt. For a frustum height h, the measurement function is simply $\vartheta = \sin^{-1}(s/h) \approx s/h$, yielding a null-tilt sensor and self-calibrating goniometer. To understand the limiting random effect of shot noise, we simulate frustum images using a physical model of electron scattering and emission. At a dose of 60 electrons per nm^2, model shifts show the possibility of submilliradian accuracy for sidewall angles greater than approximately 40 mrad. In experimental measurements, charge accumulation and hydrocarbon contamination may limit the achievable electron dose, while conical asymmetry among other systematic effects will ultimately limit accuracy. In initial experiments, we fabricate submicrometer frustum arrays in silicon using electron-beam lithography and reactive ion etching and demonstrate use of the reference structure in calibrations of a typical scanning electron microscope.

In an integrative calibration, frustum arrays are optimal structures for correlative atomic force, super-resolution optical, and scanning electron microscopy. This workflow yields reference heights and positions that allow calibration of scale factor and correction of scanfield distortion, improving the accuracy of centroid shifts to show electron beam tilt and spatial variation thereof across the imaging field.

4:00pm **NS+EM+MN-MoA-8 On Point – Accurate Integration of Quantum Dots and Bullseye Cavities**, ***Craig Copeland***, *A. Pintar, R. Dixson, A. Chanana, K. Srinivasan, D. Westly, B. Ilic, M. Davanco, S. Stavis,* NIST-Gaithersburg

Self-assembled quantum dots are promising light sources for quantum networks and sensors. These emerging technologies require the accurate integration of quantum dots and photonic structures, but epitaxial growth forms quantum dots at random positions in semiconductor substrates. Optical localization of these random positions can guide the placement of photonic structures by electron-beam lithography. This integration process requires the reliable registration of position data across microscopy and lithography systems. However, large errors can result from multiple sources, including lithographic and cryogenic variation of reference dimensions for microscope calibration, as well as localization errors from optical distortion. Such errors tend to increase across an imaging field, presenting a critical impediment to exploiting the throughput and scalability of widefield microscopy. In this study, we target this problem and show how our solution enables accurate integration to improve device performance and process yield. We develop our methods of traceable localization to calibrate a cryogenic localization microscope – an optical microscope with the sample and objective lens inside of a cryostat, and custom optics outside of the cryostat. We fabricate and characterize arrays of submicrometer pillars in silicon, creating microscopy standards with both traceable reference positions and traceable reference data for thermal expansion coefficient. We image these arrays with the cryogenic microscope at approximately 1.8 K, localize the pillar positions, and use the reference data to calibrate the microscope. Our calibration determines the scale factor of the imaging system and corrects position errors due to complex distortion, among other aberration effects. We combine the results of this cryogenic calibration with our previous assessment of fabrication accuracy by electron-beam lithography, introducing a comprehensive model of the effects of registration errors on Purcell factor. This performance metric quantifies the radiative enhancement that occurs upon integration of a quantum dot into a bullseye resonator. The Purcell factor reaches a maximum value of approximately 11 for error-free registration of the quantum dot and resonator center. Our model demonstrates the possibility of greatly improving Purcell factor across a wide field. Depending on the Purcell factor threshold, on-point integration can increase yield by one to two orders of magnitude. This foundation of accuracy will enable a transition from demonstration devices to efficient processes, leading to the reliable production and statistical characterization of quantum information systems.

4:20pm **NS+EM+MN-MoA-9 Nanostructured Gas Sensors for the Detection of Meat Spoilage**, ***Ken Bosnick***, National Research Council of Canada

The sustainability of the food industry will remain a key societal challenge in the decades ahead. In North America, over 20% of meat produced is wasted, mostly at the later stages in the supply chain [1]. When meat begins to degrade, it releases biogenic amines (e.g., putrescine, H_2N-$(CH_2)_4$-NH_2) for which early detection provides a means to sense the onset of spoilage [2]. Early detection of meat spoilage through the sensing of such amines can proactively assure quality in the food production process and potentially eliminate the need for food recalls and other waste. At the heart of these envisioned quality assurance strategies are portable devices that are capable of rapid detection of low levels of biogenic amines and that can be easily deployed at various stages in the production process. New material technologies with a selective response to low levels of biogenic amines are needed to enable these envisioned devices.

The use of MOS-type gas sensing technology represents a promising avenue for portable gas sensors with many advantages over competing sensing technologies and over more traditional analysis methods. We have investigated the application of Pd-decorated ZnO nanoflowers in a chemiresistive sensing mechanism and found an excellent response of 99.5% at 250 °C towards 400 ppm methylamine [3]. The device also shows a promising response of 45% at room temperature, making it a candidate sensing material for early detection of spoilage in meat-based products. Towards improved performance at room temperature, ZnO nanocantilevers [4] are being investigated for amine sensing and will also be discussed.

[1]FAO. 2011. “Global food losses and food waste – extent, causes and prevention”

[2]Fernanda Galgano, Fabio Favati, Malvina Bonadio, Vitina Lorusso, Patrizia Romano, “Role of biogenic amines as index of freshness in beef meat packed with different biopolymeric materials”, Food Res. Int., 42 (2009) 1147

[3]Jennifer Bruce, Ken Bosnick, Elham Kamali Heidari, “Pd-decorated ZnO nanoflowers as a promising gas sensor for the detection of meat spoilage”, Sens. Actuators B Chem. 355 (2022) 131316

[4]Kissan Mistry, Viet Huong Nguyen, Mohamed Arabi, Khaled H. Ibrahim, Hatameh Asgarimoghaddam, Mustafa Yavuz, David Muñoz-Rojas, Eihab Abdel-Rahman, and Kevin P. Musselman, “Highly Sensitive Self-Actuated Zinc Oxide Resonant Microcantilever Humidity Sensor”, Nano Lett. 22 (2022) 3196

4:40pm **NS+EM+MN-MoA-10 From Natural to Fabricated Gas Sensing Photonic Nanostructures: Unexpected Discoveries and Societal Impact**, ***Baokai Cheng***, *J. Brewer, B. Scherer, R. Potyrailo,* GE Research Center

Existing gas sensors often degrade their performance in complex backgrounds. Thus, new sensing approaches are required with improved sensor selectivity, accuracy, and stability for demanding applications ranging from homeland security, to industrial process monitoring and safety, and to monitoring of outdoor and indoor pollutants and volatile biomarkers.

In this talk, first, we will demonstrate and analyze capabilities of natural photonic nanostructures as sensors for detection of different gases and the origins of these capabilities. Next, we will demonstrate that this new acquired knowledge from studies of natural nanostructures allowed us to develop design rules to fabricate nanostructures for needed gas selectivity and stability for numerous gas monitoring scenarios at room and high temperatures. These design rules for selective gas sensors bring a multivariable perspective for sensing, where selectivity is achieved within a single nanostructured sensing unit, rather than from an array of separate sensors. We fabricated bioinspired nanostructures using several contemporary technologies and have achieved several new functionalities beyond Nature.By utilizing individual nanostructured sensors rather than sensor arrays we also have improved sensor stability by eliminating independent aging factors in separate sensors and their arrays.The use of existing and our new machine learning (a.k.a. multivariate analysis, chemometrics) tools further advanced our sensor designs and performance in detection of multiple gaseous species. The achieved performance capabilities of our developed bio-inspired photonic gas sensors complete with the capabilities of existing commercially available gas sensors and their arrays. These colorimetric sensors can be tuned for numerous gas sensing scenarios in ambient and high temperatures, in confined areas or as individual nodes for distributed monitoring.

