

Monday Morning, October 30, 2017

2D Materials Focus Topic

Room 15 - Session 2D+EM+MI+MN-MoM

Properties of 2D Materials including Electronic, Magnetic, Mechanical, Optical, and Thermal Properties

Moderator: Andrey Turchanin, Friedrich Schiller University Jena, Germany

8:20am 2D+EM+MI+MN-MoM-1 Spontaneous Mechanical Buckling in Two-Dimensional Materials: A Power Source for Ambient Vibration Energy Harvesters, *Paul Thibado, P Kumar, S Singh*, University of Arkansas

Internet-of-Things (IoT) is projected to become a multi-trillion-dollar market, but most applications cannot afford replacing batteries on such a large scale, driving the need for battery alternatives.

We recently discovered that freestanding graphene membranes are in perpetual motion when held at room temperature [1-3]. Surprisingly, the random up-down motion of the membrane does not follow classical Brownian motion, but instead is super-diffusive at short times and sub-diffusive at long times. Furthermore, the velocity probability distribution function is non-Gaussian and follows the heavy-tailed Cauchy-Lorentz distribution, consistent with Levy flights.

Molecular dynamics simulations reveal that mechanical buckling is spontaneously occurring, and that this is the mechanism responsible for the anomalous movement. Buckling in this system occurs when the local material suddenly flips from concave to convex.

The higher kinetic energy associated with this motion is derived from the surrounding thermal waste heat, and it may be converted into an electrical current and used as the active component of small power generators known as ambient vibration energy harvesters.

References:

[1] P. Xu, M. Neek-Amal, S.D. Barber, J.K. Schoelz, M.L. Ackerman, P.M. Thibado, A. Sadeghi, and F.M. Peeters, *Nature Comm.* **5**, 3720 (2014).

[2] M. Neek-Amal, P. Xu, J.K. Schoelz, M.L. Ackerman, S.D. Barber, P.M. Thibado, A. Sadeghi, and F.M. Peeters, *Nature Comm.* **5**, 4962 (2014).

[3] M.L. Ackerman, P. Kumar, M. Neek-Amal, P.M. Thibado, F.M. Peeters, and S.P. Singh, *Phys., Rev. Lett.* **117**, 126801 (2016).

8:40am 2D+EM+MI+MN-MoM-2 Topological Toughening of Graphene and other 2D Materials, *Bo Ni*, Brown university; *H Gao*, Brown University

It has been claimed that graphene, with the elastic modulus of 1 TPa and theoretical strength as high as 130 GPa, is the strongest material. However, from an engineering point of view, it is the fracture toughness that determines the actual strength of materials, as crack-like flaws (i.e., cracks, holes, notches, corners, etc.) are inevitable in design, fabrication and operation of practical devices and systems. Recently, it has been demonstrated that graphene has very low fracture toughness, in fact close to that of ideally brittle solids. These findings have raised sharp questions and are calling for efforts to explore effective methods to toughen graphene. Recently, we have been exploring the potential use of topological effects to enhance the fracture toughness of graphene. For example, it has been shown that a sinusoidal graphene containing periodically distributed disclination quadrupoles can achieve a mode I fracture toughness nearly twice that of pristine graphene. Here we report working progresses on further studies of topological toughening of graphene and other 2D materials. A phase field crystal method is adopted to generate the atomic coordinates of material with specific topological patterns. We then perform molecular dynamics simulation of fracture in the designed samples, and observe a variety of toughening mechanisms, including crack tip blunting, crack trapping, ligament bridging, crack deflection and daughter crack initiation and coalescence.

9:00am 2D+EM+MI+MN-MoM-3 Ferroelectric Domain Control of Photoluminescence in Monolayer WS_2 / PZT Hybrid Structures, *Berry Jonker, C Li, K McCreary*, Naval Research Laboratory

Single monolayer transition metal dichalcogenides (TMDs) exhibit exceptionally strong photoluminescence dominated by a combination of distinct neutral and charged exciton contributions. The dielectric screening is very low due to their two-dimensional character relative to bulk material, and their properties are thus strongly affected by their immediate environment. Because the exciton and trion binding energies are very large (~ 600 meV and ~30 meV, respectively), these characteristic emission features persist to room temperature. The samples were fabricated by mechanically transferring large area monolayer WS_2 grown by a CVD process onto 100 nm thick lead zirconium titanate (PZT) films on a conducting *n*-type strontium titanate wafer. We show here that the surface

charge associated with ferroelectric domains patterned into the PZT film with a conductive atomic force microscope laterally control the spatial distribution of neutral and charged exciton populations in the adjacent WS_2 monolayer [1]. This is manifested in the intensity and spectral composition of the photoluminescence measured in air at room temperature from the areas of WS_2 over a ferroelectric domain with polarization dipole pointed either out of the surface plane or into the surface plane. The photoluminescence from areas of the WS_2 over up polarization domains in the PZT are dominated by neutral exciton emission, while those over down domains are dominated by trion emission, consistent with the corresponding charge produced by the domains at the WS_2 / PZT interface. The hysteretic character of ferroelectric materials means that the TMD properties can be selectively reconfigured in a nonvolatile manner by changing the state of the ferroic substrate. This approach enables spatial modulation of TMD properties with a spatial resolution determined by the polarization domains in the underlying ferroelectric layer, with the potential for fabrication of lateral quantum dot arrays or *p-n* junctions in any geometry of choice.

[1] C.H. Li, K.M. McCreary and B.T. Jonker, *ACS Omega* **1**, 1075 (2016).

This work was supported by core programs at NRL and the NRL Nanoscience Institute, and by the Air Force Office of Scientific Research #AOADR 14IOAO18-134141.

9:20am 2D+EM+MI+MN-MoM-4 Mechanical Instability-driven Architecturing of Atomically-thin Materials, *SungWoo Nam*, University of Illinois at Urbana-Champaign

Mechanical deformations, such as buckling, crumpling, wrinkling, collapsing, and delamination, are usually considered threats to mechanical integrity which are to be avoided or reduced in the design of materials and structures. However, if materials systems and applied stresses are carefully controlled, such mechanical instabilities can be tailored to deterministically create functional morphologies that can enable powerful new functions. In particular, in atomically-thin material systems with ultralow bending stiffness, such as graphene, mechanical deformations enable new structural properties and device-level functionalities which surpass the limits of bulk material systems. In this talk, I will present our manufacturing technique on controlled deformation and straining of atomically-thin materials, and the emergent materials properties and applications of such deformed and strained atomically-thin materials. First, I will introduce shrink-manufacturing approaches to enable controlled deformation of atomically-thin materials. Second, I will introduce a wide range of new material properties enabled by the new class of 'architected atomically-thin materials'. I will discuss the surface plasmonics enabled by crumpled topographies of graphene and will further discuss shape reconfigurability which opens the door to tunable plasmonic resonance of crumpled graphene. In addition, I will share our ongoing research efforts on strained superlattice for the modulation of electronic properties. Third and last, I will present our work on adaptive/conformal and multifunctional electronics based on mechanically deformed atomically-thin materials. Our optoelectronic sensor is based exclusively on graphene and transforms the two dimensional material into three dimensional (3D) crumpled structures. This added dimensionality enhances the photoabsorption of graphene by increasing its areal density with a buckled 3D structure, which simultaneously improves device stretchability and furthermore enables strain-tunable photoresponsivity. Our approach to manufacturing architected atomically-thin materials offers a unique avenue for enabling new materials properties and engineering of advanced device functions.

9:40am 2D+EM+MI+MN-MoM-5 Excitons and Exciton Complexes in Transition Metal Dichalcogenide Monolayers, *Mark Hybertsen*, Brookhaven National Laboratory

Ultra-thin semiconductor crystals, realized from transition metal dichalcogenides and other Van der Waals materials, exhibit fascinating optical properties. In the limit of a single monolayer of material, the Coulomb interactions between the optically excited electrons and holes are particularly strong and specifically deviate in functional form from that familiar from bulk semiconductors ($1/\epsilon_r$) [1]. In combination with the reduced dimensionality, the resultant interaction effects are an order of magnitude stronger than those that were previously observed in quantum well structures realized in epitaxially grown multilayers. The lowest energy excitations created by optical excitation are bound electron-hole pairs (excitons). The binding energy is on the 0.5 eV scale and the ladder of bound state energies observed deviate significantly from the spectrum predicted by the conventional hydrogenic model [2]. In the presence of excess carriers, the excitons also form a bound complex with either an

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excess electron or hole (trions) [1]. As the density of optically excited excitons is increased, pairs of bound excitons form (biexcitons), with a clear spectroscopic signature [3]. All of these characteristics of excitons and exciton complexes in transition metal dichalcogenides can be understood directly from the strong and modified form of the Coulomb interaction, including both the role of the environment and the impact of the intrinsic screening response of the material. In particular, a model Hamiltonian can be fully determined from microscopic inputs and solved for the properties of the observed excitons and associated complexes. Variational solutions are semiquantitative while supplying insight. A Monte Carlo approach solves the model Hamiltonian numerically exactly and gives quantitative relationships among the exciton and exciton complex binding energies [4]. Extensions of these approaches to understand excited states in more complex combinations of such layered materials will also be discussed.

Work performed in part at the CFN, which is a U.S. DOE Office of Science Facility, at BNL under Contract No. DE-SC0012704 and with resources from NERSC under Contract No. DE-AC02-05CH11231.

[1] T. C. Berkelbach, M. S. Hybertsen, and D. R. Reichman, Phys. Rev. B **88**, 045318 (2013).

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[3] Y. You, X.-X. Zhang, T. C. Berkelbach, M. S. Hybertsen, D. R. Reichman, and T. F. Heinz, Nat. Phys. **11**, 477 (2015).

[4] M. Z. Mayers, T. C. Berkelbach, M. S. Hybertsen, and D. R. Reichman, Phys. Rev. B **92**, 161404 (2015).

11:00am 2D+EM+MI+MN-MoM-9 Mechanical Properties of Polycrystalline and Defective Graphene, Joseph Gonzales, I Oleynik, J Willman, University of South Florida; R Perriot, Los Alamos National Laboratory

Experimental investigation of mechanical properties indicates that the polycrystalline graphene grown by chemical vapor deposition is as strong as pristine. Recent experiments involving nanoindentation of graphene have also demonstrated counterintuitive increasing of Young's modulus with increasing concentrations of point defects. Using accurate description of interatomic interactions provided by novel screened environment-dependent bond order, (SED-REBO) potential, we performed large-scale molecular dynamics investigations of mechanical properties of polycrystalline and defective graphene samples under conditions mimicking nano-indentation AFM experiments. The atomically resolved characterization of the stress and strain distributions under indenter are used to understand fundamental mechanisms of graphene strength and failure. The breaking strength, the crack initiation and propagation are investigated as a function of the grain boundary structure, grain size distribution, concentration of point defects as well as the position of the indenter in respect to these extended and point defects.

11:20am 2D+EM+MI+MN-MoM-10 Properties of Single Layer Transition Metal Dichalcogenides Grown by Van der Waals Epitaxy, Matthias Batzill, University of South Florida **INVITED**

It is well documented that the electronic properties of transition metal dichalcogenides (TMDs) vary as their dimensions are reduced to a single layer. Also, variations depending on the substrate have been reported. In our studies we grow single- to few- layers of TMDs by molecular beam epitaxy on van der Waals substrates (mainly HOPG or bulk-MoS₂). Despite the weak interactions between the monolayer and the substrate the film grows rotational aligned so that a film exhibits a single crystal orientation. This enables for example electronic structure characterization by angle resolved photoemission spectroscopy. The versatile growth procedure allows us to characterize many materials systems. First we discuss the role of the substrate for semiconducting TMDs. We study the electronics structure variation for MoSe₂ grown on another TMD (MoS₂) and compare it with that grown on HOPG. While the band dispersion of MoSe₂ on HOPG resembles the expectations for free-standing MoSe₂ it is modified for MoSe₂/MoS₂ due to interlayer hybridization of the chalcogen p-orbitals. A big advantage of MBE growth in vacuum is that it enables the synthesis and study of more reactive systems – like most metallic TMDs. Thus, in the second part of this talk we investigate the properties of single layer TiSe₂. TiSe₂ is an unconventional charge density wave (CDW) material whose charge density wave transition has been associated with an excitonic insulator phase. Such an excitonic insulator is formed spontaneously if the excitonic binding energy exceeds the band gap and thus formation of excitons may become the ground state. By scanning tunneling

spectroscopy we observe significant increase in the CDW-band gap opening at the Γ -point for the monolayer compared to few-layer materials. Furthermore, the opening of the gap varies with the substrate material, consistent with expectations for excitonic binding energies. Interestingly, we observe coherence peaks in the tunneling spectra below 50 K suggesting the formation of an excitonic condensate.

MEMS and NEMS Group
Room 24 - Session MN+BI+NS-MoM

Feature Session: Large Scale Integration of Nanosensors

Moderators: Wayne Hiebert, University of Alberta and The National Institute for Nanotechnology, Robert Davis, Brigham Young University

8:20am MN+BI+NS-MoM-1 Large Scale Integration: A Not-so-simple Cure for Loneliness of Silicon Nanoresonators, Sébastien Hentz, Cea Leti, France **INVITED**

After two decades of pioneering work, Nano Electro Mechanical Systems are only starting to fulfil (some) of their huge promises, in particular for sensing. A few start-up companies have been created in the last few years, but NEMS are still far from the industrial success of their micro-counterparts. Among others, one reason is the increasing difficulty to interface the "real-world" quantities to sense with the extremely small size of nanomechanical resonators. An easy to understand example of this is mass sensing: there is huge size mismatch between the NEMS capture cross section (in the μm^2 range) and an actual particle beam size that one can produce (in the mm to 10mm^2 range). Most of the particles to detect are lost. Industrial applications may require the use of large arrays comprising from 10's to 10000's NEMS.

LETI has been working on nanomechanical resonators for a number of applications in the last ten years and have been pioneering their fabrication with Very Large Scale Integration processes. State of the art performance (signal to background ratio, signal to noise ratio, frequency stability...) has been reached with single silicon resonators and specific transduction means adapted to VLSI technologies. The real strength of VLSI though, as evidenced every day by microprocessor fabrication is the possibility to process a large number of devices operating in sync with great reproducibility and control.

We investigated several types of NEMS arrays in the past at LETI. Arrays comprising typically a few 1000 resonators all connected in parallel for gas sensing have been demonstrated. Smaller arrays with the ability to weigh and localize single particles via frequency addressing have been tested too for mass spectrometry applications. LETI has also been pioneering NEMS co-integration with CMOS in the last decade or so and several technologies have been explored. We took advantage of this know-how to fabricate large and dense arrays of NEMS-CMOS arrays for mass sensing applications.

9:00am MN+BI+NS-MoM-3 Nanomechanical Sensors (MSS, AMA) Toward IoT Olfactory Sensor System, Genki Yoshikawa, National Institute for Materials Science, Japan **INVITED**

Owing to their intrinsic versatility, nanomechanical sensors have potential to cover a wide range of olfactory sensing applications in various fields including food, agriculture, medicine, security, and environment. Based on the newly developed platform "Membrane-type Surface stress Sensor (MSS)," we are now trying to realize useful nanomechanical sensor systems which can fulfill the practical requirements, such as portability, low-cost, ease of use, in addition to the basic specifications, e.g. high sensitivity and selectivity. While the MSS provides a practical sensing element, a consumer mobile/IoT sensor system requires further optimization and integration of lots of components including receptor layers, hardware including electronics and sample handling, multidimensional data analysis, and precise calibration for high reproducibility. To establish a de facto standard for odor analysis and sensor systems employing the nanomechanical MSS technology, the "MSS Alliance" was launched jointly with companies and a university. In addition, "Aero-Thermo-Dynamic Mass Analysis (AMA)," which we have recently developed, will provide another approach to characterizing gases by directly measuring molecular weight in ambient condition without a vacuum or ionization. In this talk, the overview of the MSS, AMA, and the related technologies ranging from the optimization scheme of the sensor chip to system level developments will be presented.

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9:40am **MN+BI+NS-MoM-5 Micro-Gas Chromatography Linked with Nano-optomechanical Systems for Breath Analysis**, *Khulud Almutairi*, University of Alberta, Canada; *W Hiebert*, National Institute for Nanotechnology, Canada

One of the applications of microfabrication and nanofabrication technologies is fabricating a micro-Gas Chromatography (GC) on a chip. The miniaturized GC system is designed for the rapid determination of volatile organic compounds (VOCs) that can be used in remote locations with low consumptions and cost of utilization. It was reported that specific VOCs can be found in exhaled breath sample from patients suffering from lung cancer [1]. Therefore, designing a μ GC device can help in separating and analyzing VOCs that comes from exhaled breath samples, such as acetone, benzene and toluene.

Our group has reported that connecting Nano-optomechanical systems (NOMS) to Gas Chromatography can enhance the detection sensitivity limit of VOCs up to 1 ppb [2]. This presentation will feature our first efforts in connecting μ GC with NOMS for higher sensitivity and responsivity. In particular, we will discuss our NOMS sensor chips with microheaters for localized control of sensor temperature. One of our goals is to move toward large scale integration of GC analysis by simultaneously sensing at multiple temperatures.

REFERENCES:

[1] Mazzone, Peter J. "Exhaled breath volatile organic compound biomarkers in lung cancer." *Journal of breath research* 6, no. 2 (2012): 027106.

[2] Venkatasubramanian, Anandram, Vincent TK Sauer, Swapan K. Roy, Mike Xia, David S. Wishart, and Wayne K. Hiebert. "Nano-optomechanical systems for gas chromatography." *Nano Letters* 16, no. 11 (2016): 6975-6981.

10:00am **MN+BI+NS-MoM-6 Micro Chladni Figures and Multimode Manipulation of Breast Cancer Cells in Liquid**, *Hao Jia*, *H Tang*, Case Western Reserve University; *X Liu*, *H Liu*, Northwestern University; *P Feng*, Case Western Reserve University

Non-invasive, microscale positioning of delicate biological cells can foster fundamental research involving probing cellular properties and controlling cellular behaviors and interactions [1-3], which lead to a multitude of applications, such as disease screening, tissue engineering, etc.

Here we demonstrate that microscale manipulation of breast cancer cells can be achieved in a fast and non-invasive manner through exploiting multimode micromechanical systems. We design edge-clamped diaphragm resonators ($\sim 300\mu\text{m}$ in length scale) and piezoelectrically excite their mechanical resonances (within 50–500 kHz) in fluidic environment. The transverse vibrations induce localized, microscale hydrodynamic flow that can aggregate microbeads (3.6 μm -diameter) on device surfaces into a variety of one- and two-dimensional (1D and 2D) 'Chladni figures' [4] (optical images in Fig.1a & b). This phenomenon allows us to further manipulate single or a group of breast cancer cells (MDA-MB-231, 15 μm -diameter), in both 1D and 2D fashions, at a speed of $\sim 4\mu\text{m/s}$ (fluorescent images in Fig. 1a & b). By simply programming the piezoelectric excitation frequency, we achieve dynamic control of cancer cell spatial distributions, switching between mode patterns.

We further demonstrate that such multimode resonator platform can facilitate cellular-level biological studies, such as evaluating cellular adhesive interactions and its connection with cancer biomarker (e.g., CD44). As shown in Fig.2, by exploiting the 'Chladni figure' phenomenon, and carefully selecting 2 resonance modes of a square diaphragm, e.g., Mode (1,1) and Mode (3,3), a controlled number of MDA-MB-231 cells can be quickly manipulated into single cluster and then forced to break as the excitation voltage of Mode (3,3) gradually increases. Cancer cells with CD44 gene knocked out by CRISPR technology are named as CD44⁻ cells, while those with CD44 gene maintained named as CD44⁺ (control) cells. The break of CD44⁻ cell cluster after 0.8V_{pp} in Fig. 2 indicates that they form much weaker adhesive interactions than CD44⁺ cells do, which indicates that CD44 plays a significant role in the metastatic breast cancer cell clustering.

[1] E.E. Hui, *et al.*, PNAS **104**, 2007.

[2] H. Zhang, *et al.*, J. R. Soc. Interface **5**, 2008.

[3] X. Ding, *et al.*, PNAS **109**, 2012.

[4] E.F.F. Chladni, *Entdeckungen über die Theorie des Klanges*, 1787.