5:00pm **NS+EM+MN-MoA-11 Argon-Plasma Dry Etch of sub-Micron Feature-Size Waveguides in Thin-Film Lithium Niobate**, ***Sesha Challa***, *N. Klimov, P. Kuo,* NIST-Gaithersburg

Lithium Niobate (LN) possesses exceptional properties such as a wide transparency range, large non-linear coefficient, and high-electro-optic efficiency. These properties along with successful implementation of periodic poling in LN have spurred the development of devices finding applications in spectroscopy, remote sensing, and quantum communications.

Despite these valuable properties and possessing large second-order non-linearity, LN has taken a backseat to compete integrated photonic platforms, such as silicon, which has no second-order nonlinearity.This was due to difficulties in fabrication of a low-loss LN waveguides (WGs), integration, as well as processing on a wafer-scale.

With commercial TFLN wafers now being accessible, in parallel with the rapid advancement of scalable micro-/nano fabrication techniques, TFLN photonic devices are steadily emerging. By offering tighter conifinement compared to ion-exchanged WGs, TFLN WGs boost the performance of devices such as EOMs to have bandwidth with smaller power consumption.

Etching LN is however challenging, hence, making it difficult to fabricate low-loss WGs. In particular poor etch induces substantial roughness and non-vertical side-wall angles contributing to high propagation losses in the WGs. Low-loss LN WGs have been demonstrated over the past several years. Most of these WGs were fabricated using argon gas inductively coupled reactive ion plasma (ICP-RIE) etching. Etch recipe optimization is of utmost importance to reduce optical losses. Initial demonstrations have shown that TFLN devices can match or exceed the performance of traditional silicon or indium phosphide devices. However, low-loss TFLN waveguides are not widely available.

As a part of the effort to develop low-loss TFLN devices, we perform a systematic study of fabrication high-quality LN WGs. The main goal of this investigation is to reduce the WG's surface roughness while keeping an optimum side-wall angle profile that minimizes light propagation loss. In particular, our efforts are focused on three criteria: (1) selecting appropriate mask materials to reduce the transfer of mask defects into LN WGs, (2) establishing the optimal plasma chemistry by detailed study of various ICP-RIE etch parameters, and, (3) determining the optimum chemical cleaning protocol to remove the redeposited during ICP-RIE etch material on the WG's side-wall. In this presentation, we discuss the details of all three fabrication aspects to make high-quality TFLN devices and structures.

Chemical Analysis and Imaging of Interfaces Focus Topic
Room A105 - Session CA+AS+LS+LX+MN+SE+SS-TuM

Novel Developments and Applications of Interfacial Analysis

Moderators: Andrei Kolmakov, National Institute of Standards and Technology (NIST), **Slavomir Nemsak**, Advanced Light Source, Lawrence Berkeley National Laboratory

8:00am **CA+AS+LS+LX+MN+SE+SS-TuM-1 Hypervelocity Nanoprojectile Impacts on Graphene, Graphene-Solid/Liquid Interphases: From Mechanisms of Interaction/Ejection to Practical Applications**, ***Dmitriy Verkhoturov***, Texas A&M University; *S. Lee,* Mayo Clinic; *M. Eller,* California State University Northridge; *M. Gołuński, S. Hrabar,* Jagiellonian University, Poland; *S. Verkhoturov,* Texas A&M University; *Z. Postawa,* Jagiellonian University, Poland; *A. Kolmakov,* National Institute for Science and Technology (NIST); *A. Revzin,* Mayo Clinic; *E. Schweikert,* Texas A&M University **INVITED**

Presented here are the experiment and theory on processes accompanying the impacts of C_{60} and Au_{400} projectiles (~1 keV/atom) on graphene/matter interphases. A variety of targets were used: a) free standing graphene, b) molecules and extracellular vesicles (EVs) deposited on free standing graphene, c) interphases graphene-solids/liquids, d) EVs deposited on functionalized monocrystals.

Two custom-built Cluster ToF secondary ion mass spectrometry (SIMS) devices with similar parameters were used. The experiments were run in the event-by-event bombardment/detection mode where the regime of bombardment is super-static[1]. The analyzed surfaces were bombarded at the rate of ~1000 impacts/sec with 1-6×10^6 impacts collected on a surface area of 50-500 µm in diameter. This regime allows acquisition of individual mass spectra for each impact, thus allowing the comparison of experimental data with MD simulations at the level of single projectile impacts. The method allows detection of ejecta in reflection (3D case) and transmission (2D case) directions.

The mechanisms of ejection from 2D and 3D materials (including graphene-solid/liquid interphase) are different. For example, in the case of C_{60} impacts on a molecular layer deposited on graphene (2D case) the mechanism of ejection is described with the "trampoline" model[2]. For the 3D case of graphene-solid/liquid interphase, graphene suppresses the ejection of molecules. The compression of matter in the excitation volume around the impact is not sufficient to destroy the graphene[3].

Our method allows to test individual nano-objects. A biological example is EVs. There were anchored on functionalized Si and graphene substrates, with the EVs labeled with antibodies carrying lanthanide tags (Ab@Ln) for normal hepatic and liver cancer markers. Up to four Ab@Ln tags could be detected simultaneously, enabling analysis of population heterogeneity with single EV resolution and to distinguish between normal and cancer EVs based on surface marker expression. Using co-localization of cancer biomarkers, it is possible to find small subpopulation of EVs originating from cancerous cells potentially allowing for early cancer detection. The sensitivity of the method can be increased several folds via transmission configuration where ejecta are emitted and detected in the forward direction. In this case nano-objects, such as EVs, are anchored on graphene oxide, a 2D material.

[1]S.V. Verkhoturov et al. J. Chem. Phys. 150 (2019)

[2]R.D. Rickman et al. Phys. Rev. Lett. 92, 047601 (2004)

[3] D.S. Verkhoturov et al. Biointerphases 11, 02A324 (2016)

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Polish National Science Center 2019/33/B/ST4/01778, PLGrid Infrastructure Grant

8:40am **CA+AS+LS+LX+MN+SE+SS-TuM-3 Applying *in Situ* Bias During TOF-SIMS Analysis to Investigate Ion Migration in Perovskite Devices**, ***Steven Harvey***, National Renewable Energy Laboratory; *I. Gould,* University of Colorado, Boulder; *D. Morales, M. McGehee,* University of Colorado Boulder; *A. Palmstrom,* National Renewable Energy Laboratory

Metal Halide Perovskite Photovoltaics have the potential to be a game-changing technology in photovoltaics, with low cost solution processing inherent to the technology and a rapid progress in device efficiency and stability. Understanding ion migration in these materials has lead to improvements in both efficiency and reliability, and further understanding of these phenomena is of great importance.

Time of flight secondary ion mass spectrometry is well suited to provide unique insight for this class of materials, as it can reveal the distribution of both the organic and inorganic components of a device stack (both through the depth as well as laterally with 2-D and 3-D imaging). We will briefly cover our past work on technique development for this class of materials, before presenting new work where an *in situ* electrical bias was placed on a perovskite device while under investigation with TOF-SIMS. This was completed with simple commercial off the shelf components in an ION-TOF TOF-SIMS V instrument and could be easily implemented on other instruments. A device stack of glass / ITO / Me-4PACz / DMA0.1FA0.6Cs0.3Pb(I0.8Br0.2)3 / LiF (1 nm) / C60 (30 nm) / SnOx (15 nm)/Au (20 nm) was used for this study. An electrical bias was applied between the top gold contact and the bottom ITO contact during TOF-SIMS measurements. By applying a +0.75V and -0.75V forward and reverse bias to the device, a driving force for negatively charged halide ions is created to migrate towards the back or front of the device, respectively. The *in-situ* data shows the halide ion migration towards the back ITO contact after the forward bias is applied. The negative bias was then applied and the halide ions migrate back towards the front of the device and return to the original unbiased state. In both cases the formamidinium and lead traces do not show similar migration, showing only the charged species in the device are affected by the bias. The results show a framework that can be used for further study. Potential complications with the analysis of this type of data will be discussed.

9:00am **CA+AS+LS+LX+MN+SE+SS-TuM-4 Oxidation of a Single Fe Nanoparticle at the Nanoscale and Real-Time by Operando Atom Probe**, ***Sten V. Lambeets***, Pacific Northwest National Laboratory; *N. Cardwell, I. Onyango,* Washington State University; *T. Visart de Bocarmé,* Université libre de Bruxelles, Belgium; *J. McEwen,* Washington State University; *D. Perea,* Pacific Northwest National Laboratory

Physics governing surface chemical reactions and interfaces involved in heterogeneous catalysts fundamentally depends on the synergistic interactions between reactive gases and specific surface structures. Surface science techniques are continuously evolving to help bridge knowledge gaps between fundamental research and real-world applications. In the past decade, an increasing number of analytical techniques successfully achieved their evolution towards an in situ and operando version of themselves, and recently such approaches are being developed for atom probe microscopy (APM) techniques. In this work, we will present the recent advances in the conversion of Atom Probe Tomography (APT) to study surface dynamics of O_2/Fe using two different APM techniques and modifications: Field Ion Microscopy (FIM), and Operando Atom Probe (OAP).

APM techniques are capable of imaging the apex of sharp needles with nanometric lateral resolution, which can be seen as model nanoparticles. FIM is used to image such needles with atomic resolution and to identify the crystal orientation along with the local surface reaction dynamics during oxygen interaction with Fe. The resulting FIM image corresponds to a stereographical projection of the apex and allows the identification of the crystal orientations with atomic resolution. Regular APT, from which the OAP derives, relies on the thermally assisted field evaporation of positively charged ions from a needle shaped specimen. In regular use, the APT is performed in an Ultra High Vacuum (<10^{-11} mbar) while the sample is cooled at 50K. The OAP modification consists of performing the atom probe analysis in the presence of reactive gas at 300 K.

Once the FIM characterization is complete the sample is maintained at 300K before starting APT analysis and introducing 1.1×10^{-7}mbar of pure O_2. As soon as the O_2 is introduced, we can measure the surface formation of Fe oxides by monitoring the local concentration of Fe_2O^{n+} ion species extracted from the surface over time. We can track the local concentration over the different surface regions in real time. We observe the progressive surface oxidation starting from open facets structures, such as Fe{222} and Fe{112}, towards the central Fe(011) and the Fe{024} which show significantly higher resistance toward oxidation. The combination of the different concentrations allows us to reconstruct the full movie of the surface oxidation in real-time. However, since the measurements are

performed in the presence of very strong electric fields (>10 V/nm), it is necessary to discuss the potential influences of it on the system as well.

9:20am **CA+AS+LS+LX+MN+SE+SS-TuM-5 Reporting Interfaces: Unconventional Excitation of Interfaces Enables Exquisite Gas Sensing Toward Our Sustainable Future***, Radislav Potyrailo,* GE Research **INVITED**

As our society is developing solutions for more sustainable types of energy, the need for reliable, yet affordable tools for monitoring of emissions of greenhouse and other gases in urban and industrial environments is a substantial undertaking for two main reasons. First, to achieve a desired accuracy, existing gas monitoring solutions in complex backgrounds utilize traditional analytical instruments. While their mathematical design principles provide needed independent response outputs, their hardware design principles do not allow cost-effective ubiquitous implementations. Second, all gas sensors based on interface-driven interactions between gases of interest and sensing materials are single-output devices. By their original design principles from early last century, these sensors operate well only when levels of interfering gases are low. Once levels of interfering gases increase, existing sensors lose their accuracy because of competing interactions between the sensor interface and numerous interfering gases versus a gas of interest.

In this talk, we will present gas sensors that we built following mathematics of traditional analytical instruments but with our own different types of independent variables for detection of multiple gases with enhanced accuracy and stability. These sensors are multivariable gas sensors where independent response outputs are provided by our unconventional methodologies of excitation of interfaces between a sensing material and different ambient gases. We will show that our approach results in a reliable differentiation of one or more analyte gases in complex backgrounds of interfering gases with an individual multivariable gas sensor. This exquisite (i.e., accurate and reliable) gas sensing provides an affordable technical solution for monitoring of emissions of greenhouse and other gases in urban and industrial environments. Such technical solution is mathematically not feasible using conventional single-output sensor designs. We will also show that such multivariable gas sensors have the ability for self-correction for sensor drift. Our approach for the multi-gas detection and drift self-correction should allow implementations of gas sensors in diverse applications that cannot afford weekly, monthly, or quarterly periodic maintenance, typical of traditional analytical instruments.

11:00am **CA+AS+LS+LX+MN+SE+SS-TuM-10 A "Simple" Approach to Combine Electrochemistry and Operando Near Ambient Pressure XPS Studies***, F. Mirabella, **Paul Dietrich**, A. Thissen,* SPECS Surface Nano Analysis GmbH, Germany **INVITED**

Electrochemical water splitting is an environmentally friendly technology to store renewable energy in the form of chemical fuels. Among the Earth-abundant, first-row transition metal-based catalysts, Ni and Fe oxides have shown promising performances as effective and low-cost catalysts of the oxygen evolution reaction (OER) in alkaline media. Notably, their structure evolves under oxygen evolution operating conditions with respect to the as-prepared catalysts but these changes and consequently the active sites have not been identified yet due to the difficulties associated with surface analysis measurement under working conditions (*operando*).

In this presentation, we will demonstrate the enormous potential of laboratory NAP-XPS for investigations of solid-liquid interfaces in electrochemical systems at elevated pressures (≤ 25 mbar), also illustrating the ease of use of this specific setup. We will show a versatile three-electrodes electrochemical setup that allows for operando studies of solid-electrolyte interfaces, i.e., of nickel oxide foils as cathode for OER in alkaline environment as a simple laboratory NAP XPS experiment.

11:40am **CA+AS+LS+LX+MN+SE+SS-TuM-12 Recent Developments in Probing Buried Interfaces Using Standing-Wave Photoelectron Spectroscopy***, **Slavomir Nemsak,*** Lawrence Berkeley Lab

Standing-wave photoelectron spectroscopy of multi-layer structures proved to be a very powerful technique for probing solid/solid, but also solid/liquid and solid/gas interfaces. Its superior depth selectivity and non-destructive nature were crucial to answer key questions in problems spread over several scientific fields, such as emergent phenomena at complex oxide interfaces [1], artificial multiferroics [2], adsorption mechanisms in liquids [3], corrosion [4], and electrocatalysis [5].These achievements were only possible thanks to innovative approaches both in experiments and analyses, including development of X-ray optical simulations package [6] and its coupling with the black-box optimizer [7]. In this talk I will introduce novel tools and approaches for standing-wave experiments and I will highlight some of the recent applications [8,9,10].

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12:00pm **CA+AS+LS+LX+MN+SE+SS-TuM-13 The Influence of Surface Structure and Electrostatics on Measuring Unoccupied Electronic States via Low Energy Inverse Photoemission Spectroscopy (LEIPS)***, **James Johns,*** Physical Electronics USA

A material's energetic distribution of electronic states near the fermi level is a key physical property for determining how it behaves in electronic, chemical, and optical applications.Photoemission has long been the gold standard for measuring the occupied electronic states below the Fermi level and is one of the most common surface science techniques worldwide.Inverse photoemission (IPES), the related process whereby an electron is absorbed at the surface and a photon is emitted, is similarly a very powerful tool for measuring the unoccupied electronic states. Unfortunately, the intrinsically lower rate for IPES and technical hurdles related to relevant photodetectors has historically necessitated the use of electron sources with sufficient energy to damage all but the most chemically robust surfaces.