10:40am **MN+BI+NS-MoM-8 Microfabrication and Assembly Processes for Integrating Microelectrode Arrays into Tissue-Engineered Scaffolds for Novel Nerve Interfaces**, *Jack Judy*, *C Kuliasha*, *P Rustogji*, *S Natt*, *B Spearman*, *S Mohini*, *J Graham*, *E Atkinson*, *E Nunamaker*, *K Otto*, *C Schmidt*, University of Florida

INVITED

To advance fundamental understanding and develop therapies for neurological disease or injury, microfabricated implantable electrode arrays have been designed and manufactured to stimulate and record neural activity. The materials in these implants, as well as the processes used to integrate them together, must be carefully selected to maximize biocompatibility, device performance, and overall reliability. For upper-limb amputees, nerves are a promising neural-interface target to control sophisticated robotic limbs. Recent advances have shown that nerve stimulation can provide natural sensory feedback. In contrast, it is currently not possible to extract large-scale, high-resolution, and reliable movement-intent signals from nerves. To provide rapid and precise limb control and elicit high-resolution sensory percepts, a nerve interface needs many independent motor and sensory channels. Unfortunately, all existing non-invasive and non-regenerative nerve interfaces grossly under-sample the heterogeneous population of efferent and afferent axons. Although tissue engineering, nerve regeneration, and implantable neural-electronic interfaces are individually well-established fields, the concept of merging these fields to create scalable, and high-performance neural interfaces has not been extensively explored. To overcome the scalability challenge, we present a novel approach. Specifically, we describe a hybrid tissue-engineered electronic nerve interface (TEENI), which consists of multi-electrode polyimide-based "threads" embedded into a biodegradable hydrogel composite scaffold that is sutured to the ends of a transected nerve. Single or multiple thread sets can be incorporated in the hydrogel to enable the TEENI implant to comprehensively engage with the nerve. These polyimide threads will be fully enveloped and held precisely in position during implantation by the hydrogel scaffold, which has properties optimized to reduce foreign-body response. Eventually, the hydrogel will degrade and be replaced with regrown and maturing axons. Since the TEENI approach is scalable to high channel counts over the nerve volume, we believe TEENI nerve interfaces are well positioned to comprehensively capture movement-intent information and impart sensory-feedback information so that upper-limb amputees can get the most out of their prosthetic limbs.

11:20am **MN+BI+NS-MoM-10 Magnetically Actuated Synthetic Cilia for Microfluidics**, *Peter Hesketh*, *S Hanasoge*, *M Ballard*, Georgia Institute of Technology; *M Erickson*, University of Georgia; *A Alexeev*, Georgia Institute of Technology

INVITED

Many bacteria use cilia for swimming, sensing and signal transduction. These functions are achieved by manipulating the fluid around the cilia with continuous and synchronised asymmetric beating patterns. We have fabricated arrays of synthetic cilia using thin film deposition of NiFe thin films. The cilia are able to manipulate fluid in these creeping flow regimes by creating an asymmetry in the forward and recovery strokes. We propose to use artificial cilia in microfluidic devices to perform different functions including mixing, fluid transport, and particle capture.

We use a simple rotating magnet to actuate the cilia array and observe a large asymmetry in the bending pattern of these cilia in the oscillation cycle. We analyze the asymmetric strokes of the cilia by imaging from the side view and quantify the asymmetry between forward and recovery strokes as a function of drive frequency. These asymmetric oscillations are important in creating any microfluidic transport phenomenon such as pumping, mixing and capture in a microchannel as demonstrated in this work. Computational modeling was also used to simulate the motion of the cilia over a broader range of design parameters. We show the dependence of the ciliary performance on several non-dimensional numbers based on the balance of magnetic, viscous and elastic forces acting on the cilia.

The motivation for this work is to improve the quality of sampling for the detection of bacteria and virus in food. Detecting low concentrations of bacteria in food samples is a challenge. The pre-concentration and separation of the target bacteria from the food matrix can be enhanced using improved fluid handling. We demonstrate particle capture with cilia, by functionalizing the surface of the cilia with streptavidin protein and capturing biotin labelled particles on its surface. The functionalized cilia are incorporated inside a microchannel and biotin labelled particles are introduced into array of the cilia. Likewise, these artificial cilia find varied application in many lab on a chip devices where active fluid transport is needed.

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Vacuum Technology Division

Room 7 & 8 - Session VT+MN-MoM

Progress with Measurement in Vacuum

Moderators: Martin Wüest, INFICON Ltd., Liechtenstein, Steve Borichevsky, Applied Materials, Varian Semiconductor Equipment

8:20am **VT+MN-MoM-1 New Vacuum Standard by Ultra-Precise Refractive Index Measurement, Jay Hendricks, J Ricker, J Stone, P Egan, G Scace, K Douglass, D Olson, G Strouse, NIST**

NIST has now completed the 5th year of an Innovations in Measurement Science (IMS) initiative with the aim of developing a new paradigm in the methodology of pressure and vacuum measurement and primary standards. The research program has now successfully developed a new standard that is based on the ultra-precise measurement of gas refractive index. This advance now enables NIST to replace mercury manometer standards with a new quantum-based, photonic technique. The new standard, is based on the fundamental physics of light interacting with a gas, and when the gas is helium, the refractive index is based upon first principle quantum chemistry calculations and is realized as a primary standard. For the vacuum community, a photonic realization of the pascal represents a fundamental change in how the unit of pressure is realized in that it will be directly related to the density of a gas by the temperature, refractive index, and Boltzmann constant. The photonic technique has now achieved important benchmarks in performance when compared to the existing primary standards based on mercury manometers: The photonic technique has a 20x smaller footprint, 100x faster sensing response time, extended to 100x lower pressure, a tenth of a mPa resolution over the full range, and has demonstrated impressive accuracy, reproducibility and hysteresis for an emerging technique [1]. Data will be presented that shows this technique has now reached or surpassed mercury manometer performance which creates a new paradigm for vacuum metrology and realization of the SI unit, the pascal. Future NIST work will explore improvements that will enable the device to become a portable pressure and vacuum standard for international key comparisons in pressure and vacuum metrology.

[1] Comparison measurements of low-pressure between a laser refractometer and ultrasonic manometer, Review of Scientific Instruments, Volume 87, May 2016

Accepted: May 2016, Issue 5, 10.1063/1.4949504

8:40am **VT+MN-MoM-2 Construction and Testing of the NIST Variable Length Optical Cavity Pressure Standard, Jacob Ricker, J Hendricks, G Scace, P Egan, J Stone, NIST**

NIST is constructing and testing a new refractometer, referred to as the Variable length optical cavity (VLOC), that will redefine how pressure and vacuum is measured. NIST has shown in previous talks and papers that this technique will replace all mercury manometers in the near future. However, the critical final piece of this project is to base the traceability of pressure measurements to fundamental constants of the universe and not on the physical artifacts like mercury density in a manometer. Theoretical quantum mechanics have been used to precisely calculate the refractivity ($n-1$) of a gas. NIST will experimentally verify these calculations and provide experimental measurements of refractivity for other gasses/mixtures.

The engineering of the VLOC will be discussed along with limitations and technical complications that have arisen. Specifically, the distortions of the optical cavities and methods to overcome these limitations. Additionally, the steps required to maintain ultra-high purity gas will be discussed. Finally, the testing and final steps to achieve full operation will be discussed and the relation to the 2018 redefinition of the Boltzmann constant.

9:00am **VT+MN-MoM-3 Fast-Switching Dual Fabry-Perot Cavity-based Optical Refractometry – A Powerful Technique for Drift-Free Assessment of Gas Refractivity and Density, Ove Axner, I Silander, T Hausmaninger, Umeå University, Sweden; M Zelan, RISE Research Institutes of Sweden, Sweden**

INVITED

Since pressure has a temperature dependence it is not trivial to accurately assess gas amounts by pressuring measuring devices. However, the (number) density does not suffer from such limitations. Optical Refractometry (OR) is a powerful technique for assessment of gas refractivity and density. The highest resolution is obtained when performed in a Fabry-Perot (FP) cavity. In FP-Cavity based OR (FPC-OR) the change in the frequency of laser light, locked to a longitudinal mode of a FP cavity, is monitored while the amount of gas in the cavity is being changed. Since frequency is an entity that can be assessed with enormous precision, the

precision of FPC-OR can be extremely high. However, although potentially very powerful, FPC-OR is often limited by thermal deformation of the spacer between the mirrors. A partial remedy to this is to use two FP cavities, termed Dual FPC-OR (DFPC-OR).

We have prophesied that if measurements could be done under drift free conditions, the technique would be able to circumvent most of these limitations. A possible strategy for drift-free DFPC-OR, termed fast switching DFPC-OR (FS-DFPC-OR), is presented in which measurements are made under such short times that the drifts of the cavity can be disregarded. The methodologies developed circumvent the problem with volumetric expansion, i.e. that the gas density decreases when gas is let into the measurement cavity by performing a pair of measurements in rapid succession; the first one assesses the density of the gas transferred into the measurement cavity by the gas equilibration process, while the second automatically calibrates the system with respect to the ratio of the volumes of the measurement cavity and the external compartment. The methodologies for assessments of leak rates comprise triple cavity evacuation assessments, comprising two measurements performed in rapid succession, supplemented by a third measurement a given time thereafter.

We predict that refractivity and density can be assessed, under STP conditions, with a precision in the 10^{-9} range. The absolute accuracy is expected to be given by the calibration source. If characterized with respect to an internal standard, the accuracy can be several orders of magnitude better. The temperature dependence of FS-DFPC-OR is exceptionally small, typically in the $10^{-8} - 10^{-7}/^{\circ}\text{C}$ range, primarily caused by thermal expansion of the spacer material.

A first realization of a FS-DFPC-OR set up for assessments of gas refractivity and density will be presented and its performance will be demonstrated. We will discuss how to design an FS-DFPC-OR system for optimal performance for assessments of gas refractivity and density.

9:40am **VT+MN-MoM-5 Cold Cathode Gauge Improvements Extend Performance into UHV Pressure Range, Timothy Swinney, G Brucker, MKS Instruments, Inc., Pressure and Vacuum Measurement Group**

Cold cathode gauges (CCG) of inverted magnetron design are routinely used to measure pressure in industrial high vacuum chambers. Reduced internal outgassing, compared to hot cathode gauges, also makes CCGs well suited for accurate ultra-high vacuum (UHV) measurement in applications such as high-energy physics, surface science experiments and ultrahigh resolution mass spectrometers. In order to provide accurate and repeatable pressure measurements extending into deeper UHV levels, it is important to design CCGs that provide a consistent linear response to pressure over the entire measurement range. Our latest research efforts have focused on the understanding of gauge signal response to pressure with particular emphasis on the displacement of the magnetron knee and discharge sustain issues to lower pressures through systematic design changes. In this presentation, the linear response of CCGs to pressure is explained based on a simple pure electron plasma model. Pressure readings below the magnetron knee are described in terms of a pressure-dependent plasma model controlled by design parameters. The effect of magnetic strength, electric field and plasma boundary conditions on the onset of the magnetron knee and the ability to sustain a stable discharge into UHV levels is described. A patent-pending modification to the CCG internal electrode design is presented that extends the operational pressure of the gauge into deeper UHV levels by controlling the location of the pure electron plasma inside the ionization volume. This new understanding of CCG signal response to pressure has led to the development of enhanced sensor designs that operate at pressures one to two decades lower than legacy designs.

10:00am **VT+MN-MoM-6 Sapphire MEMS based Capacitance Diaphragm Vacuum Gauge for 0-0.1Torr Operating at 200 °C, Takuya Ishihara, Azbil Corporation, Japan; M Sekine, M Soeda, M Nagata, Azbil Corporation**

To meet with downsizing of semiconductor device, various new manufacturing processes such as Atomic Layer Deposition (ALD) and Atomic Layer Etching (ALE), are put into practical use. In particular, ALE is a new atomic level etching technique, which can be applied to high aspect ratio structure or narrow slit. Conventionally, the pressure range of capacitance manometer for etching process is mainly 0-0.1Torr for reasons such as using inductively coupled plasma. And self-heating temperature of that is usually 45 °C, or at most 100 °C for the stabilization.

In this paper, authors have assumed that towards ALE process enhancement, etching would require high temperature process operation, such like 200 °C to prevent by-product from depositing inside of

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manometer in deposition step. Therefore, there is a motivation to develop capacitance manometer with its pressure range of 0-0.1Torr operating at 150-200 °C.

Entirely sapphire-based capacitive pressure sensor chips utilizing MEMS (Micro-Electro-Mechanical Systems) processes, which can be operated at 200 °C with from 0-1 to 0-1000 Torr pressure range have been developed by authors (Fig.1). To diminish pressure range to 0-0.1Torr, we need to reduce sensor diaphragm thickness to get sufficient sensitivity, but thinner diaphragm would be influenced heavily by noises, such like vibration from vacuum pumps, diaphragm sticking, and mechanical stress from sensor package and so on.

One of the critical issue is the zero point drift which was observed under back ground vacuum level after applied pressure over 100 °C (Graph.1). As a result of various verification experiments, this phenomenon was proved to be caused by the slight difference of temperature between sensor diaphragm surface and dilute gas in back ground vacuum. In other words, thermal energy exchange between diaphragm and gas results local expansion or shrinkage of the diaphragm because of its thinner thickness, which deform diaphragm (Fig.2). In our thermal simulation like Fig.3, only 0.05 °C temperature deference causes 0.5% Full Scale zero point drift at 0.1Torr range, which is fatal for the monitoring or controlling of the process. The temperature of the background gas depends on the temperature of inside wall of the process chamber which cannot be controlled by capacitance manometer itself. To solve this problem we have developed new sensor chip structure utilizing sapphire MEMS technology in which the process gas exchanges thermal energy with sensor chip before arriving to the diaphragm (Fig.4). By this new sensor chip, the zero point drift was suppressed to under 0.1% Full Scale, which is sufficient value to apply for the processes (Graph.1).

10:40am **VT+MN-MoM-8 ROSINA/Rosetta: Exploring the Origin of our Solar System with Mass Spectrometry in Space, Kathrin Altwegg, University of Bern, Switzerland**
INVITED

On 30 September 2016 the European Space Agency's Rosetta spacecraft softly crash-landed on comet 67P/Churyumov-Gerasimenko and brought an intense period of more than 2 years of continuous investigation to an end. Rosetta data led to many discoveries about the origin of the material and the processing in our early Solar System. Among the payload instruments, ROSINA, the mass spectrometer suite, obtained fundamental properties of the comet by measuring the gases emanating from its nucleus.

Besides detecting many organic molecules never seen in space before, ROSINA was also able to measure precise isotopic abundances for noble gases, sulfur and silicon as well as D/H in water and H₂S. By following the comet from 3.8 AU to perihelion and out again to 3.8 AU desorption patterns could be followed for individual species, allowing deeper insights into the nature of cometary ice. Some of the findings clearly point to unprocessed ice from the prestellar stage which allows to study chemistry in the presolar cloud more or less "in situ".

Some of the most important findings will be discussed in the presentation like the "zoo" of volatile and semi-volatile organics, the isotopic signature of Xenon and its relation with the terrestrial atmosphere.

11:20am **VT+MN-MoM-10 Stabilization of Emission Current from Cold Field Emitters by Reducing Pressure to 10⁻¹⁰ Pa, Keigo Kasuya, T Ohshima, S Katagiri, T Kawasaki, Hitachi, Ltd., Japan**

In the presence of a strong electric field, electrons are emitted from sharply pointed cathodes at room temperature. This cold field emission (CFE) process provides a prominent electron beam with high brightness and a low energy spread, so CFE emitters are used extensively in a variety of electron microscopes.

One of the important challenges for CFE is stabilizing the emission current. The adsorption of residual gases on the emitter increases the surface work function and decreases the emission current over time. Additionally, surface sputtering by ions causes irregular current fluctuations.

One way to stabilize emission current is to reduce the pressure around the electron gun. This decreases incident gases and ions hitting the emitter and slows the current decrease. We reduced the pressure of an electron gun from 10⁻⁸ to 10⁻¹⁰ Pa by using non-evaporative getter (NEG) pumps [1]. This stabilized the emission current so that it was almost constant over a 24 hour period. The 90% decrease time, the time it takes the current to fall to 90% of the initial value, was increased from 10 minutes to 1280 minutes. In addition, the maximum emission current was increased from 30 μA to 1000

μA. With this gun, operators can use electron microscopes without the need for emission current adjustment.

By applying this technology, we developed a 1.2 MV high voltage transmission electron microscope [2]. The electron gun is equipped with a preaccelerator magnetic lens for enhancing the effective brightness of the electron beam. The pressure of the gun was 3X10⁻¹⁰ Pa, and a stable emission current was obtained. The microscope achieved the world's highest spatial resolution of 43 pm.

Part of this research was funded by a grant from the Japan Society for the Promotion of Science (JSPS) through the "Funding Program for World-Leading Innovative R&D on Science and Technology (FIRST Program)," initiated by the Council for Science and Technology Policy (CSTP).

[1] K. Kasuya et al., J. Vac. Sci. Technol. B, 34, 042202 (2016).

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11:40am **VT+MN-MoM-11 Measurement and Prediction of Quadrupole Mass Spectrometer Sensitivities, Robert Ellefson, REVac Consulting**

Accurate analysis of partial pressure and gas composition by quadrupole mass spectrometry (QMS) requires measuring the QMS sensitivities and fragmentation factors for gas species of interest. The sensitivity is the ratio of ion current for the species to the partial pressure of that species. Fragmentation factors are ratios of fragment [#] ions to the parent ion and are used to correct for species interference at the fragment mass. Measurement with pure gases of each species is the traditional method for determining sensitivities and fragmentation factors; this involves a significant investment in gases and delivery hardware. The QMS ion source operates in the molecular flow regime so that for each species, gas flow is independent of other components present. This allows known mixtures of species to be used for independently measuring multiple species sensitivities. Data using two designed mixtures are presented giving sensitivities and fragmentation ratios for nine species: H₂, He, H₂O, N₂, O₂, Ar, CO₂, Kr and Xe. Gases are delivered to the QMS with a molecular flow inlet system which delivers a broad range of predictable partial pressures for species. Sensitivity versus partial pressure determines the range of linear operation of the QMS indicated by constant sensitivity for that species over a range of pressure. A model for predicting QMS sensitivities for species not measured is also presented. The model uses the QMS sensitivities for the known gas species analyzed to determine the parameters for predicting the sensitivity of an unknown. The factors of the model are: 1. A calculated [#] ionization cross section as a function of incident electron energy data from the NIST Web Book, 2. The mass spectrum of the gas from the Web Book, 3. A model for ion transmission as a function of mass for the QMS in use, and, 4. The QMS sensitivity for N₂ as a reference point to capture the ion source geometry and unique behavior of the QMS under test. Examples of the predictive method and estimated uncertainty are given.

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Room 24 - Session MN+EM+NS-MoA

Nano Optomechanical Systems/Multiscale Nanomanufacturing

Moderators: Robert Ilic, National Institute of Standards and Technology (NIST), Meredith Metzler, University of Pennsylvania

1:40pm **MN+EM+NS-MoA-1 GHz Integrated Acousto-Optics, Mo Li, University of Minnesota** **INVITED**

Integrating nanoscale electromechanical transducers and nanophotonic devices potentially can enable new acousto-optic devices to reach unprecedented high frequencies and modulation efficiency. We demonstrate acousto-optic modulation of a photonic crystal nanocavity using acoustic waves with frequency up to 20 GHz, reaching the microwave K band. Both the acoustic and photonic devices are fabricated in piezoelectric aluminum nitride thin films. Excitation of acoustic waves is achieved with interdigital transducers with periods as small as 300 nm. Confining both acoustic wave and optical wave within the thickness of the membrane leads to improved acousto-optic modulation efficiency in the new devices than that obtained in the previous surface acoustic wave devices. In a photon-phonon waveguide, we further demonstrate strong Brillouin scattering of light by electromechanically excited acoustic waves. Our system demonstrates a novel scalable optomechanical platform where strong acousto-optic coupling between cavity-confined or guided photons and high frequency traveling phonons can be explored.

2:20pm **MN+EM+NS-MoA-3 Coupling Piezoelectric MEMS to Cavity Optomechanics, Kartik Srinivasan, NIST** **INVITED**

Establishing a link between the radio frequency (RF) and optical domains is a topic of relevance to a variety of applications in communications, metrology, and photonic quantum information science. Acoustic wave devices represent an opportunity to mediate such transduction in a chip-integrated format. The approach we are pursuing uses materials that are both piezoelectric, to couple RF waves to strain fields, and photoelastic, to couple strain fields to optical waves.