The availability of narrow bandpass optical filters at UV photon energies between 3.5 and 6 eV over the past decade have enabled the development and commercialization of Low Energy Inverse Photoemission Spectroscopy (LEIPS)[1,2].Efficient detection of low energy UV photons (lower than traditional IPES at 9-10 eV) enables the use of low energy electrons (below 5 eV) which avoid damaging sensitive materials including organics.This key innovation has revitalized interest in IPES because the technique can now be applied to molecular materials and interfaces relevant to wide range of applications *e.g.* batteries, photovoltaics, organic semiconductors and OLEDs, chemical sensors.Furthermore, optical UV filters also improve the energy resolution, further enhancing the appeal of LEIPS over traditional IPES.

Like any surface science technique, the quality of LEIPS data depends on both the instrumentation and sample preparation. Here, I will discuss the material requirements and limitations for successful LEIPS measurements, several of which differ from more common techniques such as XPS, SPM, or electron microscopy.I will also present LEIPS data from taken at the interface between two metals and explain those results using calculated trajectories of the electron beam.Finally, I will illustrate a key difference between LEIPS, which probes the true unoccupied electronic density of states, and optical methods, such as optical spectroscopy or EELS which measure the joint density of states, by presenting LEIPS spectra of an excitonic 2D material.

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Nanoscale Science and Technology Division
Room B113 - Session NS+2D+EM+MN+SS-TuM

Scanning Probe Microscopy

Moderators: Aubrey Hanbicki, Laboratory for Physical Sciences, **Fernando Castro**, National Physical Laboratory, U.K.

8:00am **NS+2D+EM+MN+SS-TuM-1 AVS Medard W. Welch Award Talk: Microscopy is All You Need: The Rise of Autonomous Science***, Sergei Kalinin*[1], University of Tennessee Knoxville **INVITED**

Making microscopes automated and autonomous is a North Star goal for areas ranging from physics and chemistry to biology and materials science – with the dream applications of discovering structure-property relationships, exploring physics of nanoscale systems, and building matter on nanometer and atomic scales. Over the last several years, increasing attention has been attracted to the use of AI interacting with physical system as a part of active learning – including materials discovery and optimization, chemical synthesis, and physical measurements. For these active learning problems, microscopy arguably represents an ideal model application combining aspects of materials discovery via observation and spectroscopy, physical learning with relatively shallow priors and small number of exogenous variables, and synthesis via controlled interventions. I introduce the concept of the reward-driven experimental workflow planning and discuss how these workflows can be implemented via domain-specific hyper languages. The applications of classical deep learning methods in streaming image analysis are strongly affected by the out of distribution drift effects, and the approaches to minimize though are discussed. The real-time image analysis allows spectroscopic experiments at the predefined features of interest and atomic manipulation and modification with preset policies. I further illustrate ML methods for autonomous discovery, where the microstructural elements maximizing physical response of interest are discovered. Complementarily, I illustrate the development of the autonomous physical discovery in microscopy via the combination of the structured Gaussian process and reinforcement learning, the approach we refer to as hypothesis learning. Here, this approach is used to learn the domain growth laws on a fully autonomous microscope. The future potential of Bayesian active learning for autonomous microscopes is discussed. These concepts and methods can be extended from microscopy to other areas of automated experiment.

8:40am **NS+2D+EM+MN+SS-TuM-3 Dielectric Constant Measurement Sensitivity in Electrostatic Force and Force Gradient Microscopy-Based Modes***, Gheorghe Stan,* National Institute of Standards and Technology (NIST)*; C. Ciobanu,* Colorado School of Mines

Understanding of the nanoscale electrostatic interaction between a conductive atomic force microscopy (AFM) probe and a dielectric film is central to the operation of various nanoscale dielectric microscopies and determination of dielectric properties of the film. There is no simple analytical description of the electrostatic interaction generated in the confined probe-sample geometry of neither the static nor dynamic AFM modes used for dielectric measurements. An accurate description of the involved physics is obtained only by means of a finite element analysis modeling of the system. However, the alternative of using numerical analysis is not very popular being slower and requiring relatively high computation resources. In this work we revised the contributions from different parts of the AFM probe to the probe-sample capacitance by both analytical and numerical methods. We tried to reconciliate the two approaches and observed the differences as a function of geometry and material parameters. Under various noise levels, the efficiency of an analytical model was tested against finite element analysis that captures in detail the electrostatic interaction in AFM-based dielectric measurements. The investigation was performed in both spectroscopic force-distance curves and constant height scans with measurements for the deflection and frequency of the AFM probe. The obtained measurement sensitivities are relevant in selecting the optimal scanning mode and its operational parameters for given film thicknesses and dielectric constants but are also showing the critical role of the numerical analysis to the correct interpretation of the measurements.

9:00am **NS+2D+EM+MN+SS-TuM-4 Measuring and Understanding the Nanomechanical Properties of Halide Perovskites and Their Correlation to Structure***, I. Rosenhek-Goldian,* Dept. of Chemical Research Support, Weizmann Inst. of Science, Israel*; I. Buchine, N. Prathibha Jasti,* Bar-Ilan Inst. for Adv. Mater. and Nanotechnol & Dept. of Chem. Bar-Ilan Univ., Israel*; D. Ceratti,* Dept. of Mol. Chem. & Materials Science, Weizmann Inst. of Science, Israel & CNRS, UMR 9006, IPVF, Institut Photovoltaïque d'Ile-de-France*; S. Kumar,* Bar-Ilan Inst. for Adv. Mater. and Nanotechnol & Dept. of Chem. Bar-Ilan Univ. Ramat Gan Israel. & Dept. of Mol. Chem. & Materials Science, Weizmann Inst. of Science, Israel*; D. Cahen,* Bar-Ilan Inst. for Adv. Mater. and Nanotechnol & Dept. of Chem. Bar-Ilan Univ. & Dept. of Mol. Chem. & Materials Science, Weizmann Inst. of Science, Israel*; **Sidney R. Cohen**,* Dept. of Chemical Research Support, Weizmann Inst. of Science., Israel

Halide perovskites, HaP, and especially Pb-based ones exhibit a plethora of remarkable properties. Of these, their photovoltaic properties are the most widely studied due to the proven potential these materials hold for significant technological impact. In addition to photoresponse, this material class is characterized by interesting physical properties, of which mechanical properties enjoy special attention, not only because of potential use in flexible devices, but also from a fundamental science point of view. The mechanical response can shed light on the materials' behavior including dynamic processes and strain-related effects on optoelectronic behavior.

In the context of these studies, particular emphasis has been placed on environmental factors which can alter, especially degrade, material functionality and device performance. Exposure to humidity, light, and oxygen rank prominently amongst these factors.

In this study we measure the humidity influence on the mechanical properties, i.e., elastic modulus (E) and hardness (H), for two series of lead halide perovskite single crystals, varying either by cation or by anion type. Our conclusions are based on comparing results obtained from several different nano-indentation techniques, which separate surface modulus from that of the bulk, and probe different manifestations of the hardness. These studies reveal the different crystalline parameters governing influence of humidity on the mechanics at the surface and in the bulk.

An atypical inverse correlation between E and H was measured (as seen in the supplementary figure a). Furthermore, humidity influenced these two properties in opposite fashion – humidity exposure led to lower H, but to higher E (supplementary figure b). This trend is opposite to that found in most materials where hydration lowers both E and H. We suggest a link between dynamic disorder, self-healing, and the intriguing relation between E and H.