One architecture that we have recently explored is based on exploiting these effects in GaAs. First, interdigitated transducers (IDTs) convert 2.4 GHz RF photons into 2.4 GHz propagating surface acoustic waves. These acoustic waves are routed through phononic crystal waveguides and are coupled to a nanobeam optomechanical cavity that supports both a highly localized 2.4 GHz breathing mechanical mode and a high quality factor 1550 nm optical mode. In contrast to non-resonant excitation of photonic structures with IDTs, here the phononic waveguide preferentially excites a localized mechanical mode, which in turn strongly interacts with the optical mode through the photoelastic effect. Finally, the optical mode can be out-coupled or excited via an optical fiber taper waveguide. Using this platform, we demonstrate preparation of the breathing mode in a coherent state at any location in phase space, and optically read out an average coherent intracavity phonon number as small as one-twentieth of a phonon. In the time-domain, we show that RF pulses are mapped to optical pulses, forming a resonant acousto-optic modulator with a sub-Volt half-wave voltage. We also observe a novel acoustic wave interference effect in which RF-driven motion is completely cancelled by optically-driven motion, enabling the demonstration of interferometric opto-acoustic modulation in which acoustic wave propagation is gated by optical pulses.

While the above platform has been shown to provide a coherent interface between the RF, optical, and acoustic domains, the overall efficiency is limited by imperfect matching across the various interfaces, e.g., IDT-to-phononic crystal waveguide, etc. In the final part of my talk, I will outline efforts to improve upon the transduction efficiency of the system.

3:00pm **MN+EM+NS-MoA-5 Collective Nano-optomechanics for Sensing Applications, Eduardo Gil Santos, W Hease, Universite Paris Diderot, France; A Lemaitre, Centre de Nanosciences et Nanotechnologies, France; M Labousse, C Ciuti, G Leo, I Favero, Universite Paris Diderot, France**

Optomechanical resonators have been the subject of extensive research in a variety of fields, such as sensing, communication and quantum technologies. Our recent investigations on the capabilities of optomechanical semiconductor disk resonators to operate as sensors in liquids have revealed an astonishing potential. Minimum mass detection of $14 \cdot 10^{-24}$ g, density changes of $2 \cdot 10^{-7}$ kg/m³ and viscosity changes of $5 \cdot 10^{-9}$

Pa-s, for 1s integration time, are extrapolated from our measurements in liquids.

After landmark experiments realized on single resonators, the use of multiple optomechanical cavities is essential to further improve their sensing capabilities, as it enlarges the sensing area while keeping their individual assets. This evolution towards collective nano-optomechanics hence bears potential for a variety of sensing applications, but for quantum or topological photonics as well. Unfortunately, the collective configurations are usually impeded by the residual disorder imposed by nanofabrication techniques, which naturally detunes high optical Q resonators and precludes resonant interactions between them. Therefore, overcoming fabrication imperfections and allowing spectral alignment of resonators is essential.

Here, we develop a new simple and scalable post-fabrication method to achieve such alignment in a permanent manner. The method introduces an approach of cavity-enhanced photoelectrochemical (PEC) etching in a fluid (gas or liquid). This resonant process is highly selective and allows controlling the resonator size with sub-pm precision, well below the material's interatomic distance. Light resonantly injected into the optical mode of an optical resonator immersed in a fluid triggers an etching process, leading to a fine-tuning of the resonator's dimensions. The evolution of dimensions is monitored continuously by tracking the resonator's optical resonance with a laser. This tuning process is naturally scalable to multiple resonators. We demonstrate it using a cascaded configuration where optomechanical disk resonators, each supporting its own localized optical and mechanical mode, are unidirectionally coupled through a common optical waveguide. The technique is illustrated by finely aligning up to five resonators in liquid and two in air, opening the way of fabricating large networks of identical resonators.

As an example of application of this tuning technique, we explore the resonant optical interaction of multiple nano-optomechanical systems. We observe a first form of collective behavior involving several distant resonators, where a unidirectional flow of light frequency-locks a chain of nano-optomechanical oscillators.

3:20pm **MN+EM+NS-MoA-6 Microporous Nanophotonic Mechanical Cantilevers for Mass Sensing, Anandram Venkatasubramanian, V Sauer, J Westwood-Bachman, University of Alberta and The National Institute for Nanotechnology, Canada; K Cui, S Roy, M Xia, National Institute for Nanotechnology, National Research Council, Canada; D Wishart, University of Alberta, Canada; W Hiebert, University of Alberta and The National Institute for Nanotechnology, Canada**

The Gas chromatography (GC) – Mass spectrometry (MS) system is the industry benchmark in chemical analysis. However the large size of the Mass spectrometry unit makes it unsuitable for portable applications. Hence a portable universal mass sensing device that can be used with portable GCs needs to be developed. In this regard, recent demonstration with nano opto-mechanical system (NOMS) devices in conjunction with a GC system have proven that these kinds of sensors have the breakthrough potential to improve the sensitivity of portable GCs. Those demonstrations using NOMS devices have shown these sensors to match the mass detection limits of nanoelectromechanical systems (NEMS) sensors and can potentially better their performance owing to their superior displacement sensitivity compared to NEMS.

In this regard, a free space interferometry system based nanophotonic sensor was developed and attached to a conventional GC. The nanophotonic sensor consists of a microring racetrack resonator (for concentration sensing) with a nanomechanical beam (for mass sensing) adjacent to it. Common method to improve the sensitivity of a nanomechanical beam is to apply surface coatings. However, the application of surface coatings can potentially affect its universal sensing characteristics. Hence an alternate way to improve the adsorption sensitivity is to increase the surface area of the nanomechanical sensor to aid in increasing the number of gas adsorption sites.

In this paper we increase the surface porosity of nanomechanical beam by stain etching. Care was taken to protect the adjacent microring resonator from stain etching as surface pores can negatively affect the performance of the ring resonator due to increased scattering. The stain etching was conducted using vanadium oxide/Hydrofluoric acid based chemistry to etch ~ 10nm pores of random morphology on the surface. Based on an estimated porosity of <15% by volume, we have noted an increase in mass adsorption of $\geq 50 - 100\%$ when tests were conducted using different volatile organic compounds. In other words, a mass adsorption enhancement factor of 1.5 to 2 has been achieved. Due to this enhanced

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adsorption, the mass detection threshold has improved by an order of magnitude ($\sim 10^{-19}$ g). To the best knowledge of the authors, this is the first time NOMS based porous nanomechanical mass sensor has been developed.

Mass Adsorption Enhancement Factor = Adsorption frequency shift for porous beam/Adsorption frequency shift for non porous beam

4:00pm MN+EM+NS-MoA-8 Tunable Resistivity in Inkjet Printed Circuit by Plasma Reduction of Particle-free, Stabilizer-free Ink, *Y Sui, S Ghosh, C Miller, M Sankaran, Christian Zorman*, Case Western Reserve University

Inkjet printing offers a low-cost, rapid methodology to produce patterned metal thin films on flexible substrates. The most commonly used ink consists of colloidal suspensions of nanoparticles prepared by wet chemical reduction of metal salts. Even after concentrating the nanoparticles through solution processing, the as-printed ink usually exhibits a low conductivity due to the presence of organic molecules that help stabilize the nanoparticles from agglomeration and precipitation. High temperature (>200 C) treatment is then required after printing to remove the insulating organics and sinter the nanoparticles. The thermal step can limit printing on polymers such as PDMS, paper, and 3D printed polymers.

Here, we present a particle-free, stabilizer-free ink and a low-temperature plasma reduction process to produce electrically conductive metallic patterns on temperature-sensitive without any additional thermal step. The ink is comprised of a metal salt, a solvent, and a viscosity modifier, and is absent of any large organic molecules that cannot be evaporated after printing. The as-printed and dried metal salt is then treated with a plasma formed in a low-pressure argon environment. Even without the presence of highly reactive atomic and molecular hydrogen, this process is found to be sufficient to reduce the metal salt to highly conductive metal with resistivities approaching bulk values. More importantly, we found the resistivity of the printed structure can be tuned over a range of 2 orders of magnitude by varying the plasma power and treatment time. Thus far, we have demonstrated this general approach for silver (Ag) and tin (Sn) from silver nitrate (AgNO_3) and tin (II) chloride (SnCl_2), respectively. Details of the material properties as assessed by materials characterization and electrical conductivity measurements, device application to RC filter circuits, and applicability to other metals will also be discussed.

4:20pm MN+EM+NS-MoA-9 Cold Forming of Shallow Spherical Micro Caps by Nano Imprinting, *Asaf Asher, E Benjamin, L Medina, S Lulinsky*, Tel Aviv University, Israel; *R Gilat*, Ariel University, Israel; *S Krylov*, Tel Aviv University, Israel

Many nonlinear systems are distinguished by bistability, which manifests itself as the coexistence of two equilibria under the same loading. Progress in the development of micro and nano structures stimulated renewed interest in the mechanics of bistable elements. Applications of these devices in the realm of micro and nanoelectromechanical systems (MEMS/NEMS) include switches, sensors, non-volatile memories, micro-pumps, micro-resonators and deformable mirrors. While curved beam-type bistable micro structures were intensively investigated both theoretically and experimentally much less attention was paid to two-dimensional bistable structures such as initially curved plates and shells (caps). One of the reasons is that lithography-based processes commonly used in MEMS/NEMS are essentially planar and are not suitable for fabrication of cap-like structures with an out-of-plane curvature. Existing approaches include gray-scale lithography or a glass blowing technique.

In this work we discuss several approaches for fabrication of shallow micro shells. One of the directions is the use of the cold forming techniques, when stamping changes a flat thin foil of ductile material into double-curvature components. While multiple variations of this process were used at the macro scale, much less works reported the implementation of this technique in MEMS. In the framework of the fabrication process used in our work, a layer of Al or Cu is deposited on top of a Si wafer. Sputtering is implemented for the creation of a thin seed layer, followed by electrodeposition used to increase the layer thickness to a desired value. Next, an opening is etched through the wafer using deep reactive ion etching (DRIE). Finally, the forming process is performed using nano-imprinting lithography tool. The tool allows very precise control of the force applied to the structure as well as the stamp temperature, displacements and rate of loading. Finite elements analysis and compact reduced order models are used for the evaluation of the desired parameters. Prior to forming, residual stress of the thin suspended membranes is estimated using a resonance method, by means of comparison of the measured natural frequencies of the device with the model predictions. We discuss suitability of different structural materials,

deposition methods and stamping techniques for the formation of non-planar three-dimensional micro structures.

4:40pm MN+EM+NS-MoA-10 Plate Mechanical Metamaterials: The Thinnest Plates You Can Pick Up by Hand, *Igor Bargatin*, University of Pennsylvania

INVITED

Recently, my group has demonstrated a new class of ultra-lightweight plate-shaped mechanical metamaterials, which we named "plate mechanical metamaterials". Using a periodic three-dimensional patterning, we fabricated free-standing plates up to 1 cm in size out of aluminum oxide (alumina) films as thin as 25 nm. Weighing as little as 0.1 gram per square meter, they are among the thinnest and lightest freestanding solids that can be handled with bare hands. We also combined multiple ultrathin layers of alumina to create a nanoscale analog of paper-based corrugated cardboard. Unlike cardboard, these plates have the ability to "pop back" to their original shape, without damage, even after undergoing multiple sharp bends by more than 90 degrees.

Like the nanotruss -based mechanical metamaterials reported by other groups, plate mechanical metamaterials are extremely lightweight and resilient due to their nanoscale thickness and microscale cellular structure. However, in contrast to the cube-shaped metamaterials that typically form a lattice easily penetrated by the ambient air, our plates form flat continuous plates. Ultralow weight, mechanical robustness, thermal insulation, as well as chemical and thermal stability of alumina make plate metamaterials attractive for numerous applications, including structural elements in flying microrobots, high-temperature thermal insulation in energy converters, testing of nanoscale strength enhancement, new types of optical and acoustic metamaterials, as well as ultrathin MEMS/NEMS sensors and ultra-lightweight hollow MEMS/NEMS resonators.

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Room 24 - Session MN+BI+EM+SS+TR-TuM

Microelectromechanics: Relays to RF/Surfaces in Micro- and Nano- Systems

Moderators: Sushma Kotru, University of Alabama, Roya Maboudian, University of California at Berkeley

8:00am MN+BI+EM+SS+TR-TuM-1 The Industrialization of MEMS through Materials Innovations, *Chris Keimel*, Menlo Micro **INVITED**

For the past 150 years, the mechanical relay was one of the original building blocks of electrical systems, for power electronics, controls, and even computing. With the introduction of the transistor in the middle of the 20th century, many industries were transformed with the introduction of ubiquitous, low-cost switches (solid-state) that could be manufactured by the billions with highly advanced equipment and manufacturing processes. Still today, many industries, especially power distribution and controls, are still not able to live with the tradeoffs of solid-state technologies (leakages, losses, lack of air-gap, thermal) and continue to employ large, slow, and costly mechanical relays which have evolved only slightly over the past 50+ years. The miniaturization of the mechanical relay through MEMS technology, coupled with materials innovations, will enable a new class of devices capable of connecting (wireless control) and controlling (distributed power) today's and the futures billions of automated electrical nodes.

We have developed electrostatically actuated MEMS relays capable of switching in ~3usec, sustaining more than 400V across its open contacts and controlling loads of 10s of watts to a few kilowatts. Ohmic MEMS switch with creep resistant metal alloy beams, and a highly reliable ruthenium contact has been developed based on methodical failure mode analysis taking into account material, mechanical and electrical constraints. The ohmic relays, when applied to RF applications, deliver multi throw configurations capable of <0.3dB insertion loss from DC to 3GHz combined with the ability to handle 25W of RF power.

A metal MEMS switch technology has been developed from the ground up through material, process, device, package and electronic integration innovations. The combination of fast microsecond switching speed and broadband (DC to RF) signal operation along with the ability to control amperes of current and sustain hundreds of volts across micron sized air gaps has enabled the miniaturization of the mechanical relay for broad ranging applications from wireless infrastructure to the Industrial IOT.

8:40am MN+BI+EM+SS+TR-TuM-3 Electron-Phonon Acoustoelectrics in MEMS, *Dana Weinstein*, Purdue University **INVITED**

The Acoustoelectric (AE) effect is a result of the interaction between free charge carriers and the electrical deformation potential produced by a propagating elastic wave in the piezoelectric. When an external DC electric field is applied across the semiconductor in the direction of the propagating wave, a drift velocity (v_d) is imparted to the free carriers. If the drift velocity is slower than (or opposite to) the acoustic wave velocity (v_s), the electrical deformation potential lags behind the strain wave. This phase lag not only decreases the acoustic wave velocity, but also transfers energy from the acoustic wave to the electrons, increasing the acoustic losses. When a sufficient DC field is applied to cause the drift velocity to exceed the acoustic wave velocity, the electrical deformation potential now leads the strain wave. This transfers energy from the electrons to the acoustic wave, resulting in an increased acoustic velocity and net acoustic gain [1,2,3,4].

A large body of work based on AE was established in the 1960s and 70s, resulting in a range of devices from phase shifters to correlators. With the development of new materials and new processing needs, there has been a recent resurgence of interest in this field, particularly for its amplifying and inherently non-reciprocal properties. Here, we discuss the implications of the AE effect for GHz frequency electromechanical signal processing. RF applications, linearity, and noise of the AE effect will be examined. Finally, benefits and limitations of prospective semiconductor/piezoelectric material systems will be discussed.

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9:20am MN+BI+EM+SS+TR-TuM-5 Autonomous Oscillations of a MEMS Resonator, *David Czaplewski*, Center for Nanoscale Materials, Argonne National Laboratory; *C Chen, D Lopez*, Argonne National Laboratory; *D Zanette*, Centro Atomico Bariloche and Instituto Balseiro; *S Shaw*, Florida Institute of Technology

Resonant MEMS and NEMS structures are used in a wide variety of applications including mass and force sensing, time keeping, and quantum information. For all MEMS and NEMS resonators, energy is lost every cycle of oscillation to the environment (modeled as a coupled bath). If this energy is not restored by an external source, the amplitude of the resonant motion will decrease toward zero. This well-known effect is commonly referred to as "ring-down". For linear resonators, the frequency of the resonator will remain constant and the amplitude will decrease exponentially while for non-linear resonators, the amplitude will decrease exponentially and the frequency will simultaneously decrease toward the linear response due to the amplitude-frequency (a-f) effect. However, we demonstrate a non-linear resonator that has constant frequency and an amplitude that does not decay for a given period of time (~ 0.1 s) after discontinuing the restoring energy to the system. We call this time "coherence time" because the amplitude and frequency of the oscillation does not decay when the restoring energy is removed. In essence, the resonator is autonomous during coherence time. Unfortunately or fortunately, this behavior does not violate the second law of thermodynamics. The behavior can be explained by looking at the entire system. We drive a non-linear MEMS resonator to a frequency where the primary mode couples with another internal mode. When the resonator is actively driven, the higher order mode receives energy from the primary mode. When the external energy is discontinued, this energy is restored back to the primary mode allowing the primary mode to continue to oscillate. However, once the energy stored in the higher order mode is depleted (its amplitude is near zero), the behavior of the primary mode begins to "ring-down". During this talk, I will show characteristics of the coupled modes including operation with constant frequency and a non-decaying amplitude for a period of time with no drive.

9:40am MN+BI+EM+SS+TR-TuM-6 Metallic Glass for MEMS Microphone Device, *MaiPhuong Nguyen*, WPI-Advanced Institute for Materials Research (WPI-AIMR)/ Micro System Integration Center (μ SIC), Tohoku University, Japan; *J Froemel*, WPI-Advanced Institute for Materials Research (WPI-AIMR), Tohoku University, Japan; *S Tanaka*, Graduate School of Engineering/ Micro System Integration Center (μ SIC), Tohoku University, Japan

Micro Electro-Mechanical Systems (MEMS) microphones have been extensively developed and introduced into mobile phones market with high performance such as high signal to noise ratio, good sensitivity, and power consumption and good reliability in terms of packages. Up to now, most studies have been focused on the improvement of sensitivity of microphone which is proportional to the compliance of the membrane. However, no significant progress has been achieved due to the limitation of material itself. Generally, single crystal and polycrystalline silicon based devices are brittle and fracture causing the interior defects during the fabrication processes. Therefore, the research of new materials to substitute polycrystalline silicon is necessary. Amorphous metals exhibit no grain boundaries, crystal defects and excellent mechanical properties such as fatigue free, large elastic limit, high strength, corrosion resistance which has been promising materials for MEMS devices such as micro-scanner, RF MEMS varactor, capacitive switch ... Metallic glasses are a kind of amorphous alloy exhibiting viscous flow at a certain temperature range so-called "supercooled liquid region". In the supercooled liquid region, metallic glasses can be easily produced through a variety of fast-cooling methods and have excellent mechanical formability. In addition, metallic glass thin films are easily prepared on Si or SiO₂ substrates by sputtering technique which is compatible with MEMS processes such as photolithography, dry or wet etching and lift off processing. Therefore, characterization and fabrication of metallic glasses films deposited by sputtering for MEMS microphone will be studied.

The CoTaB films with thicknesses in the range of 100 nm to several micrometers have been successfully deposited on thermal SiO₂ substrates by

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rf-sputter technique. The amorphous structure with smooth surface and negligible magnetic property was confirmed by TEM, AFM, XRD and SQUIDS measurement, respectively. The metallic glass behavior was investigated by DSC analysis which shows the glass transition and crystalline temperature of 700 and 720.9 C, respectively. In addition, the mechanical properties such as stress, stress gradient and Young modulus have been studied by using pointer and cantilever structure. Co-based metallic glass exhibited tensile and compressive stress depending on sputter conditions, thicknesses as well as further treatment process. Additional results will be presented in detail at the conference with an emphasis on the dependence of the process conditions.

11:00am MN+BI+EM+SS+TR-TuM-10 Role of Surfaces in Assembly of Ceria Nanostructures, *Sudipta Seal*, University of Central Florida **INVITED**

Cerium is a rare earth element of the lanthanide series with a fluorite lattice structure. The cerium atom can exist in either 3+ or 4+ states, and may alternate between the two in a redox reaction that is more pronounced in nanoparticles. However, the physicochemical properties of a nanocrystal assembly can be different from the properties of both the individual nanoparticles and the bulk phase. We have synthesized ceria nanoparticles in various medium and studied the self-assembly of particles to octahedral and star shaped nanostructure assembly. It was further identified that the concentration of Ce⁴⁺ in nanoceria decreases over time, further controlling the surface chemistry. We will also highlight some of the key aspects of self-assembly of CeO₂ into nanorods. The surface area available and the orientation of crystallographic planes in ceria nanostructures highly regulate the catalytic property at nanoscale as evident by high resolution TEM. Further we discuss the role of Madelung energy and its relation to the catalytic activity, which is important in sensing and other analyte interactions. The surface chemistry or the ratio of Ce³⁺/Ce⁴⁺ can be extensively modulated by the assembly process. At the end we report, the feasibility of a novel H₂O₂ based electrochemical sensor that directly measures the current response of multivalent ceria in presence of H₂O₂. The fabricated sensor showed a picomolar range limit of detection while remaining insensitive to interfering species. Peroxide sensing is very important in biologically relevant oxidative stress in cells. It was observed that a lower ratio of Ce³⁺:Ce⁴⁺ redox states elicits a greater current response towards H₂O₂. The detection of such electroactive analytes make it easier to detect using normal nanoparticle modified electrodes, thereby eliminating the use of organic mediators.

11:40am MN+BI+EM+SS+TR-TuM-12 Optimization and Nano-characterization of Electrostrictive Response of Gd-doped Ceria Actuators, *Sidney Cohen, E Mishuk, E Makagon, E Wachtel, K Rechav, R Popovitz-Biro, I Lubomirsky*, Weizmann Institute of Science, Israel

Gd-doped ceria (GDC) recently attracted great interest due to its non-classical (non-Newnham) electrostrictive behavior. Although the material is well-known for its ionic conduction properties and use in solid-oxide fuel-cells, it also holds great promise for incorporation into MEMS devices because it is completely inert with respect to silicon compounds. The fact that GDC is lead-free is particularly appealing.

Here, we demonstrate fabrication and testing of membrane actuators formed with near 100% yield by a relatively simple, low temperature process. Preparation of these devices involves magnetron-sputtering of a thin film of GDC onto Si, and further processing using standard micromachining, resulting in free-standing membranes. Bridge and cantilever structures have been fabricated as well, to explore the possibility for diverse functional devices. The films were structurally characterized by electron microscopy and by x-ray diffraction, whereas electrical characterization was performed using impedance spectroscopy and cyclic voltammetry. These electrical tests revealed details of the conduction mechanism, role of the contacts, and charge-trapping.

Scanning probe microscopy was applied to quantitatively characterize the energetics and mechanics of the electromechanical response: Displacement of a circular membrane was measured by recording displacement of the cantilever probe under feedback as a function of frequency and applied voltage, and temporal Joule heating recorded using a scanning thermal probe. These measurements support calculations of heat-induced strain at high frequencies. These measurements showed that displacements obtained are sufficient for practical applications and provided insights on the factors controlling performance.

12:00pm MN+BI+EM+SS+TR-TuM-13 Sustainable Thermoregeneration of Plastrons on Superhydrophobic Surfaces, *Tomer Simovich*, Ruhr-University Bochum, Germany; *J Arnott*, The University of Melbourne, Australia; *A Rosenhahn*, Ruhr-University Bochum, Germany; *R Lamb*, Canadian Light Source, Canada

A popular and desirable function of superhydrophobic coatings is their remarkable ability to retain an entrapped layer of air, called a plastron, when submerged underwater. The drawback is that the air layer is short lived due to solvation into the surrounding liquid. Liquid gas extraction has been explored for the purpose of respiration through oxygen filtering or generation via chemical reaction. Manipulating solubility through temperature has been attempted but due to its inefficiencies has not been developed further into functioning technologies. This paper introduces a novel method of extracting gas from water to generate enough air to permanently stabilize a plastron on superhydrophobic surfaces for sustained anti-fouling, rust resistance and drag reduction abilities. This method involves locally heating the liquid surrounding a superhydrophobic coating, reducing gas solubility causing the gas to migrate to the liquid-air interface. Due to the low surface energy of superhydrophobic coatings, nucleation of supersaturated gasses occurs preferentially at the coating interface, thereby replenishing the plastron. This requires a relatively low energy input, due to the small volume of water required to be locally heated combined with the small temperature differential induced between substrate and liquid. This process may be more environmentally sustainable in comparison to competing methods. With a constant supply of equilibrated water and minimal energy input, the plastron can survive indefinitely without need for the mechanical application of additional gas.

2D Materials Focus Topic

Room 16 - Session 2D+BI+MN+SS-TuA

Surface Chemistry, Functionalization, Bio and Sensor Applications

Moderator: Matthias Batzill, University of South Florida

2:20pm **2D+BI+MN+SS-TuA-1 Preserving Chemically Modified Graphene from Thermal and Chemical Loss of Functionality**, *Keith Whitener*, *W Lee*, Naval Research Laboratory; *R Stine*, NOVA Research; *J Robinson*, *D Kidwell*, *C Tamanaha*, *P Sheehan*, Naval Research Laboratory

Chemical functionalization can dramatically alter graphene's properties, enabling one to tune its chemical and physical properties for a wide range of applications. To be useful, these modifications must be stable; however, some of these chemical modifications can be unstable, allowing the material to partially revert to unfunctionalized graphene over time. In this talk, we present our detailed studies of the kinetics of graphene hydrogenation and dehydrogenation. Single layer hydrogenated graphene can be dehydrogenated via thermal, mechanical, and chemical routes. Interestingly, bilayer graphene is much more robust to both chemical and thermal dehydrogenation than is single layer graphene. Possible mechanisms for this difference in reactivity will be discussed. Finally, we leverage the insights from these studies to first fabricate functional chemistries and electronic devices on graphene and then to transfer the devices *in toto* onto arbitrary substrates including biological ones. This enables graphene to act like a chemical "sticky note", transferring chemical and physical properties from one surface to another.

2:40pm **2D+BI+MN+SS-TuA-2 Chemical Vapor Sensing with 1T/2H Phase Engineered MoX₂ Films**, *Adam Friedman*, *A Hanbicki*, *K Perkins*, *G Jernigan*, *J Culbertson*, *P Campbell*, Naval Research Laboratory

Transition metal dichalcogenides (TMDs) show remarkable potential for use in chemical vapor sensor devices. They are inexpensive, inherently flexible, low-power, can be grown in large areas, and have shown high sensitivity and selectivity to electron donor analyte molecules. However, for most devices the conductance response is dominated by Schottky contacts, to the detriment of the sensitivity and obscuring the intrinsic sensing capability of the devices. We use contact engineering to transition the contacts in a MoS₂ FET-based chemical vapor sensor to the 1T conducting phase, leaving the channel in the 2H semiconducting state, thus providing functional Ohmic contacts to the device. We show that the resultant sensors have greatly improved electrical characteristics, are more selective, and recover fully after chemical vapor exposure—all major improvements to previous MoS₂ sensor devices. We study the dynamics of the sensing reactions identifying two possible models for the chemical sensing reaction with physisorption likely dominant. Additionally, we present both conductance and optical evidence that the phase transition can be induced in MoX₂ films by a saturating dose of strong electron donor vapor. We find that the conductance response to strong electron donors in both monolayer MoS₂ and MoSe₂ FET devices ceases after moderate exposure, with final value of the conductance being on order of that expected for the 1T phase. We also examine chemically exposed TMD films intermittently interrogated with Raman and photoluminescence spectroscopy. We observe the appearance of weak characteristic 1T phase Raman features for MoS₂ and we observed a quenching of the photoluminescence of both TMD films that is recoverable with annealing. The data cannot be explained solely by doping mechanisms. Our results suggest a mechanism for a new type of passive chemical vapor sensor.

[1] F.K. Perkins, A.L. Friedman, et al., *Nano Lett.* **13**, 668-673 (2013).

[2] A.L. Friedman, F.K. Perkins, et al., *Sol. St. Elec.* **101**, 2-7 (2014).

[3] A.L. Friedman, F.K. Perkins, et al., *Nanoscale*, **8**, 11445 (2016).

3:00pm **2D+BI+MN+SS-TuA-3 Nanopores in 2D Materials**, *Aleksandra Radenovic*, Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland
INVITED

Atomically thin nanopore membranes are considered to be a promising approach to achieve single base resolution with the ultimate aim of rapid and cheap DNA sequencing. Recently, we made advances in using nanopore platform for its integration with 2D materials such as graphene or MoS₂. Translocation of various types of DNA exhibits a signal amplitude that is five times higher than in the case of solid-state Si₃N₄ membranes and a SNR of more than 10. These features are highly desirable for event detection and we take advantage of them by showing the electric-field induced unfolding of a 48 kbp long DNA molecule within the nanopore which manifests itself in the quantization of the current drop. Although

single nucleotide identification and DNA sequencing using biological pores have already been demonstrated their fragility, difficulties related to measuring pA-range ionic currents together with their dependence on biochemical reagents, make solid state nanopores an attractive alternative. In this talk I will address novel applications that address identification of single nucleotides but as well go beyond DNA sequencing. We use novel solid state nanopore platform based on atomically thin nanopore membranes in 2D materials such as graphene or molybdenum disulfide for DNA detection, sequencing, water desalination and osmotic power generation.

4:20pm **2D+BI+MN+SS-TuA-7 Spectroscopic Observation of Oxygen Dissociation on Nitrogen-Doped Graphene**, *Mattia Scardamaglia*, University of Mons, Belgium; *T Susi*, University of Vienna, Austria; *C Struzzi*, University of Mons, Belgium; *R Snyders*, University of Mons, Belgium; *G Di Santo*, *L Petaccia*, Elettra-Sincrotrone Trieste, Italy; *C Bittencourt*, University of Mons, Belgium

The reactivity of carbon nanomaterials towards oxygen is very poor, limiting their potential applications as low-cost, high-yield catalysts. However, nitrogen doping is an established way to introduce active sites that facilitate interaction with gases [1,2]. This boosts the materials' reactivity for gas/bio sensing and enhances their catalytic activity for the oxygen reduction reaction, promising to substitute expensive metals in fuel cell cathodes. Despite this interest, the role of differently bonded nitrogen dopants in the interaction with molecular oxygen is obscured by experimental challenges and has so far resisted clear conclusions. We study the interaction of molecular oxygen with graphene doped via nitro-gen plasma by *in situ* high-resolution synchrotron techniques, supported by density functional theory core level simulations [3,4]. The interaction with oxygen gas leads to the dissociation of the molecule and the formation of carbon-oxygen single bonds on the graphene surface, along with a band gap opening and a rounding of the Dirac cone. The change of the N 1s core level signal indicates that graphitic nitrogen is responsible for the observed mechanism: it catalyses the dissociation of an adsorbed oxygen molecule, after which the two O atoms chemisorb with epoxy bonds to the nearest and next-nearest carbon neighbours of the graphitic nitrogen. Our findings help resolve existing controversies and offer compelling new evidence of the ORR pathway.

1. Liu, X., Dai, L. (2016) Carbon-Based Metal-Free Catalysts. *Nat. Rev. Mater.*, **1**, 16064.

2. Ni, S., Li, Z., Yang, J. (2012) Oxygen Molecule Dissociation on Carbon Nanostructures with Different Types of Nitrogen Doping. *Nanoscale*, **4**, 1184-1189.

3. *Scardamaglia, M.* et al., (2016) Tuning Nitrogen Species to Control the Charge Carrier Concentration in Highly Doped Graphene. *2D Mater.*, **3**, 11001.

4. *Scardamaglia, M.* et al., (2017) Spectroscopic observation of oxygen dissociation on nitrogen-doped graphene. Submitted

4:40pm **2D+BI+MN+SS-TuA-8 Back to Black: Can Molecular Networks Preserve the Surface of Black Phosphorus?**, *Vladimir Korolkov*, The University of Nottingham, UK; *I Timokhin*, *R Haubrichs*, CristalTech Sàrl, Switzerland; *S Yang*, *M Schröder*, University of Manchester, UK; *P Beton*, The University of Nottingham, UK

Black phosphorus (BP), one of several allotropic forms of phosphorus, has a layered structure and is a narrow gap semiconductor with a bulk band gap of ~0.3 eV. Similar to other layered materials it can be exfoliated with scotch tape to form a single layer of black phosphorus known as phosphorene. Unlike gapless graphene, phosphorene has a band-gap which was predicted, and later confirmed to be ~2 eV. The band gap is thickness dependent and thus can be easily tuned. Since the first reports of exfoliation of BP, and some 100 years after the first high-pressure synthesis of black phosphorus crystals by Bridgman in 1914, phosphorene or few layered BP has been widely used to construct transistors, including flexible devices.

One of the biggest challenges in BP and phosphorene research remains its stability under atmospheric conditions.

In this work we explore a new route to the solution of this problem through an investigation of the compatibility of BP with the formation of supramolecular networks which have monolayer thickness and are stabilised by non-covalent in-plane interactions, specifically hydrogen bonding. We find that supramolecular networks can be formed on BP and demonstrate this for a mono-component nanoporous array of trimesic acid (TMA) and the bimolecular network formed by cyanuric acid (CA) and

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melamine (M). While the more open TMA array does not passivate the BP surface, the hexagonal melamine cyanurate (CA.M) array is highly effective and provides protection under ambient conditions over a period of more than three months. In addition, we identify the orientation of the CA.M relative to the rows of phosphorus atoms at the surface and, normal to the rows, observe moiré effects which are characteristic of a well-ordered interfacial structure. We have further demonstrated that CA.M monolayers on BP provide a stable platform for the sequential growth of additional molecular layers, for example, 1,2,4,5-tetrakis(4-carboxyphenyl)benzene (TCPB), leading to the formation of a supramolecular heterostructure and demonstrating the facility for further functionalisation of the BP substrate.

Our work demonstrates that a single layer of CA.M can successfully passivate the surface of BP and preserve it intact for at least 3 months. We believe that this facile approach of depositing a passivating organic monolayer stabilised by in-plane non-covalent bonding could be extended to the protection of other two-dimensional materials with air sensitive atomically flat surfaces, and is likely compatible with other solvents and molecules.

The work also presents outstanding examples of high resolution AFM imaging achieved under ambient conditions.

5:00pm **2D+BI+MN+SS-TuA-9 Defect-mediated Properties of Single-layer MoSe₂**, *Sara Barja*, Materials Physics Center, San Sebastián, Spain; *S Wickenburg*, *Z Liu*, *Y Zhang*, Molecular Foundry, Lawrence Berkeley Lab; *A Pulkin*, Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland; *S Refaely-Abramson*, *B Schuler*, Molecular Foundry, Lawrence Berkeley Lab; *H Ryu*, Lawrence Berkeley National Laboratory; *D Qiu*, University of California at Berkeley; *M M. Ugeda*, CIC nanoGUNE, Spain; *Z Shen*, Stanford Institute of Materials and Energy Sciences; *S Mo*, *M Salmeron*, Lawrence Berkeley National Laboratory; *M Crommie*, University of California at Berkeley; *D Ogltree*, Molecular Foundry, Lawrence Berkeley Lab; *O Yazyev*, Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland; *J Neaton*, *A Weber-Bargioni*, Molecular Foundry, Lawrence Berkeley Lab **INVITED**
Properties of two-dimensional transition metal dichalcogenides are highly sensitive to the presence of defects in the crystal structure. A detailed understanding of the defect electronic structure may lead not only to the control of the material's properties through defect engineering towards a particular device application, but also may lead the emergence of novel physico-chemical functionalities. We show how linear mirror twin boundaries and individual atomic defects in single-layer MoSe₂ alter the electronic structure of the pristine semiconductor. Such linear and point defects tend to be highly localized in the plane, which imposes the need of experimental and theoretical characterization of the defects at the atomic level. Using non-contact atomic force microscopy and scanning tunneling spectroscopy, we directly correlate the morphology and electronic properties of structural defects in MoSe₂ at the defect-length scale. We provide direct evidence for the existence of isolated, one-dimensional charge density waves at mirror twin boundaries in single-layer MoSe₂. We also determine the local density of states of Se vacancies in monolayer MoSe₂ and discuss the correlation to density functional theory calculations, studying the role of the GW approximation to reproduce the energetics of the valence and conduction band as measured in the experimental dI/dV spectra.

5:40pm **2D+BI+MN+SS-TuA-11 Scalable Flexible Graphene Gate TMD Biosensors**, *RamSurya Gona*, *C Naylor*, *C Johnson*, University of Pennsylvania

Two dimensional transition metal dichalcogenides, such as MoS₂ and WS₂, have been shown to be promising materials for use in bio-sensing. I will present our work on the fabrication of scalable flexible MoS₂ field effect transistors with patterned graphene back-gate. Flexible devices were fabricated on a Kapton substrate and incorporating graphene as the back-gate material due to its biocompatibility and its favorable physical properties. Monolayer MoS₂ single-crystal flakes were grown over large area by chemical vapor deposition, and then transferred onto a pre-patterned electrode array, resulting in a device yield > 70% and an average mobility of 1.0 cm²V⁻¹s⁻¹. To create nano-biosensors, the surface of the MoS₂ was functionalized via a reengineered mu-opioid receptor and the devices were tested against opioid solutions of various concentrations. This work provides a pathway for the integration of MoS₂ and other TMDs onto flexible/wearable/implantable devices that for trace detection of opioids or

other chemicals. This work was supported by the National Science Foundation through EFRI 2DARE ENG-1542879

6:00pm **2D+BI+MN+SS-TuA-12 Development and Validation of Polarized Models for Peptide-Graphene Interactions**, *Amanda Garley*, University of Colorado Boulder; *N Saikia*, Michigan Technological University; *R Berry*, Air Force Research Laboratory; *H Heinz*, University of Colorado Boulder
Biosensor technologies require the understanding of interactions between organic and inorganic materials to tune electric response functions, such as peptide assembly on graphitic substrates. Laboratory characterization of specific interactions and molecular assembly can be complemented by atomistic molecular simulations, as well as by quantum-mechanical analysis of band gaps and expected conductivity.

As a first step, we improved common dispersive interatomic potentials for graphite to include pi electron density at virtual sites. The new model reproduces experimental cation-pi energy, X-ray structure, density, cleavage energy, hydration energy, contact angle and elastic constants. As a result we have improved existing models which gave the wrong sign of hydration energies and deviations up to 1000% in these properties from experiment. The parameters are embedded in CHARMM, CVFF, TEAM-AMBER, and other common force fields as part of the INTERFACE force field. An analysis of binding residues, binding energies, conformations, and dynamic information of molecular mobility on the surfaces will be presented.

Nanometer-scale Science and Technology Division Room 19 - Session NS+EM+MN+PS+SS-TuA

Nano-Photonics, Plasmonics and Mechanics

Moderators: Joshua Ballard, Zyvex Labs, Christian Zorman, Case Western Reserve University

2:20pm **NS+EM+MN+PS+SS-TuA-1 Nonlinear Interactions of Coupled MEMS Cantilevers**, *Christopher Wallin*, National Institute of Standards and Technology, Center for Nanoscale Science and Technology; *R De Alba*, *D Westly*, NIST/CNST; *S Grutzik*, Sandia National Laboratories; *A Zehnder*, *R Rand*, Cornell University; *V Aksyuk*, NIST/CNST; *S Krylov*, Tel Aviv University, Israel; *B Ilic*, NIST/CNST

Micro- and nano-electromechanical systems (M/NEMS) offer tremendous opportunities for technological advancement in mechanical resonator applications including mass, force and energy sensing, microwave amplification, optomechanics, and energy harvesting. These M/NEMS resonators have many favorable qualities including high mechanical quality factors and compatibility with integrated circuit architectures. More specifically, nonlinear, coupled M/NEMS resonating cantilever arrays have been shown to possess complex system dynamics such as intrinsically localized modes, wave propagation, and sensitivity to defects. The collective behavior of these nonlinear interacting cantilever arrays is remarkably sensitive to the slightest perturbation which makes them an excellent candidate for ultra-sensitive sensors. Moreover, custom device responses can be achieved by tuning the electrostatic fringing field coupling, altering the mechanical coupling via the device's overhang, or by introducing precisely engineered structural imperfections into the arrays. With our work, we have found that the cantilever arrays exhibit distinct propagation bands, abrupt transitions between standing wave patterns, and synchronization.

Various device geometries including interdigitated arrays, opposing element arrays, and di-element arrays were constructed using both silicon and silicon nitride as device layers. The arrays generally consisted of 100 cantilevers or more which limited boundary effects in the devices. Gold electrodes were patterned on top of the cantilevers for parametric electrical actuation and for fringing field electrostatic coupling between adjacent cantilevers. Mechanical coupling in the arrays was achieved through the large overhangs produced during the device release. The amplitude envelope of the out of plane motion of the cantilevers was captured using a CMOS camera using a frame rate of 30 s⁻¹. The devices were driven electrically and using a piezoelectric transducer under ambient and vacuum conditions. Large, nonlinear vibrational amplitudes were observed in the arrays along with hysteresis. The cantilever arrays exhibited unique standing wave patterns which were sensitive to defects and external loading. Since the dynamics of M/NEMS coupled cantilevers are highly sensitive to local changes in their environment, we envision the practical implementation of coupled arrays for ultra-sensitive chemical, biological, and force sensors in the future.