9:20am **NS+2D+EM+MN+SS-TuM-5 3D Nanoprinting of Advanced AFM Nano-Probes***, Harald Plank, M. Brugger-Hatzl, R. Winkler, L. Seewald,* Graz University of Technology, Austria

The demand for correlative microscopy is still increasing, as it enables a superior ensemble of information by using various methods to combine individual strengths. The highest level of that approach are hybrid microscopes, which enable individual characterization at the very same spot in a consecutive or even parallel way. With that, however, comes the demand of a conflict-free integration of different microscopes, which require a radical redesign of the instrumentation. A major step in that direction is a recently introduced dual system called FUSIONScope, which is a deeply integrated scanning electron microscopy (SEM) and atomic force microscopy (AFM) solution. While the former enables high-resolution guidance towards the region of interest, the latter complements SEM capabilities by true quantitative 3D surface information, which together exploit their full potential by the possibility to precisely land the AFM tip on highly exposed regions. Even more importantly, advanced AFM modes such as conductive AFM (CAFM), magnetic force microscopy (MFM), electrostatic / Kelvin force microscopy (EFM/KFM), scanning thermal microscopy (SThM) or mechanical mapping, provide functional information beyond SEM capabilities. For that, special nano-probes are required, which typically achieve their intended functionality by additional thin film coatings, which contains two main disadvantages. First, they increase the apex radii and limit the lateral resolution, which is in conflict with the still decreasing feature sizes. Secondly, coatings are prone to delamination during operation, which affects resolution, lateral correlation and reliability. Therefore, to exploit the full potential of advanced AFM modes, it is of great interest to develop new approaches for the fabrication of functional nano-probes. Following that motivation, we joined forces with industry and apply the additive direct-write technology focused electron beam induced

[1] **Medard W. Welch Award Winner**

deposition (FEBID) for the development of novel 3D nano-probe concepts with industrial relevance. In this contribution, we briefly discuss the 3D nano-printing process and then go through a variety of advanced, FEBID-based tip concepts for CAFM, EFM, MFM and SThM. The joint element for all probes is the coating-free character, which eliminates the aforementioned risks during operation. Additionally, the apex regions are routinely in the sub-10 nm regime, which allows for high-resolution imaging. Aside of comparisons to traditionally used nano-probes, which reveal the superior performance of FEBID-based nano-tips, we discuss on currently ongoing research towards multi-functional AFM tips, based on FEBIDs flexibility.

11:00am **NS+2D+EM+MN+SS-TuM-10 Chemical, Mechanical, and Morphological Evolution of Nanostructures on the Surfaces of Asphalt Binders**, *L. Lyu, J. Pei,* Chang'an University, China*; E. Fini,* Arizona State University*; L. Poulikakos,* EMPA (Swiss Federal Laboratories for Materials Science and Technology), Switzerland*;* ***Nancy Burnham****,* Worcester Polytechnic Institute

Bitumen (asphalt binder) holds roads together. It is a complex, dynamic, nanostructured material that comes from the bottom of an oil refinery stack—a non-renewable resource. It ages, and it ages more quickly under the influence of heat and light. Can additives made from waste materials increase the longevity of bitumen, and thus roads?

In this study, atomic force microscopy (topography, phase imaging, PF-QNM) and its combination with infrared spectroscopy (AFM-IR) were used to explore the chemical, mechanical, and morphological evolution of the surface of bitumen without and with additives. Aging is assumed to begin at the surface.

Samples of bitumen were made with and without introducing bio-modified rubber additives. Each sample was exposed to several thermal and UV aging protocols. Evolution of surface under aging was studied. Depending on the additive and type of aging (thermal, UV, or combined), the nanostructures changed their chemistry, mechanical properties, and size. Furthermore, the matrices and phases immediately surrounding the nanostructures evolved differently upon aging than the included nanodomains. In general, carbonyl and sulfoxide IR bands became more prevalent, the samples became stiffer and less adhesive, and the phase immediately surrounding the nanostructures became smaller. One additive made from two different waste materials was found to enhance the stability of the surfaces.

By understanding the evolution of asphalt binders and which additives promote their stability, longer lasting roads might be designed and built, thereby lowering the need for a non-renewable resource.

11:20am **NS+2D+EM+MN+SS-TuM-11 Identifying Potential Carbon Sources for Direct Carbon Material Production by AI Assisted HR-AFM**, ***Percy Zahl***, Brookhaven National Laboratory*; Y. Zhang,* ExxonMobil Technology and Engineering Company*; S. Arias,* Brookhaven National Laboratory

High-resolution Atomic Force Microscopy (HR-AFM) has proven to be a valuable and uniquely advantageous tool for studying complex mixtures such as petroleum, biofuels/chemicals, and environmental or extraterrestrial samples. However, the full potential of these challenging and time-consuming experiments has not yet been fully realized. To overcome these bottlenecks and enable further research into solutions for the energy transition and environmental sustainability, automated HR-AFM in conjunction with machine learning and artificial intelligence will be crucial [1].

In this study, we focus on identifying potential carbon sources suitable for more direct carbon material production by analyzing various pitch fractions based on their solubility in toluene. Specifically, we present the first comprehensive AI-assisted study of hydrocarbon fractions derived from petroleum and coal tar pitch, using and refining our previously introduced "Automated HR-AFM" tools. We explored four classes derived from Petroleum Pitch (PP) and Coal Pitch Tar (CPT), separated into toluene soluble (TS) and toluene insoluble (TI) fractions. Our analysis revealed differences in the structural characteristics of the molecules, which we binned based on the number of aromatic rings.

(Please see also the in our supplemental document included figures 1 and 2)

Overall, our results demonstrate the potential of automated HR-AFM and AI-assisted analysis for understanding complex mixtures and identifying potential carbon sources for direct carbon material production. This work represents an important step towards more sustainable and environmentally-friendly energy solutions.

Reference:

[1] Yunlong Zhang, Energy & Fuels 35(18), 14422 (2021)

11:40am **NS+2D+EM+MN+SS-TuM-12 Automated Microscopy for Physics Discovery: From High-Throughput to Hypothesis Learning-Driven Experimentation**, ***Yongtao Liu****, R. Vasudevan, M. Ziatdinov, S. Kalinin,* Oak Ridge National Laboratory

In this work, we explore the ferroelectric polarization switching in relation to the applied pulse bias including bias voltage and time in scanning probe microscopy (SPM). We perform two types of automated and autonomous experiments. First, we conduct automated high-throughput experimentation to gain a comprehensive understanding of the relationship between pulse biases and ferroelectric domain growth. Second, we employ an autonomous experimentation driven by machine learning (ML) algorithm to optimize experimental conditions based on real-time experiment results.

SPM has proven to be a powerful tool for manipulating and visualizing ferroelectric domains at the nanoscale. Investigations of ferroelectric domain size and stability can advance our knowledge of ferroelectrics application in memory devices, such as operating time, retention time, and bit size. However, conventional SPM measurements have been time-intensive and dependent on experienced researchers to perform repetitive tasks and make real-time decisions regarding measurement parameters. For example, researchers determine and manually tune the parameters for next iteration of experiment according to the previous results.Here, we perform automated and autonomous experiments in SPM to explore the mechanism of ferroelectric polarization. The first experiment is a high-throughput experiment of applying various bias pulse conditions to write ferroelectric domains followed by imaging domain structure using piezoresponse force microcopy. In this automated experiment, we systematically adapt the bias pulse parameters to gain a comprehensive understanding of their relationship with the resulting domain structures. We discovered different polarization states that show up upon different bias conditions. In the second experiment, we implement a hypothesis active learning (HypoAL) algorithm based on structured Gaussian process to control the SPM for ferroelectric domain writing. The HypoAL analyzes the relationship between the bias pulse conditions and the written domain size in real-time experiments, and determines the bias pulse parameters for the next iteration. The goal of HypoAL is to establish the best physical hypothesis for the material's behaviour within the smallest number of experiment step.The HypoAL identifies that the domain growth in a $BaTiO_3$ film is governed by kinetic control. The approaches developed here have the potential to be extended to other experiments beyond SPM in the future to accelerate the discovery of new materials and advances in physics.