2:40pm **NS+EM+MN+PS+SS-TuA-2 Silicon Carbonitride Nanoresonator Arrays for Proteomic Analysis**, *W Zheng*, University of Alberta, Canada; *R Du*, University of Alberta and The National Institute for Nanotechnology; *Y Cao*, University of Alberta and The National Institute for Nanotechnology, Canada; *M Mohammad*, *S Dew*, University of Alberta, Canada; *M McDermott*, University of Alberta and The National Institute for Nanotechnology; *Stephane Evoy*, University of Alberta, Canada

Analysis of biological molecules is vital in many fundamental problems of molecular biology. ELISA is a widely employed array-based technique for the parallel analysis of biological analytes. This technique however requires fluorescent tagging, which may disrupt the biochemical properties being investigated. Other platforms such as quartz crystal microbalance (QCM) and surface plasmon resonance sensors (SPR) offer alternatives for the analysis of molecular mixtures. However, these platforms are not readily scalable towards large arrays. Resonant mechanical sensors operate by monitoring shifts of resonance frequencies associated to the binding. Such approach enables the frequency modulation of the output, improving the stability/noise-immunity of the reading. In addition, the adsorption sensitivity per unit area of resonators scales favourably as their dimensions are reduced, offering a compelling path for the development large arrays with exquisite mass-sensitivities.

Suspended silicon resonators as narrow as 45 nm were initially reported by Carr, Evoy et al.¹ The brittle properties of this material however limited the yield of these structures to less than 25 %, precluding their use in large arrays. We have recently reinvented the overall approach employed in NEMS fabrication. This new approach combines surface and bulk machining techniques for the release of the device, as opposed to the widely-accepted sacrificial layer approach. We are now routinely fabricating ultra-large arrays of SiCN nanostring resonators as narrow as 8 nm and a yield approaching 100%. These are the narrowest devices produced by any machining method. Each device offers a detection threshold as small as 200 Da. These arrays have successfully been employed for the detection and analysis of protein mixtures. Diazonium modification was developed onto the SiCN surfaces and validated by X-ray photoelectron spectroscopy. Similarly modified nanostrings were then covalently functionalized with anti-rabbit IgG as molecular probe. Specific enumeration of rabbit IgG was successfully performed through observation of downshifts of resonant frequencies. The specificity of this enumeration was confirmed through proper negative control experiments. Helium ion microscopy further verified the successful functionalization of nanostrings.

¹D. W. Carr, S. Evoy, L. Sekaric, H. G. Craighead, J. M. Parpia, *App. Phys. Lett.* 75, 920 (1999).

3:00pm **NS+EM+MN+PS+SS-TuA-3 Cavity Optomechanical Coupling in Chip-Scale Plasmonic and Photonic Transducers for Nanoscale Measurements and Optical Signal Control**, *Vladimir A. Aksyuk*, *S An*, NIST Center for Nanoscale Science and Technology; *B Dennis*, Rutgers University and NIST CNST; *T Michels*, *B Roxworthy*, *J Zou*, NIST Center for Nanoscale Science and Technology

INVITED

Devices controlling light via mechanical motion are ubiquitous, from a simple camera's zoom lens to arrays of moving mirrors correcting for atmospheric distortions in telescopes and digitally projecting movies on the cinema screens. The same optomechanical coupling provides one of the best known techniques for measuring mechanical motion, covering length scales from atomic force microscopy to kilometer scale LIGO interferometers to the red shift measurements over billions of light years. We study optomechanical coupling in micro and nanoscale systems that combine electromechanics with photonics and plasmonics, and apply such chip based optomechanical transducers to solve nanoscale measurement problems. In one example, integrated cavity-optomechanical sensing breaks the common trade-off between sensitivity and bandwidth in atomic force microscopy, allowing extremely low noise motion readout of very fast, nanoscale/picogram mechanical probes. Reducing the probe size not only increases the transduction bandwidth, but also reduces drag and therefore the fundamental thermodynamic force noise when operating in air. Even though the cantilever cross-section is much smaller than the optical wavelength, the near-field coupled high quality factor photonic cavity makes our motion readout exquisitely sensitive. As a second example, I will discuss nanomechanical plasmonic systems, where extreme confinement of the gap plasmon optical modes leads to some of the largest optomechanical coupling coefficients ever observed. I will present electro-mechanical gap plasmon phase modulators and nanomechanically tunable deep subwavelength gap plasmon resonators with potential applications for motion metrology, novel nanoscale sensing and signal transduction and

arbitrary wavefront control via nanoelectromechanically tunable optical metasurfaces.

4:20pm **NS+EM+MN+PS+SS-TuA-7 An Active Plasmomechanical System for Optical Modulation and Mechanical Lasing**, *Brian Roxworthy*, *V Aksyuk*, NIST

Plasmonic structures can couple electromagnetic radiation into volumes much smaller than the limits imposed by diffraction. This strong confinement of light transforms these static metallic nanostructures into sensitive bio-chemical sensors, near-field probes for imaging, nanoscale light sources, and effective optical tweezers [1-4]. Advancing the plasmonics paradigm to include active devices, whose resonant properties can be dynamically tuned *via* various electrical, mechanical, or thermal inputs, has great potential to advance nanoscale optical sensing and transduction and for building functional metamaterial devices [5,6].

We present a tunable plasmomechanical system that couples the localized gap plasmon (LGP) resonances of individual subwavelength structures to mechanical, electrical, and thermal modes. By engineering extremely strong optomechanical coupling of the LGPs, we achieve broad tuning of the localized resonances at megahertz frequencies using small voltages < 5 V, producing ≈ 40 % amplitude in the far field and $> \pi$ phase shift of the re-radiated light. We furthermore show selective, sub-diffraction optical transduction of nanomechanical motion with < 10 fm Hz^{-1/2} sensitivity. Coupling of LGPs to thermal modes results in strong thermomechanical backaction capable of driving regenerative mechanical oscillations of cantilever devices – mechanical lasing – using an isolated, subwavelength plasmonic element. Our platform opens the door to smart metamaterials having programmed responses across physical domains, tunable metasurfaces and optical components, and studying optically-powered nonlinear nanomechanics.

[1] J. Anker *et al.*, *Nat. Mater.* 7, 442–453 (2008).

[2] D. K. Gramotnev and S. I. Bozhevolnyi, *Nat. Photon.*, 83–91 (2010)

[3] Y.-J. Lu *et al.*, *Science* 337, 450–453 (2012)

[4] B. J. Roxworthy *et al.*, *Nano Lett.* 12, 794–801 (2012)

[5] N. Zheludev and E. Plum, *Nat. Nanotech.* 11, 16–22 (2016).

[6] B. J. Roxworthy and V. A. Aksyuk, *Nat. Commun.* 7, 13746 (2016).

4:40pm **NS+EM+MN+PS+SS-TuA-8 Plasmon-enhanced Photo-catalysis Using Collapsible Nano-fingers**, *Yunxiang Wang*, *B Song*, *W Wu*, *S Cronin*, University of Southern California

1. Introduction

Photocatalytic decomposition plays an important role in the treatment of pollutants. It utilizes light radiation to decompose contaminants into non-toxic substances. While TiO₂ is one of the most widely used photocatalysts, visible light can hardly be used to drive TiO₂ due to the short wavelength cutoff of TiO₂. Plasmon-enhanced photo-catalysis can extend the wavelength range due to higher order effects. However, previously reported work has limited efficiency, because the hot spots were not optimized and the TiO₂ located outside the hottest part of the hotspots. Here, we invented a technology to fabricate collapsible nano-fingers to achieve large-area high density optimized hotspots with TiO₂ film located at the hottest part of the hotspots. We demonstrated highest photo-catalysis efficiency that we are aware of.

2. Device fabrication

First, pillar arrays were patterned on the top two layers using UV-curable nanoimprint lithography (NIL) and reactive ion etch (RIE), as shown in Fig. 1(a) (b) (c). Au film was deposited on the sample followed by lift-off process to form gold nanoparticle arrays with diameter of 50 nm and pitch of 200 nm on the bottom layer, as shown in Fig. 1(d) (e). After nano-fingers were fabricated using RIE, 2 nm TiO₂ film was deposited on the sample using atomic layer deposition (ALD), as shown in Fig. 1(f) (g). After the arrays were exposed to ethanol solutions and air-dried, the fingers closed together in groups of two or four. The scanning electron microscopic (SEM) image of the collapsed nano-fingers is shown in Fig. 2.

3. Results and Discussion

The photocatalytic activities were tested using methyl orange (MO) photodegradation as the model reaction. The decay in absorbance of the solution was monitored by Varian Cary 50 UV-Vis spectrophotometer after 8 h exposure to green laser (532 nm, 3 W) irradiation. MO solution and sample were added into a standard quartz cuvette sealed with a sealing film.

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The absorption spectra taken before and after irradiating are used to quantify the photocatalytic decomposition rate, as shown in Fig. 3. As a control experiment, we firstly performed experiment under same illumination condition with a silicon wafer coated with 2nm TiO₂ film, no MO photodecomposition was observed even after 12 h irradiation. For the monomers, the absorption of the MO solution is observed to drop by 4.9% after 8 h illumination. However, with collapsed sample, a 30% reduction in the MO absorbance is observed. This over 6-fold enhancement demonstrates a stronger plasmonic enhancement after nano-fingers being collapsed, which means this novel structure is a great platform to study plasmonic enhancement.

5:40pm **NS+EM+MN+PS+SS-TuA-11 Ultra-High Resolution Photonics-based Thermometry**, *Nikolai Klimov, T Herman, K Douglass, M Chojnacky, Z Ahmed*, National Institute of Standards and Technology

Temperature measurements play a crucial role in various aspects of modern technology ranging from medicine and manufacturing process control, to environmental and oil-and-gas industry. Among various temperature measurement solutions, resistance-based thermometry is a time-tested method of disseminating temperature standards [1]. Although industrial resistance thermometers can routinely measure temperatures with uncertainties of 10 mK, their performance is sensitive to multiple environmental variables such as mechanical shock, thermal stress and humidity. Drift of sensor resistance over time necessitates expensive, time-consuming recalibrations using ultra-sensitive reference thermometers. These 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 temperature measurement solutions such as photonics-based temperature sensors. A wide variety of innovative photonic sensors have been proposed recently including functionalized dyes [2], hydrogels [3], fiber optics-based sensors [4], and silicon micro- and nanophotonic devices [5,6]. These innovative temperature sensors have the potential to leverage advances in frequency metrology to provide cost-effective measurement solutions. Here we present the results of our efforts in developing novel on-chip integrated silicon photonic temperature sensors with nanoscale footprint and ultra-high resolution as an alternative solution to legacy-based resistance thermometers. These sensors are Fabry-Perrot cavity type silicon photonic devices that are based on photonic crystal nanobeam cavity (PhCC), whose high-Q resonant frequency mode is highly sensitive to even ultra-small temperature variations. In this talk we describe nanofabrication, fiber coupling and packaging of these thermometers, as well as their performance. We will present a direct comparison of our photonic thermometers to Standard Platinum Resistance Thermometers, the best in class resistance temperature sensors used to disseminate the International Temperature Scale of 1990. The preliminary results indicate that our PhCC nanothermometers are capable of detecting changes of temperature as small as 10 μ K and can achieve measurement capabilities that are on-par or even better than the state-of-the-art resistance thermometry.

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6:00pm **NS+EM+MN+PS+SS-TuA-12 Size-Controlled Synthesis of Gold Nanostars and their Excellent SERS and Fluorescence Quenching Properties**, *Waqar Ahmed, H Khan, M Khalid*, COMSATS Institute of Information Technology Islamabad, Pakistan

Noble metal nanoparticles have attracted great attention recently owing to their fascinating optical properties. They work as nanoscopic antennas by amplifying the incident and scattered electromagnetic beam. The incident electromagnetic radiation can excite the surface plasmons of nanoparticles, leading to the confinement of electromagnetic energy around the nanoparticles. This makes the metallic nanoparticles an excellent candidate for the surface enhanced Raman scattering (SERS) applications. Anisotropic nanoparticles such as nanostars are much superior for SERS applications over their spherical counterparts owing to the special surface morphology.

We have developed a facile method for the synthesis of gold nanostars with tunable sizes ranging from 50nm to about 1 μ m. To the best of our knowledge, this is the widest size range reported for gold nanostars. More importantly, we have observed that these nanostars are excellent for SERS based detection owing to their large enhancement factors and efficient

fluorescence quenching properties. Fluorescence is known to interfere with and overshadow the SERS signal, thus affecting the trace detection capabilities of SERS. Therefore, usually off resonance excitation lasers must be used for SERS studies of fluorophores, which limits the universal applicability of the SERS technique. We believe that non-compact surfactant coating of nanostars in our case give the target fluorophores access to nanostar's surface, thus enabling the quenching of fluorescence through Förster resonance energy transfer (FRET). The absence of fluorescence background markedly enhances the appearance of Raman peaks. We were able to achieve a limit of detection of 10pM using an excitation laser source in resonance with the electronic excitation of the target fluorophore. This makes gold nanostars universal substrates for SERS based trace detection.

Vacuum Technology Division Room 7 & 8 - Session VT+MN-TuA

Pumping

Moderators: Tamirisa Apparao, SHI Cryogenics Group, Julia Scherschligt, NIST

2:20pm **VT+MN-TuA-1 Silicon-micromachined Turbomolecular Pump**, *Wei Yang*, PD Sciences LLC **INVITED**

Deep miniaturization of sensing and analytical instruments, such as mass spectrometers, vacuum electron devices, atomic clocks, and cold atom devices, are pushing the limit of conventional vacuum packaging technologies in micro scales. Ultra-high vacuum (UHV) of 10⁻⁶ to 10⁻¹⁰ torr which are beyond the capability of current passive packaging technologies, have become increasingly necessary for stable operation and high performance. Although 10⁻⁶ torr and higher vacuum levels are routinely achieved in macro scale systems by passive sealing and getters, maintaining such vacuum at chip-scale has unique challenges arising from scaling laws and practical limitations. Therefore, a micro scale UHV pump is highly desirable as an enabling component for a wide range of mobile or miniature instruments.

Integration of silicon MEMS and precision metal machining offers a viable path to new capabilities unattainable in their own native environments. We will present such an accomplishment in the development of a micro turbomolecular pump that takes advantage of the high-density microstructures from silicon microfabrication, and the range of motion from a precision spindle. Major achievements include compression ratio over 10⁶ and maximum stall pressure of 100 Torr at relatively low tip speed of 120 m/s. This is a major milestone in the pursuit of moving UHV systems from laboratories to mobile platforms. Of particular significance, the successful demonstration of the molecular pumping against such a high exhaust pressure, a direct consequence of dimensional downscaling, points to the feasibility of a single-stage system from UHV to atmospheric pressure in miniature scales. We will discuss key technical challenges such as silicon fabrication, high-tolerance bonding, scaling analysis and simulation methodology, and touch on potential applications in small-scale thermal mechanical systems.

3:00pm **VT+MN-TuA-3 A Rigorous Approach to Effluent Gas Management for the Vacuum Processing Industry**, *Paul Dozoretz*, MKS Instruments, Inc. **INVITED**

Vacuum processing is consistently gaining momentum in the manufacturing of novel materials relying on thin film coating and implantation technologies. The vacuum systems developed for such new applications borrow from many of the standard vacuum processing techniques but consistently push the design limits in terms of the amount of precursor gas consumption and by-product mass generation. In order to handle the large amounts of effluent produced in some of these processes it has become essential to better understand gas dynamics for the effluent flowing out of the chamber and into the pumping systems. In most cases, effluent gas must be captured or trapped before it can reach and irreversibly damage the pumps and before it can become a danger for the personnel operating the vacuum manufacturing tools. With the aid of gas dynamic modeling software, our engineering team has been able to better understand flow through effluent lines and develop more efficient gas trapping solutions for very novel applications spanning from the semiconductor to the aeronautical industries. In this presentation we describe the rigorous methodology used to guide and validate the design effluent gas handling systems. Detailed understanding of process chemistries and effluent physicochemical properties, combined with gas

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dynamic flow modeling, has revolutionized the way our team approaches effluent gas management and improved the speed at which customer effluent needs can be addressed.

4:20pm VT+MN-TuA-7 Compatibility of NEG Pumps with Particle-sensitive Applications: A Review of Recent Experimental Evidences, *P Manini, E Maccallini, Marco Urbano, M Mura, T Porcelli, F Siviero*, SAES Getters, Italy
Non Evaporable Getter (NEG) pumps are frequently used when large pumping speeds for H₂ and active gases (i.e., H₂O, O₂, CO, CO₂) are required in conjunction with very small weight and size, reduced magnetic interference and vibration, or negligible power consumption. Thanks to these qualities NEG pumps are widespread in basic and applied research, such as particle accelerators, storage rings, synchrotrons and physics projects to achieve UHV or XHV conditions. Moreover, their use is becoming familiar in UHV analytical instrumentation such as SEM, TEM, surface science as well as portable mass spectrometry and transportation vacuum boxes.

In spite of the excellent results in terms of pressure (10⁻¹¹ mbar are currently achieved in many machines and values lower than 10⁻¹² mbar have been measured in various experiments using NEG pumps), application in cryogenic superconductive radio frequency (SRF) cavities and other particle sensitive systems is not common so far. As a matter of fact, the use of NEG pumps is limited as a precaution against potential dust emission, which can be transported inside the vacuum envelope and may interfere with the electromagnetic fields and promote unwanted quenching phenomena.

Nevertheless, these systems could greatly benefit from the high pumping speed and compactness of NEG pumps, so that an assessment of the actual risk of dust release is gradually being undertaken by different players, including potential users in the accelerator community. Here we present and compare experimental data on particle emission collected with several techniques both on compressed and sintered NEG elements, discussing the differences. In particular, a class of sintered getters based on the ZAO[®] alloy proved to have extremely low particle emission, as shown by tests carried out in actual SRF cavities, where no measurable particle contamination as well as detrimental effect on the cavity efficiency and performances was observed.

4:40pm VT+MN-TuA-8 NEG Coated Chambers for XHV, *Marcy Stutzman, P Adderley, M Poelker*, Thomas Jefferson National Accelerator Facility

Non-evaporable getter (NEG) thin films are typically applied to uniform diameter tubes, such as used for accelerator beamlines. We have been extending the successful application of NEG coating to larger diameter and non-uniform chambers, such as the 36 and 41 cm diameter chambers for the Jefferson Lab polarized electron source, as well as atom trap chambers for MIT and JILA. We show that by combining the NEG coating with a small ion pump to handle the non-reactive gasses, the chamber can reach the low 10⁻¹² Torr range, and adding additional NEG pumping yields extreme high vacuum (XHV), with measured pressure below 8x10⁻¹³ Torr. With this demonstration of a reliable and reproducible method to achieve room temperature XHV, we hope to demonstrate the benefits of NEG coated chambers beyond accelerator physics applications to other fields of physics and materials research.

5:00pm VT+MN-TuA-9 Ion Pump Noble Gas Stability Mechanism of Titanium Cathode Material, *Anthony Wynohrad*, Gamma Vacuum

It has long been established that ion pumps with titanium cathodes cannot pump large quantities of noble gases without releasing them back into the vacuum environment. Argon is the typical gas chosen for study of this phenomenon due to its prevalence in atmospheric composition and tendency for use in vacuum depth profiling applications. Traditional resolutions to Ar release is through the addition of denser cathode material (Tantalum) or titanium cathode architecture manipulation (triode). Various reports have shown the long term Ar stability of these methods to be subject to manufacturer claims.

To resolve reported discrepancies of Ar stability in ion pumps with titanium and tantalum cathodes, a detailed study of titanium with various physical attributes was conducted. Five different titanium/titanium alloys were tested for Ar instability at standard depth profiling pressures rather than accelerated high pressure testing. The conclusion was reached that varying the physical properties of the titanium can cause ion pumps to become Ar stable or Ar instable. Additionally, the time to reach instability is directly in correlation to the physical attributes of titanium.

5:20pm VT+MN-TuA-10 Ricor's MicroStar/Nanostar Compact Water Vapor Cryopump: Applications and Model Overview, *Rodney Harris*, Ricor-USA, Inc.; *I Nachman, T Tauber, M Kootzenko, B Barak, E Aminov, D Gover*, RICOR Cryogenic & Vacuum Systems, Israel

Ricor Systems has developed a compact, single stage cryopump that fills the gap where GM and other type cryopumps can't fit in. Stirling cycle technology is highly efficient and is the primary cryogenic technology for use in IR, SWIR, HOT FPA, and other IR detector technology in military, security, and aerospace applications.

Current GM based dual stage cryopumps have been the legacy type water vapor pumping system for more than 50 years. However, the typically large cryopanel head, compressor footprint, and power requirements make them not cost and use effective for small, tabletop evaporation / sputtering systems, portable analysis systems, load locks, and other systems requiring small volume vacuum creation from medium, high, and UHV levels. The compact NanoStar configuration was designed specifically to address this vacuum chamber size area.

This single stage cryopump works well in-line with diffusion and molecular turbopumps. Studies have shown effective cooperation with non-evaporable getter technology as well for UHV levels.

Further testing in this area are ongoing. Temperatures created by Stirling cycle cryogenic coolers develop a useful temperature range of 40 to 150K. Temperatures of approximately 100 K are sufficient to condense water and all hydrocarbons oil vapors. The wide temperature range can freeze out many other gaseous compounds.

MEMS and NEMS Group

Room Central Hall - Session MN-TuP

MEMS/NEMS Poster Session

MN-TuP-1 Method for Patterning Crystal Colloidal Masks Using Poly (Acrylic Acid), Connor Smith¹, S Burkett, The University of Alabama

Nanosphere lithography is a nanopatterning technique which has been a useful method for creating nanoscale features, such as nanopillars, that are used in MEMS and NEMS devices. This is achieved by ordering nanospheres in close-packed crystal colloidal masks on a substrate and physically etching said substrate via the interstitial spaces between the nanospheres. Methods for ordering these nanospheres into crystal colloidal masks has been accomplished in many ways, with spin-coating being one of the most cost effective and simplest to implement. Unfortunately, only a few methods exist for patterning these crystal colloidal masks, and few utilize traditional optical lithography techniques. In this work, a method for patterning crystal colloidal masks that are formed via spin-coating is introduced. This method involves spin-coating nanospheres in a solution of water and poly (acrylic acid), and then using modified traditional optical lithography and plasma ashing techniques to pattern the resulting crystal colloidal mask. Once the mask is patterned, normal physical etching methods may be used to further pattern the substrate below the nanosphere embedded poly (acrylic acid) layer. With this new method, patterning crystal colloidal masks for use in nanosphere lithography should be easier due to the wide spread availability of traditional optical lithography tools and instruments.

MN-TuP-2 Understanding the Influence of Space Charge Region on Electrical Behavior of (Pb_{0.95}La_{0.05})(Zr_{0.54}Ti_{0.46})O₃ Thin Film Capacitors Designed using Top Electrodes of Different Various Work Functions, Vaishali Batra², S Kotru, G Cabot II, V Harshan, The University of Alabama

Ferroelectric lanthanum modified lead zirconate titanate (PLZT) material has excellent electronic and optical properties due to which it meets the requirements for various device applications such as optical modulators/transducers, MEMS and smart sensors. Recently this material is being explored for photovoltaic applications. To design devices, the material is integrated with conducting electrodes to prepare metal/ferroelectric/metal (MFM) heterostructures. Electrical behavior of the devices strongly depend on the properties of the electrode/s used to design the MFM structures. In this work, we investigated the dependence of electrical properties of metal/PLZT/Pt capacitors, on a variety of top electrodes, having different work functions (Au, Pt etc.) with Pt serving as bottom electrode.

Thin films of Pb_{0.95}La_{0.05}Zr_{0.54}Ti_{0.46}O₃ (PLZT) were fabricated on Pt(111)/Ti/SiO₂/Si(100) substrates by chemical solution deposition method. Various metal electrodes, with varying work functions, were deposited on top of these films to prepare metal/PLZT/Pt capacitors. The prepared MFM heterostructures form space charge regions at the interfaces between PLZT film and electrode material. Due to different top electrode used to design the capacitor structure, the top interface differs from each other. A detailed analysis of the polarization-electric field (P-E) curves, capacitance-voltage (C-V) characteristics, permittivity-frequency (ϵ_r -f), and current-voltage (I-V) measurements of each of the capacitors allowed us to understand the variation in electrical properties as a function of top electrodes. This variation is mainly attributed to the modification in metal/ferroelectric Schottky contact at the top interfaces which results in creation of different interface electric field, thus altering the properties of PLZT thin film capacitors. Results obtained from this study can guide us to choose the correct electrode material for designing capacitors for a particular application (photovoltaic devices or other types of sensors).

MN-TuP-3 Tribology and Locomotion of Untethered Scratch Drive Actuators with Applications to MEMS Microrobotics, Ratul Majumdar, University of Illinois at Chicago; L Stan, R Divan, Argonne National Laboratory; I Paprotny, University of Illinois at Chicago

Untethered Scratch Drive Actuators (USDAs) [1] have been widely used for actuation of MEMS devices, for example the assembly of 3D MEMS structures [2] and as propulsion mechanisms for stress-engineered MEMS microrobots [1]. These electrostatic actuators show fast and reliable motion on a power delivery substrate along with the ability to provide

forward force of 30 μ N [2]. The power delivery substrate of these USDAs consist of parallel set of interdigitated metal electrodes with a high-k dielectric layer deposited on top [1,3]. The metal electrodes consist of Cr/Au/Cr(10/50/10nm) layers of 2 μ m spacing patterned by electron beam lithography. Sputtered yttria-stabilized zirconium oxide (YSZ) of 500 μ m thickness is used as the dielectric layer. The sputtering parameters, especially the deposition pressure, along with the gas flow of argon and oxygen play an important role in determining the dielectric constant and hence, the power transferred to the USDA for actuation. Variation in the deposition pressure can improve adhesion of the dielectric layer and reduce the delamination during USDA motion. Application of ac voltage between the two parallel set of electrodes results in coupling of capacitive force to the microrobot chassis, thus supplying power to backplate and bushing of the USDA for translation.

For straight motion, a waveform primitive (Fig. 1, supplemental file) with amplitude ranging from 0 to 200V and frequency (f) of 15Hz was applied to the substrate. The waveform is symmetric along horizontal X axis to prevent accumulation of charge on the substrate. The lowest (V_{low}) and highest (V_{high}) value of the voltage waveform is varied and the corresponding motion of the USDA is recorded. The cutoff point at which USDA shows actuation depends on the quality of the dielectric (Fig.2). Interesting interaction with the substrate during release stage is observed by analyzing the V_{low} (Fig. 3 and 4). Improved efficiency of the substrate by transferring more power to the USDA is observed for the power delivery substrate with the YSZ grown at higher pressure (25mTorr). That results in reliable motion and less delamination of the dielectric surface.

References

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MN-TuP-5 Effect of Seeding Material on Sc_{0.125}Al_{0.875}N c-axis Orientation, Erica Douglas, M Henry, T Young, B Griffin, Sandia National Laboratories

While piezoelectric AlN is presently implemented into several commercial applications for electronic devices (such as bulk acoustic wave (BAW) and surface acoustic wave (SAW) filters), alloying AlN with Sc is actively being investigated as a method for increasing the piezoelectric coefficient d_{33} . Sc_xAl_{1-x}N, with x approaching 40%, has reported an increase in the piezoelectric coefficient of almost 4X, with potential for high impact for wireless applications by improving bandwidth and decreasing insertion loss. However, the addition of Sc into AlN has led to secondary grain growth, observed even for low Sc content thin films.

This work will demonstrate enhanced c-axis orientation of polycrystalline Sc_{0.125}Al_{0.875}N on <100> silicon by reactive sputtering with various seeding material, including Si, Pt, and AlCu. We will characterize (100) secondary grain growth on Si as a function of film thickness, as well as X-ray diffraction rocking curve full width half maximum (FWHM) of the (002) orientation. As film thickness increases, rocking curve FWHM approach 1.3 degrees for a 2.1 μ m film; low FWHM values are known to have high piezoelectric coupling. In addition, c-axis orientation was investigated with metal seeding material such as Pt and AlCu. 750nm Sc_{0.125}Al_{0.875}N exhibited a 20% reduction of (002) FWHM and complete suppression of (100) secondary grain growth when seeding on AlCu as compared to Si.

To quantify the effect of Sc_{0.125}Al_{0.875}N growth, contour mode width extensional resonators (CMRs) were fabricated and tested to extract fundamental device parameters such as quality factor(Q) and effective piezoelectric coupling coefficient (K_{eff}^2). The method for suppression of (100) secondary grain growth and improved (002) FWHM utilizing AlCu is CMOS compatible and is shown to create CMRs with -4 dB insertion losses and K_{eff}^2 values of 2.3%.

This project was supported by the LDRD program at Sandia National Laboratories. Sandia National Laboratories 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 U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA-0003525. The authors acknowledge and thank the staff of Sandia's MESA fabrication facility.

¹ MEMS/NEMS Best Paper Award Finalist

² MEMS/NEMS Best Paper Award Finalist

Tuesday Evening Poster Sessions, October 31, 2017

MN-TuP-8 MEMS-Based, High-Resolution Nanocalorimeter for Characterizing Phase Transitions in Samples in the Sub-Microgram Range, Zhu Diao, Stockholm University / Halmstad University, Sweden; D Campanini, A Rydh, Stockholm University, Sweden

High quality thermodynamic measurements are among the essential tools to investigate fundamental properties of materials. Novel materials are often only available in minute amount when first synthesised. Hence, it is of paramount importance to develop thermodynamic measurement techniques for small-sized samples. However, studies involving samples of sub- μg in masses are extremely difficult to perform. Conventional calorimeters are marked by the large heat capacity of the calorimeter cell (addenda), thus, not suitable for measuring sub- μg samples. Rapid development in the MEMS industry has led to the creation of nanocalorimeters [1]. Typically, these devices are constructed on bulk micromachined membranes, whose contribution to the device addenda is vanishingly small.

We have developed a MEMS-based, truly differential nanocalorimetry platform, which is capable of performing specific heat measurements on sub- μg of high-quality, single crystalline samples in a wide temperature range between 0.3 K – 400 K [2,3]. It operates according to a refined ac steady-state method, leading to both ultrahigh resolution (better than several in 10^{-5}) and superior absolute accuracy (1 – 2%) [2]. The calorimeter consists of two 150-nm-thick SiN_x windows, one serving as the sample cell while the other being the reference cell. Each calorimeter cell contains a GeAu thermometer, a titanium ac heater, and an offset heater. The annealed GeAu thermometer shows an almost temperature-independent relative sensitivity $|\text{dlnR}/\text{dlnT}| \sim 1$, covering the whole temperature span of interest.

We demonstrate the capability of our nanocalorimeter through measurements of high purity gallium samples with masses in the range of several hundred nanograms to a few micrograms. α -Ga, the stable polymorph of solid gallium, melts at 302.9 K. Upon cooling, significant supercooling occurs and it may solidify into the metastable β -phase. Compared with α -Ga, β -Ga retains significantly enhanced electron-phonon coupling, leading to an elevated superconducting transition temperature above 6 K. We show that the melting and solidification transition of gallium can be monitored in-situ on our nanocalorimeter utilizing the offset heater, and present specific heat data of both phases across the full temperature range. The high-resolution of our calorimetry scheme also allows in-depth characterization of the superconducting transition of β -Ga and the deduction of a number of important superconducting parameters.

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MN-TuP-9 PLD covering the Innovation Chain to Accelerate the Commercial Uptake of Novel Thin Film Materials, Matthijn Dekkers, A Janssens, Solmates, Netherlands

It is well known that Pulsed Laser Deposition (PLD) is a very flexible and versatile technique allowing fast optimization of new and complex material thin films. The unique features of PLD allow for the integration of “Beyond Moore” materials in CMOS and new devices. However, mainly because of the sample size, the developed materials and processes in PLD research tools only just make it into demonstrator devices. In order to make it into commercial applications, next generation PLD equipment is needed to bridge the gap between demonstrator and the prototype – pilot – production stages.

The Solmates PLD platform is the next step beyond fundamental PLD research. The reliable hardware is flexible for fast process optimization and allows uniform thin film deposition up to 200 mm diameter with high reproducibility. The automated software ensures easy operation and stable performance. These characteristics enable the integration of PLD thin films in applications for (pilot) production and commercialization.

In this contribution the latest performance and specifications of Solmates PLD platform are addressed. Data on stability and reproducibility of wafer scale deposition of PZT thin films with excellent properties will be presented. Furthermore, two qualified processes Indium Tin Oxide and Aluminum Oxide thin films will be used to show some key capabilities of PLD such as damage free deposition on organic electronics or control of thin films density and microstructure for optical or sensing applications.

MEMS and NEMS Group

Room 16 - Session MN+2D-WeM

2D NEMS

Moderators: Zenghui Wang, Case Western Reserve University, Zhu Diao, Halmstad University/Stockholm University

8:00am **MN+2D-WeM-1 Micro-patterned Graphene Temperature Sensors on Different Substrates**, *B Davaji*, Marquette University, Cornell University; *H Cho*, Dongguk University; **Jong-Kwon Lee**, National Nanofab Center in Korea; *T Kang*, Dongguk University; *C Lee*, Marquette University

Since the performance of electronic devices suffers from elevated temperatures as a result of self-heating, outstanding thermal properties of graphene are considered to be suitable for both instrumentation and integrated microelectronic applications [1]. Also, recently developed techniques for fabricating complex graphene structures in micro/nano scale [2, 3] make graphene a great candidate for temperature sensor applications due to its excellent electrical properties, outstanding mechanical strength, and high thermal conductivity.

In this study, micro-fabricated single-layer graphenes on a SiO₂/Si, a SiN membrane, a suspended architecture, and a spatially nano-modulated Si substrate are presented for their use as temperature sensors. These graphene temperature sensors act as resistance temperature detectors, showing a quadratic dependence of resistance on the temperature. The observed resistance change of the graphene temperature sensors are explained by the temperature dependent electron mobility relationship ($\sim T^4$) and electron-phonon scattering. The transient response analysis of the graphene temperature sensors on different substrates shows that the graphene sensor on the SiN membrane exhibits the highest sensitivity due to low thermal mass, whereas the sensor on SiO₂/Si reveals the lowest one. In addition, the graphene on the SiN membrane reveals the fastest response, as well as better mechanical stability in comparison with the suspended graphene sensor. Therefore, we can expect that the graphene temperature sensors with an extremely low thermal mass will be used in various applications requiring high sensitive and fast operation.

References

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8:20am **MN+2D-WeM-2 Characterizing the Resonant Behavior and Quality Factors of 3C-SiC Diaphragms Using Frequency Analysis and the Ring-Down Technique**, *Yongkun Sui*, *H Chong*, *K Shara*, *C Zorman*, Case Western Reserve University

Silicon carbide (SiC) has become a mainstream material for microelectromechanical systems (MEMS) due to its unique combination of outstanding electrical, mechanical and chemical properties, making it the preferred choice for applications in harsh environments where Si is not well suited. SiC is an attractive material for MEMS that utilize mechanical transduction due to its high Young's modulus, mechanical strength and chemical inertness. The cubic polytype of SiC (3C-SiC) is particularly attractive for resonant sensing applications because SiC diaphragms can readily be fabricated from thin films by Si bulk micromachining.

This abstract reports the findings of a study to characterize the resonant behavior of MEMS-based single crystalline 3C-SiC diaphragms. The 1 x 1 mm² diaphragms consisted of 3C-SiC films that were heteroepitaxially grown on Si by APCVD and created by conventional bulk micromachining. The diaphragms were excited into resonance under vacuum using a piezoelectric PZT crystal and their vibratory behavior was assessed using a custom-built optical interferometer.

Over 20 resonant peaks were observed from a 250 nm-thick diaphragm for frequencies up to ~ 2 MHz. Quality factors were initially determined by analyzing the full-width-at-half-maximum of particular resonant peaks from the frequency spectrum. Although the fundamental mode exhibited a quality factor of ~ 3000 , at least 3 other modes had high Q's of $>20,000$, with the highest being over 119,000. For those high quality factor resonance modes, the vibration energy took ~ 1 s to fully dissipate. As such, the frequency response had to be measured in a relatively slow manner

otherwise the residual energy would propagate, resulting in a broadened peak. The ring-down test, which specifically characterizes the vibration energy dissipation rate, was used to measure the high quality factors. The highest Q at (2,3) mode was found to be 195,981 using ring-down test compared to 119,200 from the FWHM method. The resonance modes of the SiC diaphragm showed a non-linear Duffing behavior when the drive voltage exceeded 200 mV. The resonance peaks exhibited jump discontinuities and one of the half-power points ceased to be experimentally visible. In the nonlinear regime, quality factors measured by ring-down test differ only 1% from those in the linear region measured by both the FWHM and ring-down techniques.

8:40am **MN+2D-WeM-3 Ion Radiation Effects in Silicon Carbide (SiC) Crystal Probed by Multimode Diaphragm Resonators**, *Hailong Chen*, *V Pashaei*, Case Western Reserve University; *W Liao*, *C Arutt*, Vanderbilt University; *H Jia*, Case Western Reserve University; *M McCurdy*, Vanderbilt University; *C Zorman*, Case Western Reserve University; *R Reed*, *R Schrimpf*, *M Alles*, Vanderbilt University; *P Feng*, Case Western Reserve University
Radiations effects from energetic particles (ions) and electromagnetic waves (photons) on electronics (*e.g.*, MOSFETs and ICs) have been widely investigated for applications in radiative harsh environments including space and nuclear reactors [1]. Radiation effects in mechanical domain, however, remain largely unexplored due to challenges in capturing and detection [2]. Meanwhile, most of preliminary studies on radiation effects in mechanical domain have been limited to Si structures and devices [3-4], while those on more intriguing radiation-durable materials such as SiC have not been investigated yet.

Here we report on experimental investigation and analysis of energetic ion radiation effects on silicon carbide (SiC) crystal, by exploiting a novel scheme of 4 vertically stacked resonant micromechanical SiC diaphragms. The SiC diaphragms are fabricated using a standard photolithographic and wet etching process to form resulting diaphragms (1 mm x 1 mm x 2.1 μ m). An S-series Pelletron system is employed to irradiation oxygen ions into the SiC diaphragms (14.3MeV, 8h radiation, corresponding to a total fluence of 5.6 $\times 10^{13}$ /cm²). Before and after radiation, multimode resonances are characterized in vacuum by using an ultrasensitive optical interferometry system. We have observed as large as $\sim 9\%$ frequency shifts (the largest value to date) in the multimode resonances of the 3rd diaphragm (counting from top in the stack) where most ions are expected to stop, as well as obvious quality (Q) factor degradation, which result from ionizing and displacement radiation damage. The 1st and 2nd diaphragms, which ions have mostly penetrated, exhibit moderate multimode frequency downshift of $\sim 2\%$ owing to displacement damage, while the 4th diaphragm shows the least frequency shift $\sim 0.1\%$. The diaphragm stack exhibits outstanding capability for probing radiation damages in MEMS, not only able to capture the radiation events obviously but also help analyze different amount and types of damages induced in each stacking layer. Combining the data with a mixed elasticity model (that takes into account both flexural rigidity and pre-tension effects), we find: (i) the diaphragms operate in the *transition* regime (between *'plate'* and *'membrane'* but closer to the latter). (ii) after radiation behavior moves further towards the *'plate'* regime, suggesting reduction in *built-in tension* and possible reduction in Young's modulus as well.

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- [2] H. Shea, *et al.*, J. Micro/Nanolith. MEMS MOEMS. **8**, 2009.
- [3] B. Bhandaru, *et al.*, IEEE Trans. Nucl. Sci. **62**, 2015.
- [4] H. Gong, *et al.*, IEEE Trans. Nucl. Sci. **64**, 2017.

9:00am **MN+2D-WeM-4 High-Aspect Ratio, Multi-Electrode, Carbon Nanotube Array**, *Berg Dodson*, *G Chen*, *R Vanfleet*, *R Davis*, Brigham Young University

We demonstrate a carbon nanotube based, high aspect ratio (~ 1 mm tall and 20 μ m diameter posts) multi-electrode array with individually addressable electrodes. The mechanical robustness and electrical conductivity of the carbon nanotubes make them a good candidate for the multi-electrode array. The electrode is made out of CNT posts which were grown on a patterned conductive substrate and kept vertically aligned with supporting hedges. The supporting hedges were subsequently removed to leave an isolated CNT post array. The fabrication method makes the structure compatible with a variety of surface coatings, including carbon, metals, and ceramics. Good electrical connection is made to individual elements of the array despite the insulating alumina barrier that is used between the substrate and CNT forests.

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11:00am **MN+2D-WeM-10 Interferometric Motion Detection in Atomic Layer 2D Nanoelectromechanical Systems (NEMS), Zenghui Wang**, University of Electronic Science and Technology of China, China; P Feng, Case Western Reserve University

Atomic layer crystals have emerged as a new class of two-dimensional (2D) materials, exhibiting great promises for both fundamental research and technological applications. Their outstanding mechanical properties make these materials ideal for constructing novel 2D nanoelectromechanical systems (NEMS), providing opportunities for coupling their material properties across multiple information-transduction domains, at scales down to individual atomic layers. One particularly interesting prototype of 2D NEMS is 2D nanomechanical resonators. While various electrical, mechanical, and optical motional signal transduction schemes have been employed for 2D NEMS resonators, laser optical interferometry [1][2] clearly stands out as one of the most important and widely used techniques. To date, it is the only technique capable to measure the completely undriven thermomechanical motions in these 2D nanostructures.