Nanoscale Science and Technology Division
Room B113 - Session NS1+2D+EM+MN-TuA

Nanofabrication and Characterization of Low-Dimensional Materials

Moderator: Georg Fantner, EPFL

2:20pm **NS1+2D+EM+MN-TuA-1 Atomic-Scale Design and Defect Networks at the 2D/3D Interface**, ***Kate Reidy***, MIT **INVITED**

'Mixed dimensional' 2D/3D van der Waals heterostructures, where 3D metallic nanostructures are integrated with suspended two-dimensional (2D) van der Waals materials, show unique functionalities including light-matter coupling, charge transfer, and enhanced catalytic activity. To enable such integration, an understanding of how structure and defects at the 2D/3D interface affect heterostructure properties is required. Moreover, 2D/3D heterostructures display fluctuations of opto-electronic properties in nanometer spatial range; and it is advantageous to probe position-dependent properties at the same spatial scales. In this seminar, I will share work exploring the local properties of the 2D/3D interface using a combination of atomic resolution scanning transmission electron microscopy (STEM), in situ ultra-high vacuum (UHV) TEM, and monochromated high-energy resolution electron energy-loss spectroscopy (EELS). We demonstrate epitaxial, single-crystalline metallic nanoisland growth of technologically relevant metals (Au, Ti and Nb) with with ultra-low defect density interfaces and facetted morphologies on several thin suspended 2D materials. We then explore the key parameters of 2D/3D growth, including the role of temperature, defects, moiré, surface chemistry, and thermodynamic equilibrium shapes. Lastly, we fabricate more complex heterostructure stacks with defect densities controlled by the compliance of the 2D material substrate. Through fundamental understanding of the structure-property-performance relationship, we suggest that future electronic, magnetic, and optical nanodevices will utilize versatile fabrication of 2D/3D heterostructures with well-characterized interfaces and morphologies.

3:00pm **NS1+2D+EM+MN-TuA-3 Highly Asymmetric Doping of Epitaxial Bilayer Graphene by Targeted Bonding of the Intercalated Gadolinium**, ***Marek Kolmer***, Ames National Laboratory; *J. Hall,* Ames National Laboratory and Department of Physics and Astronomy, Iowa State University; *S. Chen, M. Tringides,* Ames National Laboratory, Department of Physics and Astronomy, Iowa State University

Heterostructures consisting of vertically stacked two-dimensional (2D) materials have recently gained large attention due to their highly controllable electronic properties. Particularly, mechanically stacked multilayered systems offer exceptional control over a stacking sequence or interlayer twist angles. On the other hand, epitaxially grown 2D materials express unprecedented quality and stability over wafer-scale lengths. In both cases controlling the interlayer coupling can generate novel electronic and topological phases and its effective implementation is commonly done with a transverse electric field. However, phases generated by high displacement fields are elusive.

Here, we introduce an exceptionally large displacement field by structural modification of a model system: AB-stacked epitaxial bilayer graphene (BLG) on a SiC(0001) surface. We show that upon intercalation of gadolinium with two specific interlayer locations, electronic states in the top two graphene layers exhibit a significant difference in the on-site potential energy (~1 eV), which effectively breaks the interlayer coupling between them. As a result, for energies close to the corresponding Dirac points, the BLG system behaves like two electronically isolated single graphene layers. We prove this fact by a comprehensive multi-technique methodology based on low-temperature scanning tunneling microscopy/spectroscopy (STM/S) and angle-resolved photoelectron spectroscopy, which are corroborated by density functional theory, tight binding, surface diffraction and multiprobe STM transport. The work presents charge transfer from intercalated metal atoms as a promising approach for the synthesis of 2D graphene heterostructures with electronic phases generated by giant displacement fields.

References

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[2]M. Kolmer, W. Ko, J. Hall, S. Chen, J. Zhang, H. Zhao, L. Ke, C.-Z. Wang, A.-P. Li, M.C. Tringides, "Breaking of Inversion Symmetry and Interlayer Electronic Coupling in Bilayer Graphene Heterostructure by Structural Implementation of High Electric Displacement Fields", J. Phys. Chem. Lett. 13, 11571 (2022). https://doi.org/10.1021/acs.jpclett.2c02407.

4:20pm **NS1+2D+EM+MN-TuA-7 AVS Dorothy M. and Earl S. Hoffman Scholarship Recipient Talk: Exfoliated 2D Nanosheets for Large-Area, Solution-Processed Optoelectronics**, ***Lidia Kuo***[1], *S. Rangnekar, V. Sangwan, M. Hersam,* Northwestern University

Two-dimensional (2D) materials exhibit thickness-dependent optoelectronic properties due to their atomically thin nature, unlike their bulk layered crystalline counterparts. In particular, semiconducting MoS_2 undergoes an indirect to direct bandgap transition as the thickness is decreased to the monolayer limit, leading to enhanced optical absorption and emission at the atomically thin scale. Liquid-phase exfoliation (LPE) is a scalable and cost-effective method for obtaining 2D materials from bulk crystals. However, the yield of monolayer sheets by LPE has been impractically low in previous work. The resulting LPE-processed optoelectronic devices have fallen short compared to nanosheets derived from mechanical exfoliation or chemical vapor deposition. Here, we demonstrate that LPE coupled with megasonic exfoliation – i.e., processing at megahertz frequencies compared to the kilohertz frequencies commonly utilized for LPE – yields an unprecedentedly high fraction of monolayer MoS_2. As a result, megasonic exfoliation enables ultrahigh responsivity in printed MoS_2 photodetectors as well as the first demonstration of electroluminescence for large-area, solution-processed MoS_2 films. This work establishes megasonic exfoliation as a scalable and generalizable approach for achieving optoelectronic-grade 2D semiconductors via LPE.

[1] **AVS Dorothy M. and Earl S. Hoffman Scholarship Recipient**

MEMS and NEMS Technical Group
Room C120-122 - Session MN1-WeM

MEMS Processes, Materials, and Fabrication

Moderators: Benjamín Alemán, University of Oregon, **Jaesung Lee**, University of Central Florida

8:00am **MN1-WeM-1 Additive Manufacturing for 3D Metal Microsystems***, **Robert Roberts**,* The University of Texas at El Paso **INVITED**

Recent developments in precision additive manufacturing technology offer a new dimension in microfabrication design and fabrication. In this talk, the use of additive manufacturing for the realization of three-dimensional metal microsystems is explored via micro laser sintering (MLS). MLS is able to directly fabricate metal features from precursor metal powder with lateral resolutions of 15µm and layer heights of 5µm, making it well suited for the applications of MEMS, BioMEMS, and microsystems packaging.

An overview of the micro laser sintering fabrication process will be detailed, along with developments in new materials for MLS use. Multiple microsystems fabricated via this technology will then be highlighted including microelectrode arrays, high-temperature microfluidic reactors, and electrostatic relays.The use of metal additive manufacturing for the fabrication of metal microsystems packaging is presented. These system-in-packages (SiP) feature embedded microfluidic thermal management structures towards heterogeneous integration microsystems applications with high-power electronics. The additively manufactured devices were able to decrease the thermal resistance to 1.57^{o}C/W, compared to 5.25^{o}C/W for a conventional packages design.