Toward pushing the ultimate limits, it is highly desirable to quantitatively understand the detection efficiency and its dependence on the device parameters and interferometric conditions. Here, we present a systematic study [3] of the intrinsic motion responsivity in 2D NEMS using a Fresnel-law-based model, analyzing the dependences of motion responsivity upon parameters in device structure, probing wavelength, and type of 2D material. We find that the best responsivity is achieved as the vacuum gap varies (with crystal thickness) around integer multiples of half of the probing wavelength. The optimized device thickness depends on both the type of crystal and probing wavelength. Specifically, when using 633nm He-Ne laser, the $\sim 300\text{nm-SiO}_2\text{-on-Si}$ substrate commonly used in 2D research offers close-to-optimal motion responsivity for several 2D crystals over a wide range of thickness, provided that the oxide is fully removed underneath the 2D layer. We further show that different type of 2D layered materials exhibit different patterns in the same parameter space due to their different band structure and dielectric constants.

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11:20am **MN+2D-WeM-11 NEMS on Flexible Substrates for Strain Engineering in Sensing Applications, Swapnil More**, Indian Institute of Science, India

Although nanoelectromechanical systems (NEMS) utilizing 2D material are potent instruments for ultra-sensitive mass spectroscopy, the onset of nonlinearities severely reduces their dynamic range. However, strain tuning of dynamic range is possible if the strain is introduced by methods other than electrostatic gating. Here, we present a method for the fabrication of nanoelectromechanical resonators (NEMRs) from 2D materials on flexible substrate, which allows straining devices through substrate bending, which is independent of electrostatic excitation of the of the resonator. This device platform can be a basis for studying dynamic range of NEMRs as a function of strain in the resonating membrane, along with studying new novel concepts for sensors involving strain engineering. With the advent of new 2d materials having exotic strain dependent properties, strain engineering opens whole new set of opportunities for the sensing technologies employing NEMS, other than strain tunable dynamic range.

11:40am **MN+2D-WeM-12 Parametric Amplification in MoS₂ Drum Resonator, Parmeshwar Prasad¹, N Arora, A Naik**, Indian Institute of Science, India

Transition metal dichalcogenide (TMDC) materials offer an alternative to carbon based materials, due to their unique mechanical, electrical and optical properties [1]. Molybdenum disulphide (MoS_2) is one such material which is being explored for NEMS applications. It has ultra-low mass density of $3.3 \text{ fg}/\mu\text{m}^2$ and high Young's modulus 0.3 TPa . Furthermore, its semi-conducting property allow its mechanical motion to be transduced electrically. NEMS devices based on 2D materials perform exceptionally well in terms of quality factor at low temperatures. Quality factors (Q) as high as 10^5 have been observed at cryogenic temperatures[2]. However, at room temperatures quality factors are typically pegged at 100. Low quality factor of these resonators make them difficult to transduce the motion of these resonators and thus hinder applications as potential ultra-sensitive detectors. In this paper, we amplify the motion of these resonators by parametric amplification. We report enhancement of mechanical response in MoS₂ drum resonator using parametric amplification and achieve $\sim 10\text{dB}$ gain. We also show quality factor enhancement in the resonator with parametric amplification at 397 K. The signal enhancement is similar to the recently reported NEMS devices [2], However, the amplification is significantly lower as compared to the reported MEMS devices [3]. We investigate the effect of Duffing (cubic) non-linearity in the current work and show that it plays significant role in the maximum achievable gain from NEMS devices using parametric amplification. The experiments are performed using direct capacitive measurement technique on near insulating $\sim 1\text{G}\Omega$ device. This shows the ability to perform electrical capacitive actuation and detection technique in very high resistance NEMS devices.

References:

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12:00pm **MN+2D-WeM-13 Anisotropic Thermal Conductivity of Suspended Black Phosphorous Probed by Opto-thermomechanical Resonance Spectromicroscopy, Arnob Islam², P Feng**, Case Western Reserve University

Two-dimensional (2D) black phosphorus (P) exfoliated from its layered bulk crystals has attracted great attention due to its unique in-plane anisotropic properties along armchair (AC) and zigzag (ZZ) directions [1-2]. Probing the anisotropic properties in the black P is important for both exploring fundamental science and engineering device performance. Here, we employ 2D nanoelectromechanical systems (NEMS) platform to study anisotropic thermal conductivity (k) of black P.

In this study, for the first time, we use thermomechanical motion with localized laser heating (Fig. 1a) (*opto-thermomechanical spectromicroscopy*) in combination with finite element modeling (FEM) to precisely determine anisotropic k_{AC} and k_{ZZ} of black P. We fabricate a black P circular drumhead resonator (thickness of $t \sim 80\text{nm}$ and diameter of $d \sim 9\mu\text{m}$) using a dry-transfer method [3]. Before resonance measurement, polarized reflectance measurement is performed to determine the crystal orientation of the black P flake (Fig. 1b) [4]. We then employ a 633nm laser (laser power of $P=1.6\text{mW}$, spot size of $1\mu\text{m}$) to photothermally heat up the device and interferometrically detect Brownian motion. We obtain the fundamental mode frequency at $f_{res} \sim 9\text{MHz}$ when the laser is located on the center of the device. We move the laser spot location along AC/ZZ on the resonator, and track f_{res} along the path. We find that measured f_{res} values are higher when laser spot is moving in AC direction ($f_{res,AC}$) than that in ZZ direction ($f_{res,ZZ}$) (Fig. 1c) at same distance from the center. This can be attributed to anisotropic k_{AC} and k_{ZZ} , which dictates different temperature distribution on the device as the laser is moving along AC/ZZ, providing uneven biaxial thermal expansion thus frequency shift.

We employ FEM simulation to model the coupling between thermal transport from optothermal heating and resonance characteristics of the black P drumhead resonator. By fitting the modeling to the experimental results, we are able to determine anisotropic thermal conductivities along

¹ MEMS/NEMS Best Paper Award Finalist

² MEMS/NEMS Best Paper Award Finalist

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AC/ZZ orientations ($k_{AC}=15\text{Wm}^{-1}\text{K}^{-1}$ and $k_{ZZ}=55\text{Wm}^{-1}\text{K}^{-1}$) (Fig. 1c and 1d), which are consistent with k_{AC} and k_{ZZ} obtained by other methods [2].

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2D Materials Focus Topic

Room 16 - Session 2D+EM+MN+NS-WeA

2D Device Physics and Applications

Moderator: Humberto Gutierrez, University of South Florida

2:20pm 2D+EM+MN+NS-WeA-1 Capacitance-voltage Characteristics of Graphene-gate MOS Devices: The Effect of Graphene Quantum Capacitance, *Ruixue Lian, A Ural, University of Florida*

There has been significant research interest in graphene for electronics applications, due to its good electrical conductivity, high optical transparency, mechanical flexibility, and two-dimensional structure. However, the potential of graphene as a channel material replacing silicon is limited due to the absence of a bandgap. On the other hand, graphene is an excellent candidate as a transparent, conductive, and flexible electrode for electronic and optoelectronic devices.

Unlike conventional metals, whose Fermi level is typically pinned at the surface, the Fermi level and hence workfunction of graphene can be tailored by electrostatic gating, chemical doping, or surface engineering. As a result, graphene is also a promising candidate as the gate electrode in metal-oxide-semiconductor (MOS) devices, particularly when transparency or workfunction tunability is a requirement.

In real graphene sheets, charged impurities cause electron-hole puddles and random local electrostatic potential fluctuations (statistically described by a Gaussian distribution), which leads to a modified density of states (DOS). In this work, using this modified DOS, we numerically compute the quantum capacitance of graphene as a function of the graphene electrostatic potential at different temperatures and strengths of the potential energy fluctuations. We compare the exact results to various approximations made in the literature when fitting experimental data. We find that the largest discrepancy between the exact results and the approximations occurs near the Dirac point.

In capacitance-voltage (C-V) characterization of graphene-gate MOS devices, what is measured is not the quantum capacitance versus the graphene potential, but the total gate capacitance versus the gate voltage. We numerically compute the gate voltage as a function of the graphene potential and the resulting C-V characteristics at different temperatures, strengths of the potential energy fluctuations, and equivalent oxide thicknesses. We also consider the effect of series and parallel parasitic impedance to the overall shape of the C-V curves. Furthermore, we numerically compute the full C-V characteristics at different values of the equivalent oxide thickness, silicon doping density, and Dirac voltage of graphene. Finally, we fit our recent experimental C-V data with these theoretical calculations to extract the strength of the potential energy fluctuations and the parasitic impedances.

These results provide important insights into the effect of the graphene quantum capacitance on the C-V characteristics of MOS devices and the potential of graphene as a gate electrode in future MOS technology.

2:40pm 2D+EM+MN+NS-WeA-2 *in-situ* Electrical Characterization of Surface Functionalization and Gate Dielectric Deposition Processes on 2D Transition Metal Dichalcogenides Transistors, *Antonio T. Lucero, J Lee, L Cheng, H Kim, S Kim, J Kim, University of Texas at Dallas*

Two-dimensional transition metal dichalcogenide (TMD) materials are a subject of intense research for use as future, low-power semiconductors. The successful fabrication of TMD based transistors requires a scalable dielectric deposition process. Atomic layer deposition (ALD) is commonly used to grow high-k gate dielectrics, though deposition of thin, pin-hole free dielectrics is challenging due to the chemically inert basal plane of most TMD materials. To overcome this limitation, surface functionalization processes have been developed to improve ALD nucleation.

In order to elucidate the effects of surface functionalization and subsequent ALD on the electrical characteristics of TMD transistors we use an *in-situ* electrical characterization system to measure the electrical properties of TMD transistors at various steps during the deposition process. MoS₂ backgated transistors are loaded into an ultra-high vacuum (UHV) cluster tool where samples can be transferred under UHV conditions between various chambers. The cluster tool is equipped with a thermal ALD chamber, a hollow cathode plasma enhanced ALD chamber, a plasma enhanced chemical vapor deposition chamber, and a UHV electrical probe station. Results for ozone, nitrogen radical, and nitrogen plasma functionalization will be presented. The effect of surface dipoles, precursor adsorption and coverage, and nucleation during the ALD process will be discussed as they relate to the electrical characteristics of the device.

This work was supported by the SWAN Center, a SRC center sponsored by the Nanoelectronics Research Initiative and NIST, and by NRF (No. 2015M3D1A1068061) in Korea. We thank TMEIC for providing the ozone generator and nitrogen radical generator used in this work.

3:00pm 2D+EM+MN+NS-WeA-3 High-K Gate oxide by Low Temperature ALD Technique for 2D Materials and Inert Metal Surfaces, *Il Jo Kwak, University of California at San Diego; J Park, University of California at San Diego, Republic of Korea; S Fathipour, A Seabaugh, University of Notre Dame; C Pang, Z Chen, Purdue University; A Kummel, University of California at San Diego*

2D materials such as TMDs (Transition Metal Dichalcogenides), Graphene and BN have attracted great attention as new channel materials for future devices due to their excellent electronic and optical properties. For such devices, sub nanometer thick and defect free gate oxide is an essential part. However, due to the inert surface of the materials, high K oxide such as Al₂O₃ and HfO₂ selectively nucleates on defect sites or step edges. Therefore, for successful integration, preparation of uniform and insulating gate oxides are a matter of importance. In this study, Al₂O₃ was deposited on 2D materials surface by low temperature ALD using trimethylaluminum (TMA) and H₂O without any seeding layer or surface treatments. Using short purge time between two precursor pulses at 50C, a CVD component was induced to provide uniform nucleation sites on the surface. The CVD component generates subnanometer AlO_x particles [s1] [file:///C:/Users/kwak1/Downloads/2017_AVS_abstract_bilayer_oxide.docx#_msocom_1] on the surface which provide uniform nucleation sites. In order to obtain lower EOT layer, 10 cycles of Al₂O₃ ALD was deposited at 50C as a seeding layer and 40 cycles of HfO₂ ALD was deposited with Tetrakis(dimethylamido)hafnium (TDMAH) and H₂O at 250C. The same oxide was deposited on a SiGe substrate to compare the oxide characteristics. After ALD, MOSCAPs were fabricated to measure electrical properties. AFM measurement revealed that uniform and defect free oxide layers were nucleated on the surfaces. Capacitance-voltage measurement showed that Cox of the bilayer oxide was 2.5 uF/cm² and the gate leakage current of the oxide was about 10⁻⁵ A/cm² which was comparable to the oxide on a SiGe substrate. Identical bilayer oxide layer was deposited on a dual gated WSe₂ FETs. Top gate oxide leakage of the device was about 10⁻⁶ A/cm². In order to assess the quality of the oxide, a benchmarking study of current density versus EOT of 2D semiconductor FET devices and Si based devices was investigated. The study showed that record-low EOT (1.2 nm) and leakage current (10⁻⁸ μA/μm²) comparable to the best Si devices with La₂O₃ gate oxide by Iwai *et al* was achieved by the WSe₂ FET. This technique was also applied to initiate nucleation [s2] [file:///C:/Users/kwak1/Downloads/2017_AVS_abstract_bilayer_oxide.docx#_msocom_2] on inert metal surfaces which are important for logic memory devices including selectors. Using the bilayer oxide, insulating oxide was prepared on Au electrodes of a MOSFET device. The leakage current of the oxide was as low as 10⁻⁷ A/cm².

3:20pm 2D+EM+MN+NS-WeA-4 Exploration and Comparison of Optoelectronic Properties of MoS₂ Monolayers with Multilayer Flakes and Mo_xW_{1-x}S₂ Ternary Compounds, *Sourav Garg, J Waters, A Mollah, S Kim, P Kung, University of Alabama*

2D transition metal dichalcogenide (TMDC) semiconductors, including MoS₂, WS₂, and more recently ternary compounds, exhibit exceptional structural, electrical and optical properties that make these materials of great interest for nano-optoelectronic devices. For example, unlike graphene, TMDCs have a bandgap, which has the remarkable characteristic of becoming direct when the material is in monolayer form, while it is indirect when the material is composed of multiple layers.

Here, we report the synthesis of monolayer MoS₂, WS₂, ternary Mo_xW_{1-x}S₂ ternary compounds and MoS₂/WS₂-based heterostructures, by chemical vapor deposition (CVD) process at temperatures in the range 950-1000 C, without the use of seeds to avoid contamination. The material was extensively characterized using micro-Raman spectroscopy, micro-photoluminescence, and electron microscopy.

Using such large area CVD grown materials, large-area MoS₂ photoconductive detector devices were fabricated using conventional photolithography to realize of interdigitated metal fingers. The electrical and spectral photoresponse from monolayer and multilayer MoS₂ have been compared, in terms of responsivity and specific detectivity. The

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monolayer devices exhibited high photoconductive gain and detectivity near 10^{12} Jones, which was also found to be higher than in the case of multilayer MoS₂ devices. The rise and decay time of passivated monolayer devices was investigated and shown to be much faster than the unpassivated devices.

4:40pm **2D+EM+MN+NS-WeA-8 Dielectric Properties of Carbon Nanomembranes prepared from aromatic Self-Assembled Monolayers investigated by Impedance Spectroscopy**, Paul Penner, E Marschewski, X Zhang, Bielefeld University, Germany; T Weimann, P Hinze, Physikalisches Technische Bundesanstalt, Germany; A Beyer, A Götzhäuser, Bielefeld University, Germany

Carbon nanomembranes (CNMs) are two-dimensional materials made by cross-linking self-assembled monolayers (SAMs) of aromatic molecules via low energy electron irradiation. Previous study of molecular junction incorporating SAMs and CNMs of oligophenyl thiols has been carried out by using conical eutectic Gallium-Indium (EGaIn) top-electrodes¹ and conductive probe atomic force microscopy (CP-AFM). Here we investigate the dielectric properties of pristine SAMs and CNMs with an EGaIn top electrode by impedance spectroscopy. Analysis and comparison of the tunneling resistance and capacitance density of pristine and cross-linked SAMs revealed a thickness dependent capacitance associated with the monolayer as well as a thickness independent capacitance. We adopted an equivalent circuit to take into account the contribution of the interfacial capacitance as well as the oxide layer of the EGaIn top electrode. The obtained tunneling decay constant remains unaffected after electron irradiation, which exhibits a value of about 0.5 \AA^{-1} for both systems. A determination of dielectric constants of SAMs and CNMs from the impedance spectra will also be analyzed and discussed. Furthermore we characterize stacks of CNMs sandwiched with graphene and other 2D materials.

¹ P. Penner, X. Zhang, E. Marschewski, et. al, J Phys Chem C 2014, 118, 21687.

5:00pm **2D+EM+MN+NS-WeA-9 2D Crystals for Next-Generation Ultra Energy-Efficient Electronics**, Kaustav Banerjee, University of California at Santa Barbara

INVITED

I will highlight the prospects of two-dimensional (2D) materials for innovating energy-efficient transistors, sensors, and interconnects targeted for next-generation electronics needed to support the emerging paradigm of the *Internet of Things*. More specifically, I will bring forward a few applications uniquely enabled by 2D materials and their heterostructures that have been demonstrated in my lab for realizing ultra-energy-efficient electronics. This will include the world's first 2D-channel band-to-band tunneling transistor that overcomes a fundamental power consumption challenge in all electronic devices since the invention of the first transistor in 1947 (Nature 2015), as well as a breakthrough interconnect technology based on doped-graphene-nanoribbons, which overcomes the fundamental limitations of conventional metals and provides an attractive pathway toward a low-power and highly reliable interconnect technology for next-generation integrated circuits (Nano Letters 2016). I will also bring forward a new class of ultra-sensitive and low-power sensors as well as area-efficient and high-performance passive devices, both enabled by 2D materials, for ubiquitous sensing and connectivity to improve quality of life.

Electronic Materials and Photonics Division

Room 14 - Session EM+2D+MI+MN-WeA

Materials and Devices for Quantum Information Processing

Moderators: Rachael Myers-Ward, U.S. Naval Research Laboratory, Steven Vitale, MIT Lincoln Laboratory

2:20pm **EM+2D+MI+MN-WeA-1 Controlling the Valley Degree of Freedom in 2D Transition Metal Dichalcogenides**, Tony Heinz, Stanford University / SLAC National Accelerator Laboratory

INVITED

Monolayer transition metal dichalcogenide crystals in the MX₂ family with M = Mo, W and X = S, Se have been shown to provide attractive possibilities for access to the valley degree of freedom both optically and through the valley Hall effect. In this paper we will summarize recent advances in the electrical and optical control of the valley degree of freedom in this class of materials.

The optical selection rules in the transition metal dichalcogenide monolayers permit selective creation of excitons in either the K or K' valley

through the use of circularly polarized light. Excitons consisting of coherent superpositions of both valleys can also be produced through excitation with linearly polarized light. While these results have already been demonstrated experimentally, to date there has been no report of an approach to *manipulate* the valley exciton pseudospin after its creation. In this paper we present our recent use of the optical Stark effect to dynamically modify the valley pseudospin. The approach is based on selectively altering the energy of one valley vis-a-vis the other through application of a sub-gap optical pulse with circular polarization. This perturbation leads to a rapid rotation of the exciton valley pseudospin, as revealed by a change in the polarization state of the exciton emission.

In a second line of investigation, we have applied to spin-valley Hall effect in transition metal dichalcogenide monolayers to produce spatially separated regions with enhanced valley (and spin) populations. This is achieved by running a current through a hole-doped monolayer and relying on the anomalous velocity terms to separate the holes spatially. The resulting spin-valley spatial profile has been directly imaged on the micron scale and characterized using measurements based on the optical Kerr effect. The magnitude of this spin-valley imbalance and its dependence on doping and bias fields have been investigated and compared with theoretical predictions.

3:00pm **EM+2D+MI+MN-WeA-3 VOI-based Valleytronics in Graphene**, Yu-Shu Wu, National Tsing-Hua University, Taiwan, Republic of China

INVITED

Electrons in gapped graphene carry a unique binary degree of freedom called valley pseudospin, in association with the two-fold valley degeneracy at the Dirac points (K and K') of Brillouin zone. Such pseudospin carries an intrinsic angular momentum and responds to external electromagnetic fields in ways similar to those of an ordinary electron spin [1,2]. We examine the response and address the important issue of valleytronics - the electrical manipulation of valley pseudospin. A unified methodology called VOI based valleytronics will be presented, which exploits the valley-orbit interaction (VOI) between an in-plane electric field and a valley pseudospin for the implementation of valleytronics. Based on the VOI mechanism, a family of fundamental structures have been proposed with important device functions, such as valley qubits, valley filters, and valley FETs [3]. We will report recent theoretical developments in these structures.

[1] Rycerz et al., Nat. Phys. **3** (2007),172.

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[3] Wu et al., Phys. Rev. B **84**, (2011), 195463; *ibid* B **86** (2012), 165411; *ibid* B **88** (2013), 125422; *ibid* B **94** (2016), 075407.

4:20pm **EM+2D+MI+MN-WeA-7 Creating Quantum Technologies with Spins in Semiconductors**, B Zhou, David Awschalom, University of Chicago

INVITED

There is a growing interest in exploiting the quantum properties of electronic and nuclear spins for the manipulation and storage of information in the solid state. Such schemes offer fundamentally new scientific and technological opportunities by leveraging elements of traditional electronics to precisely control coherent interactions between electrons, nuclei, and electromagnetic fields. Although conventional electronics avoid disorder, recent efforts embrace materials with incorporated defects whose special electronic and nuclear spin states allow the processing of information in a fundamentally different manner because of their explicitly quantum nature [1]. These defects possess desirable qualities - their spin states can be controlled at and above room temperature, they can reside in a material host amenable to microfabrication, and they can have an optical interface near the telecom bands. Here we focus on recent developments that exploit precise quantum control techniques to explore coherent spin dynamics and interactions. In particular, we manipulate a single spin in diamond using all-optical adiabatic passage techniques [2], and investigate the robustness of the acquired geometric (Berry) phase to noise as well as novel strategies to overcome traditional speed limits to quantum gating. Separately, we find that defect-based electronic states in silicon carbide can be isolated at the single spin level [3] with surprisingly long spin coherence times and high fidelity, can achieve near-unity nuclear polarization [4] and be robustly entangled at room temperature [5]. Finally, we identify and characterize a new class of optically controllable defect spin based on chromium impurities in the wide-bandgap semiconductors silicon carbide and gallium nitride [6].

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5:00pm **EM+2D+MI+MN-WeA-9 Diamond as an Electronic Material: Opportunities and Challenges**, *Steven Vitale, J Varghese, M Marchant, T Wade, M Geis, T Fedynyshyn, D Lennon, M Hollis*, MIT Lincoln Laboratory
Diamond possesses extraordinary semiconductor properties including carrier mobility, saturation velocity, and thermal conductivity which far exceed those of silicon and essentially all other semiconductor materials. In spite of these incredible qualities diamond has not yet become a mainstream transistor material, for two primary reasons. First, existing small single-crystal substrates have not been able to take advantage of commercial microelectronics processing equipment and growth of wafer-scale single-crystal diamond has not been vigorously pursued. Second, deep donor and acceptor levels in diamond imply that the impurity ionization fraction is quite low at room temperature which results in low carrier density in conventional FET architectures.

However the situation has changed dramatically in the past few years. Plasma-enhanced CVD promises to create large-wafer single-crystal diamond through mosaic or novel catalytic growth.¹ Additionally, the discovery of the diamond surface FET has addressed the problem of low carrier density.² Together, these advancements may allow development of practical diamond transistors with unparalleled performance for high-power, high-frequency applications. Many unit process and process integration challenges remain to develop diamond surface FETs into commercial technology. This paper will report on the state of the art in diamond surface FET technology and will examine current unmet needs.

We have developed diamond surface FETs with current densities in excess of 100 mA/mm. This is enabled by a novel surface activation process using a high concentration of NO₂ in air to react with a hydrogen-plasma-treated diamond surface. The electron accepting nature of the modified surface abstracts an electron from the diamond, resulting in a 2D hole gas (2DHG) in the diamond. We measure a hole mobility of 30-130 cm²/V-s and a repeatable surface resistance of ~ 1.5 kΩ sq⁻¹ using this technique. 2DHG formation has been demonstrated using other surface moieties as well, including photoacid radical generators and trinitrotoluene. Pros and cons of these different surface adsorbates will be discussed. The performance of Au, Mo, Pt, Al, Pd, Ti, Cr contacts, as well as combinations of these metals will be presented, with a record-low diamond contact resistance of 0.6 ohm-mm and good ohmic behavior.

¹ M. Schreck, et al, *Sci. Rep.* 7, 44462 (2017).

² M. Kasu, *Japanese Journal of Applied Physics* 56, 01AA01 (2017).

5:20pm **EM+2D+MI+MN-WeA-10 Studies on Influence of Processing on Optical Characteristics of Electron Irradiated 4H-SiC Nanostructures**, *Shojan Pavunny*, ASEE Research Fellow at U.S. Naval Research Laboratory; *H Banks*, NRC Research Fellow at U.S. Naval Research Laboratory; *P Klein*, U.S. Naval Research Laboratory; *K Daniels*, NRC Research Fellow at U.S. Naval Research Laboratory; *M DeJarld*, ASEE Research Fellow at U.S. Naval Research Laboratory; *E Glaser*, *S Carter*, *R Myers-Ward*, *D Gaskill*, U.S. Naval Research Laboratory

Spin-coherent single silicon defect centers (V_S) in wide bandgap silicon carbide polytypes have recently drawn great research interest for future applications in information technologies such as scalable quantum computing, sensing and metrology. Identification of these deep defects, gaining a thorough knowledge of their characteristics, active control of their concentrations, isolation of single spin defects and understanding the effects of semiconductor processing on their properties are crucial challenges for the realization of SiC based quantum electronic and integrated photonic devices. These color centers coupled to photonic crystal cavities (PCC) have the capability of high efficiency emission of zero phonon lines which can significantly improve the performance of on-chip photonic networks and long-distance quantum communication systems, as compared to conventional solid-state emitters. Here we investigate the impact of fabrication process on the photoluminescence properties of PCCs realized using three techniques: hydrogen implantation to form thin SiC

layers on an oxide layer that can be easily etched away to form an air gap under the PCC, wafer bonding and mechanical thinning of the SiC, also on an oxide layer, and selective electrochemical anodization of an n-p epitaxial SiC structure to form an air gap. We also comment upon the impact of electron irradiation for these three fabrication techniques.

5:40pm **EM+2D+MI+MN-WeA-11 Ab Initio Simulations of Point Defects in Solids Acting as Quantum Bits**, *Adam Gali*, Wigner Research Centre for Physics, Hungarian Academy of Sciences, Hungary **INVITED**
Luminescent and paramagnetic point defects in insulators and semiconductors may realize quantum bits that could be the source of next generation computers and nanoscale sensors. Detailed understanding of the optical and magnetic properties of these defects is needed in order to optimize them for these purposes.

In this talk I show our recent methodology developments in the field to calculate the ground and excited state of point defects and to determine their Auger-rates, hyperfine tensors and electron spin – electron spin couplings, and intersystem crossing rates. We show recent results on the nitrogen-vacancy center in diamond as well as divacancy and other defects in silicon carbide that we have found a very promising alternative to the well-established nitrogen-vacancy center for integration of traditional semiconductor and quantum technologies into a single platform.

Nanometer-scale Science and Technology Division Room 19 - Session NS+MN+MS+SS-WeA

Nanopatterning, Nanofabrication and 3D Nanomanufacturing

Moderator: Brian Borovsky, St. Olaf College

2:20pm **NS+MN+MS+SS-WeA-1 Site-controlled Si Nanodot Formation for a RT-SET via Ion Beam Mixing and Phase Separation**, *Xiaomo Xu¹, G Hlawacek, D Wolf, T Prüfer, R Hübner, L Bischoff*, Helmholtz Zentrum Dresden-Rossendorf, Germany; *M Peregó*, Institute for Microelectronics and Microsystems (IMM-CNR), France; *A Gharbi*, Laboratoire d'électronique des technologies de l'information (CEA-Leti), France; *H Engelmann, S Facsko, K Heinig, J von Borany*, Helmholtz Zentrum Dresden-Rossendorf, Germany

The increased use of personal computing devices and the Internet of Things (IoT) is accompanied by a demand for a computation unit with extra low energy dissipation. The Single Electron Transistor (SET), which uses a Coulomb island to manipulate the movement of single electrons, is a candidate device for future low-power electronics. However, so far its development is hindered by low-temperature requirements and the absence of CMOS compatibility. By combining advanced top-down lithography with bottom-up self-assembly of Si nano dots (NDs) we will overcome this barrier.

In this work, Si NDs – suitable as RT Coulomb islands – are formed via ion beam mixing followed by thermally stimulated phase separation. Broad-beam Si⁺ and Ne⁺ beams followed by a rapid thermal annealing (RTA) treatment were utilized to create a layer of NDs, which are subsequently visualized by Energy-Filtered Transmission Electron Microscopy (EFTEM). The conditions for ND formation, namely the dependence on ion type, primary energy, irradiation fluence, layer thickness and thermal budget during RTA, are optimized based on an extensive survey of this multidimensional parameter space. The presented work is guided by TRIDYN simulations of the Si excess in a SiO₂ layer due to ion beam mixing and 3D Kinetic Monte-Carlo (3DkMC) simulation for the phase separation during the thermal treatment. To tailor towards a single Si ND, the focused Ne⁺ beam from the Helium Ion Microscope (HIM) is utilized to create user defined patterns of NDs in planar layer stacks. This allows to achieve a mixing volume small enough for restricted Ostwald ripening and successful single ND formation. The existence of the formation of spatially controlled single NDs with a diameter of only 2.2 nm is confirmed by comparing the EFTEM Si plasmon-loss intensity with simulated plasmon loss images.

In the future – by combining conventional lithography, direct self-assembly (DSA) and ion beam mixing – nanopillars with a single embedded ND will be integrated in a CMOS-compatible way. EFTEM and electrical characterization techniques will be used for realizing this novel pathway towards a room-temperature SET device.

¹ NSTD Student Award Finalist

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2:40pm **NS+MN+MS+SS-WeA-2 Scanning Tunneling Microscope Fabrication of Atomically Precise Devices, Richard Silver, NIST; X Wang, University of Maryland, College Park; P Nambodiri, J Wyrick, S Schmucker, M Stewart, R Murray, J Hagmann, C Richter, NIST**

Atomically precise device fabrication is a technique that enables a new class of atom-based electronic structures with applications ranging from novel low dimensional materials to devices for quantum information processing. Deterministic placement of individual dopant atoms in the Si lattice is achieved using hydrogen-based scanning probe lithography. Controlling the position and electronic or quantum state of deterministically placed atoms in a solid state environment enables novel devices such as single atom transistors and solid state qubits.

However, fabricating functional atom-based devices is particularly challenging because of the need for exceptional ultra-high vacuum, near perfect atomic order, and low temperature epitaxial silicon overgrowth. This, coupled with sensitivity of atomic positional accuracy to thermal processing, and variability in scanning tunneling microscope patterning conditions, make exquisite control of process conditions essential.

In this presentation, we will focus on measurements and characterization of ultra-thin, atomically abrupt, highly doped low-dimensional devices and strategies for contacting these devices. We will describe our methods to align and contact buried devices and address significant challenges in making robust electrical contact to buried devices. We will present low-temperature electrical measurement results from atomically abrupt wires and tunnel junctions with coplanar gates. We have studied the effects of process conditions on device dimensionality and electrical performance in the context of extensive analysis of delta layer formation with optimized locking layer epitaxial growth techniques to enhance the confinement of Phosphorus dopant atoms. Low temperature transport measurements are used to investigate materials properties, effects from atomic imperfection and quantum transport phenomena.

3:00pm **NS+MN+MS+SS-WeA-3 Contacting Buried Atomic-Precision Devices in Si using Kelvin Probe and Optical Microscopy, Jonathan Wyrick, P Nambodiri, X Wang, R Murray, J Hagmann, K Li, S Schmucker, M Stewart, C Richter, R Silver, NIST**

STM based hydrogen lithography has proven to be a viable route to fabrication of atomic-precision planar electronic devices. These devices are realized by a patterning step followed by dopant deposition and incorporation, and ultimately encapsulation with epitaxial Si. Atomically precise tunnel junctions, SETs, and Quantum Dots are examples of components that can be fabricated using hydrogen lithography.

The strength of this technique is the ability to control the lateral placement of phosphorus atoms in a single atomic layer of Si with sub-nanometer precision. At the same time, it presents challenges that must be overcome if devices are to be interfaced to the outside world. Locating and then fabricating aligned electrical contacts to buried devices is non-trivial, and becomes easier as the size of buried features is increased, but this is done at the expense of increased writing times and exposure to potential contamination.

We present a strategy for contacting buried devices aimed at minimizing the write-times associated with STM based fabrication by maximizing the positional accuracy with which we can locate subsurface structures. This is done by employing STM fabricated fiducials, AFM topography scans, Kelvin Probe Microscopy, and dark field optical microscopy. The data from each technique can be aligned and corrected for distortions, allowing us to determine buried device locations to better than 200nm accuracy.

3:20pm **NS+MN+MS+SS-WeA-4 Quantifying Liquid Transport and Patterning using Atomic Force Microscopy, N Farmakidis, Keith Brown, Boston University**

Atomic force microscopy (AFM) provides unique insight into the nanoscale properties of materials. It has been challenging, however, to use AFM to study soft materials such as liquids or gels because of their tendency to flow in response to stress. Here, we propose an AFM-based technique for quantitatively analyzing the transport of soft materials from an AFM probe to a surface. Specifically, we present a method for loading an AFM probe with a single 0.3 to 30 pL droplet of liquid, and subsequently measuring the mass of this liquid by observing the change in the vibrational resonance frequency of the cantilever. Using this approach, the mass of this liquid was detected with pg-scale precision using a commercial AFM system. Additionally, sub-fL droplets of liquid were transferred from the probe to a surface with agreement found between the real-time change in mass of the liquid-loaded probe and the volume of the feature written on the surface.

To demonstrate the utility of this approach in studying nanoscale capillary and transport phenomena, we experimentally determine that the quantity of liquid transported from the tip to a surface in a given patterning operation scales as the mass of liquid on the probe to the 1.35 power. In addition to providing new avenues for studying the dynamics of soft materials on the nanoscale, this method can improve nanopatterning of soft materials by providing *in situ* feedback.

4:20pm **NS+MN+MS+SS-WeA-7 Positioning and Manipulating Single Dopant Atoms Inside Silicon, Andrew Lupini, B Hudak, J Song, Oak Ridge National Laboratory; H Sims, Vanderbilt University; C Troparevsky, Oak Ridge National Laboratory; S Pantelides, Vanderbilt University; P Snijders, Oak Ridge National Laboratory**

INVITED

The ability to controllably position single atoms inside materials could enable production of a new generation of atomically precise artificial materials with direct relevance for many areas of technology. For example, spins from individual donors in a semiconductor comprise one of the most promising architectures for quantum computing. However, fabrication of the 'qubits' that would make up a quantum computer is still unreliable and many fundamental materials science questions remain unanswered. Perhaps the key technical difficulty is the task of accurately positioning single atom dopants inside a solid with control, or at least understanding, of their local environment.

Silicon is the ideal substrate to explore such ideas because of the ability to obtain isotopically purified samples (a "spin-vacuum") and compatibility with existing electronic components and manufacturing technologies. Group V elements are promising candidates for use as single-atom qubit dopants in Si, and it has recently been argued that bismuth could be an excellent dopant for such applications, because of its anomalously high spin-orbit coupling. Bi, in particular, has a large atomic number relative to Si, making it an ideal candidate to study using Z-contrast scanning transmission electron microscope (STEM). However, both precise doping and the imaging of single dopant atoms present many scientific challenges. For example, Bi is not very soluble in Si, meaning that the dopant atoms tend to migrate out of position during sample growth.

Here we will show examples of sample growth including novel nanostructures and single atom dopants. We will show dopant atoms imaged inside Si samples, and demonstrate electron-beam directed movement of single dopants.

Research sponsored by US Department of Energy, Office of Science, Basic Energy Sciences, Materials Sciences and Engineering Division; and by the Laboratory Directed Research and Development Program of Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U. S. Department of Energy. DFT calculations were supported by DOE Grant No. DE-FG02-09ER46554.

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5:00pm **NS+MN+MS+SS-WeA-9 Characterization of Butyl Tin Photoresists for Nanoscale Patterning, J Diulus, R Frederick, Oregon State University; M Li, Rutgers University; D Hutchison, M Olsen, I Lyubinetsky, L Árnadóttir, Oregon State University; E Garfunkel, Rutgers University; M Nyman, Oregon State University; H Ogasawara, SLAC National Accelerator Laboratory; Gregory Herman, Oregon State University**

Inorganic photoresists are of interest for nanomanufacturing due to the potential for high resolution patterning with low line edge roughness, while having high sensitivity to extreme ultraviolet (EUV) radiation. The combination of high absorption coefficient elements and radiation sensitive ligands can improve inorganic photoresist sensitivity while providing high contrast. Inorganic clusters are ideal candidates for photoresists since they have nanometer particle sizes with high particle size uniformity, and the ligand chemistries can be tuned for radiation induced chemistries that control relative solubility differences. In this presentation, characterization of a promising inorganic cluster-based EUV photoresist will be presented, where the goal of the studies is to better understand patterning mechanisms. In these studies, we are investigating butyl tin Keggin cluster that has recently been synthesized, and has shown

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promising properties as an inorganic photoresist. Key to these clusters, for application as an EUV photoresist, are the high EUV absorption coefficient for Sn, and the radiation sensitive carbon-tin bond. Removal of the organic ligand changes the polarity of the film, which provides the necessary solubility contrast for nanopatterning. We have used temperature programmed desorption, electron stimulated desorption, and ambient pressure X-ray photoelectron spectroscopy to characterize both thermal and radiation induced processes in thin films formed from these clusters. We have found that butyl group desorption occurs through both thermal and radiation induced processes, and have determined both the carbon-tin bond strength and electron desorption cross-sections. Studies performed in different ambient conditions, and photon energies, have shown large effects on the radiation induced chemistries, where a significant enhancement in carbon decay was observed for O₂ pressures up to 1 torr. These studies provide a means to better understand the radiation induced processes that result in the solubility contrast of these materials, and may guide in the development of improved EUV photoresists for nanolithography.

5:20pm **NS+MN+MS+SS-WeA-10 Impact of Polymer Templated Annealing on Gold Nanowires**, *Tyler Westover, R Davis, B Uptrey, J Harb, A Woolley, S Noyce*, Brigham Young University

The formation of gold nanowires using bottom up nanofabrication has resulted in wires of small dimension or high conductivity, but not both. We form nanowires on DNA origami through directed assembly of nanoparticles or nanorods followed by electrochemical plating. These metal deposition processes result in non-ideal microstructure and correspondingly low conductivities. To remedy this we have sought to reduce the grain boundary density and surface roughness through annealing. However annealing causes the wires to coalesce into beads. We have found that through the use of a polymer the wires can be templated to retain their overall morphology, while improving surface roughness, throughout a low temperature anneal. We have measured these wires to have less than 1kOhm resistances by electron beam lithography, in a two point configuration. Using electron beam induced deposition we have successfully made four point contacts to measure the change in resistance due to annealing. We will present results on polymer templating, showing that the wires maintain their overall morphology with improved conductivities during low temp (200° C) annealing.

5:40pm **NS+MN+MS+SS-WeA-11 Dynamic Growth of Nanopores on Graphene via Helium Ion Microscope**, *S Kim, Anton Ievlev, M Burch, I Vlasiouk, A Belianinov, S Kalinin, S Jesse, O Ovchinnikova*, Oak Ridge National Laboratory

Controlling atomic-to-nanoscale defect formation on graphene is of significance as defects can modify properties as well as functionality of graphene. Especially, controlled formation of nanopores in graphene can be used for energy harvesting/storage, analysis of biomolecules and the separation of gases or liquids. Nanopores can be fabricated either by using high energy focused electron beam or by focused helium ion beam with high precision. However, focused electron beam has very low throughput to form nanopores despite its superiority in pore size control. On the contrary, focused helium ion beam has much higher throughput in nanofabrication with its capability to form sub-5nm pores. In this study, we utilized the focused helium ion microscope to fabricate nanopores on graphene and demonstrated atomic scale control in growth of nanopores by helium ion irradiation. We demonstrated the size control of nanopores down to ~ 3nm in a diameter. Formation and growth kinetics of nanopores by different helium ion irradiation conditions were explored and analyzed using the image data analytics. Also, Raman spectroscopic measurements was performed to demonstrate the effect of a helium ion dose on the change of initial defect density on graphene which leads to different behaviors and growth kinetics of nanopore formation.

This work was supported by the Oak Ridge National Laboratory's Center for Nanophase Materials Sciences (CNMS), which is a U.S. Department of Energy, Office of Science User Facility.

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