8:40am **MN1-WeM-3 Epitaxial Materials and Devices for High Performance RF Acoustics***, **Vikrant Gokhale**, B. Downey, D. Katzer, M. Hardy, J. Roussos, S. Mack, J. Champlain, A. Lang,* US Naval Research Laboratory*; P. Dhagat, A. Jander,* Oregon State University*; E. Jin,* US naval Research Laboratory*; N. Nepal, V. Wheeler, D. Meyer,* US Naval Research Laboratory **INVITED**

Thin film piezoelectric materials are crucial for creating acoustic micromechanical systems (MEMS) for RF signal processing applications. Conventionally, the metal-piezoelectric-metal transducer materials have been deposited via sputtering, which generally lead to well-textured polycrystalline films. Recent advances in the epitaxial growth of Group III-Nitride (III-N) piezoelectrics, and transition metal nitrides (TMNs) electrodes have enabled all-epitaxial RF acoustic devices with crystalline, lattice-matched and acoustic impedance-matched layers. Such heterostructures are capable of direct integration with GaN RF electronics. In this talk, we shall discuss new opportunities provided by epitaxial RF acoustics, as well as constraints on materials selection, heterostructure growth, and fabrication processes.

Epitaxial RF acoustic devices developed at NRL include surface acoustic wave (SAW) devices, and epitaxial high overtone bulk acoustic wave resonators (epi-HBARs) with a III-N/SiC and III-N/TMN/SiC heterostructure, respectively. Epitaxial SAW devices are used as resonators and delay line filters for low-loss RF signal processing while epi-HBARs are multi-mode phonon cavities with a comb-like RF spectrum capable of extending beyond 40 GHz (Ka band) with extremely low intrinsic loss, which results in record (f×Q) values and long cavity phonon lifetimes [1, 2]. Cryogenic experiments verify that epi-HBARs approach the fundamental limits of intrinsic anharmonic phonon loss [3] and may be well suited for applications such as RF oscillators and in quantum acoustodynamic systems. The near lattice-matched epitaxy of these heterostructures on 4H-SiC results in void-free films with high crystallinity, well-controlled phase and orientation, low defect density, and low surface/interface roughness [1, 4].

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2. *X – Ka Band Epitaxial ScAlN/AlN/NbN/SiC High-Overtone Bulk Acoustic Resonators.* IEEE Electron Device Letters, 2023: p. 1-1.
3. *Temperature evolution of frequency and anharmonic phonon loss for multi-mode epitaxial HBARs.* Applied Physics Letters, 2020. **117**(12): p. 124003.
4. *Control of phase purity in high scandium fraction heteroepitaxial ScAlN grown by molecular beam epitaxy.* Applied Physics Express, 2020. **13**(6): p. 065509.

9:20am **MN1-WeM-5 Slanted Wire Diffraction Gratings Fabricated by Two-Photon Polymerization for Micro-Mechanical Applications***, **V. Paige Stinson**, U. Subash, M. Poutous, T. Hofmann,* University of North Carolina at Charlotte

The use of diffraction gratings in communication and sensing technology is expansive, ranging from their use in integrated optics and spectral analysis to quantum electronics [1]. Their effectiveness in these applications depend on the manipulation of reflected and transmitted efficiencies by the principles of interference and diffraction. Slanted wire gratings are a particularly useful grating geometry as they can be operated at normal incidence. The transmitted diffraction efficiencies of slanted wire gratings are sensitive to changes in slant angle [1]. In this study, this slant angle sensitivity is used to design a mechanically tunable diffractive grating. Motivated by the successful fabrication of infrared optics as well as micro-mechanical structures by two-photon polymerization, a resin compatible for fabrication by this approach (IP-Dip) is selected for modeling [2,3]. A rigorous coupled wave approach is used to determine a geometry which optimizes sensitivity to changes in slant angle. A geometry is selected such that the diffraction efficiency is transferred between the 0th and +1st orders. The efficiency transferred modulates between 0% and 75% for changes in slant angle between 45° to 35°. Realistically, this change in slant angle can be achieved by applying compression to the grating. The mechanical capabilities of the grating are investigated using finite-element method simulations. A prototype slanted wire grating is fabricated by two-photon polymerization and the quality is characterized using scanning electron microscopy. The results of the numerical modeling are presented and potential micro-mechanical applications for their use as transmissive pressure sensors and tunable beam splitters are discussed.

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9:40am **MN1-WeM-6 Very High Frequency Stability of Single-Crystal Silicon Thermal-Piezoresistive Resonators with Phase-Locked Loop***, C. Watkins,* University of Florida, Gainesville*; **Jaesung Lee**,* University of Texas at El Paso*; J. McCandless,* Cornell University*; H. Hall,* Air Force Research Laboratory*; P. Feng,* University of Florida, Gainesville

The Si piezoresistive MEMS heat engine first reported in 2011 [1] marked the start of research to examine the effects of self-sustained thermal-piezoresistive resonators (TPR) in applications such as sensing [2] and signal amplification [3]. TPRs are among a new class of actively transduced devices that offer performance benefits, specifically low motional resistance [4]. Besides, TPRs have the advantage of reduced thermal control needs thanks to the thermal-piezoresistive effect inherent to their operation and thus they could offer very high frequency stability. This paper reports a single-crystalline silicon (Si) TPR achieving very high frequency stability with phase-locked loop (PLL) measurement. A pair of resonators operating in a balanced-bridge configuration is presented, with one device being driven at resonance and the other used to systematically null the undesirable background response. The resonance frequency of the single Si TPR is collected over 40 hours in closed-loop tracking by PLL and yields an Allan deviation of σ_A≈2.66ppb for averaging time of τ≈4.95 seconds - the lowest ever reported among all Si TPRs studied to date. This result is significant because it suggests that such Si TPRs can potentially achieve frequency stabilities comparable to, or better than, existing state-of-the-art resonators used in oscillator circuits, with significantly reduced thermal control requirements (ovenization) and subsequent power demands. In this work we examine the potential impact of utilizing the Si TPR to generate an ultra-stable frequency source. We hypothesize that the intrinsic internal feedback offers enhanced quality (Q) factor which could contribute to a higher frequency stability. Additionally, an external DC power feedback loop

could improve Si TPR stability by removing the linear long-term background frequency drift that is present in all of our PLL measurements.

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MEMS and NEMS Technical Group
Room C120-122 - Session MN2-WeM

Nanomechanics

Moderators: Vikrant Gokhale, US Naval Research Laboratory, **Robert Roberts**, The University of Texas at El Paso

11:00am **MN2-WeM-10 Spatial Mapping and Analysis of Graphene Nanomechanical Resonator Networks***, Benjamín Alemán,* University of Oregon **INVITED**

Networks of nanoelectromechanical (NEMS) resonators are useful analogs for a variety of many-body systems and enable disruptive applications in sensing, phononics, and mechanical information processing. A challenge toward realizing practical NEMS networks is the ability to characterize the constituent resonator building blocks and their coupling. In this work, we demonstrate a scalable optical technique to spatially map graphene NEMS networks and read out the fixed-frequency collective response as a single vector. Using the response vectors, we introduce an efficient algebraic approach to accurately quantify the site-specific elasticity, mass, damping, and coupling parameters of network clusters. In a departure from multiple regression, our algebraic analysis uses just two measured response vectors to fully characterize the network parameters, and does so without any a priori parameter estimates or iterative computation. We apply this suite of techniques to single-resonator and coupled-pair clusters, and find excellent agreement with expected parameter values and broader spectral response. Our approach offers a new, non-regressive means to accurately characterize a range of classical and quantum resonator systems and fills in a vital gap for programming NEMS networks.

11:40am **MN2-WeM-12 Nonlinear Stiffness and Nonlinear Damping in Atomically Thin MoS_2 Nanomechanical Resonators***, Tahmid Kaisar,* University of Florida, Gainesville*; J. Lee,* University of Texas at El Paso*; D. Li, S. W. Shaw,* Florida Institute of Technology*; P. Feng,* University of Florida, Gainesville

Resonant micro/nanoelectromechanical systems (MEMS/NEMS) exhibits rich nonlinear responses because of their relatively small size and high vibration amplitude [1]. In this work, we provide experimental results and a quantitative study of nonlinear dynamics in atomically-thin nanomechanical resonators made of single-layer, bi-layer, and tri-layer (1L, 2L, and 3L) molybdenum disulfide (MoS_2) vibrating drumheads. For these two-dimensional (2D) MoS_2 resonators operating in the very high-frequency band, a synergistic study with calibrated measurements and analytical modeling on the observed nonlinear responses have resulted in nonlinear damping and cubic and quintic order nonlinear stiffness. We find that the *quintic* force can be ~20% of the Duffing force at larger amplitudes, and thus it generally cannot be ignored in a nonlinear dynamics analysis. Though the nonlinear stiffness of 2D NEMS has been studied in literature, to date, there has been no experimental demonstration and investigation of nonlinear damping in 2D semiconductor NEMS resonators. This study provides the first quantification of nonlinear damping and frequency detuning characteristics in 2D semiconductor nanomechanical resonators and elucidates their origins and dependency on engineerable parameters, setting a foundation for future exploration and utilization of the rich nonlinear dynamics in 2D nanomechanical systems.

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MEMS and NEMS Technical Group
Room Oregon Ballroom 203-204 - Session MN-ThP

MEMS/NEMS Poster Session

MN-ThP-1 Ferroelectric and Photovoltaic Properties of $Pb_{0.95}La_{0.05}Zr_{0.54}Ti_{0.46}O_3$ Films as a Function of Film Thickness, ***Sneha Kothapally**, S. Kotru,* The University of Alabama

Ferroelectric materials are being extensively studied for optoelectronic, electrical, and photovoltaic applications. Ferroelectric photovoltaics (FEPV) are gaining research interest as their photovoltaic properties are tunable by controlling the ferroelectric polarization with the applied electric field. However, the low photocurrent obtained from FEPV materials is a significant problem that inhibits their practical applications. The surface quality and the crystalline structure of these films are shown to vary with the film thickness which can influence the photovoltaic response. In this work, ferroelectric $Pb_{0.95}La_{0.05}Zr_{0.54}Ti_{0.46}O_3$ (PLZT) films of different thicknesses were prepared on platinized silicon substrate using the sol-gel deposition technique. As-deposited films were annealed to promote crystallinity. The electrical measurements were taken using a capacitor structure (top electrode/PLZT/Pt), where the top electrodes were sputter deposited. The effect of the thickness of the PLZT layer on microstructure, ferroelectric, photovoltaic, and optical properties was investigated to find the optimal thickness for obtaining the best photo response. Enhanced photovoltaic properties coupled with ferroelectric properties exhibit potential applications for energy conversion in microelectromechanical systems (MEMS), and photovoltaic sensors such as UV sensors.

MN-ThP-2 An In-Situ Reflectometry Parylene Deposition Technique for Highly Accurate and Repeatable Film Thickness and Uniformity, ***Steven Larson**, K. Coombes, A. Mings, J. Norris,* Sandia National Laboratories

Parylene is a chemical vapor deposition (CVD) coating process used for conformal coating, energy harvesting, piezoelectric sensors,acoustic resonators, and many other application. Despite its use in a wide variety of applications, parylene deposition system manufacturers provide minimal methods to control film thickness. While methods have been developed for controlling parylene thickness, their widespread adoption has been limited by scalability, complexity, and cost. In this presentation we report a simple, scalable, and cost effective insitu reflectometry technique for parylene deposition that significantly increases the repeatability and accuracy of deposition thickness. We report film accuracy as a relative standard deviation of 2% as opposed to commercial parylene systems which we measure to a relative standard deviation of 17%. We then improve thickness uniformity with deposition flux modeling and a custom stage with planetary motion. Here we show a significantly improved wafer uniformity of 0.2%.

MN-ThP-3 The Effect of CH_4/H_2 Gas Admixture on the Selectivity Towards Pt in Dry Etching of PZT Thin-Films by ICP-RIE, ***Madeleine Petschnigg**, N. Andrianov, S. Azeem,* Silicon Austria Labs, Austria*; S. Trolier-McKinstry,* The Pennsylvania State University

Due to their high piezoelectric coefficients, ferroelectric thin-films based on lead zirconate titanate (PZT) are among the leading materials for active layers in actuator type microelectromechanical systems (MEMS) such as inkjet print heads, adaptive optics like micro mirrors or autofocus devices, and micromachined acoustic and ultrasonic transducers [1–3]**.**

Well defined patterning of PZT films is crucial for the fabrication of MEMS based on this material. Inductively coupled plasma reactive ion etching (ICP-RIE) allows etching of deep trenches with high aspect ratios and anisotropic etch profiles. While Ti and Zr oxides can be etched chemically by the formation of volatile halides [4,5], the removal of PbO is mainly achieved by physical means, which limits the selectivity against masking and electrode materials. A selective patterning process is especially important for large diameter substrates due to typically radial non-uniformities.

This work illustrates the effect of introducing CH_4/H_2 to the etch chemistry on the selectivity in dry patterning of PZT thin-films via ICP-RIE and discusses the potential chemical mechanism entailed. Figure 1 shows a significant increase in selectivity towards photoresist and Pt upon the addition of CH_4/H_2 to a $CHF_3/Cl_2/BCl_3$ gas mixture wihle the PZT etch rate remains approximately constant up to 25 vol% added CH_4/H_2 as illustrated by Figure 2. This approach is expected to simplify the process flow and increase throughput in industrial PZT patterning by allowing a precise etch stop while maintaining high etch rates and aspect ratios using only a single process.

References

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MN-ThP-4 Nanowatt Chemical Sensor for Unattended Sensing, ***Mieko Hirabayashi**, S. Yen,* Sandia National Laboratories*; O. Faruqe, B. Calhoun,* University of Virginia*; P. Miller, J. Moody,* Sandia National Laboratories

To detect sarin, a potent nerve agent, in unattended applications, we propose a sensor system pairing a sol gel-based transducer with a nanowatt readout circuit. Preliminary measurements demonstrate detection of a sarin surrogate producing a "wake up" signal output while consuming 380 nW of power. This work clears the path to future optimization of the sol gel transducer, use of the multiple channels, and development of the sensor packaging.

Sarin is nerve agent that, when inhaled, induces vomiting and diarrhea, miotic pupils, bradycardia, bronchorrhea, muscle spasms, weakness, flaccid paralysis, seizures, respiratory failure, and tachycardia [1]. For these reasons, sarin gas is notorious as a chemical weapon, and thus its detection at low concentrations is crucial.

Sarin sensors are often carbonnanotube (CNT)-based because this material enables a large surface area and unique electrical properties. These types of sensors are generally sensitive and operate at room temperature [2], but are often fabricated via drop coating, leading to less consistent and less suitable for mass production. By using spray coating of a based catalyzed sol-gel, we are facilitating wafer level sensor fabrication and system integration thereby increasing consistency between fabrication runs and decreasing the overall system size.

With a low power read-out circuit (nano-watt range), we enable unattended, battery-powered detectors for sensing at locations where a wired, full-powered sensor would be difficult to install. Our sensor demonstrates detection of a sarin surrogate using only 380 nW of power. The nW power consumption extends battery life to years for infrequent events [3]. Our results show a single-sensor result (for "waking up" a high-power analysis) with multiple channels for further development for applications and selectivity.

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