## **Tuesday Morning, October 20, 2015**

#### MEMS and NEMS

Room: 211A - Session MN+MG-TuM

#### Multiscale Phenomena & Interactions in Micro- and Nano-Systems (8:00-10:00 am) & Optical MEMS/NEMS, Photonics, and Quantum Nanosystems (11:00 am-12:20 pm)

**Moderator:** Robert Davis, Brigham Young University, Robert Ilic, National Institute of Standards and Technology (NIST), Meredith Metzler, University of Pennsylvania

#### 8:00am MN+MG-TuM1 Microengineering for Mechanobiology, Beth L. Pruitt, Stanford University INVITED

Living organisms generate and respond to mechanical forces and these forces are sensed and created by specialized cells in the body. Force generation and sensing, or more broadly the mechanobiology coupling tissue (cell) mechanics and biology, are essential in normal development, wound healing, and tissue homeostasis. Our mechanical senses of hearing and touch allow us to navigate our environment and interact with one another, yet they remain the least understood of our perceptive senses. Basic life sustaining functions such as breathing, circulation, and digestion are driven autonomously by coordinated contraction of specialized muscle cells, yet how these functions incorporate active feedback via force sensing at the cellular level is an area of active study. Meanwhile, a variety of specialized stretch activated receptors and mechanically mediated biochemical signaling pathways have been identified in recent years. Importantly, defects in proteins of these mechanically mediated pathways and receptors have been implicated in disease states spanning cardiovascular disease, cancer growth and metastasis, neuropathy, and deafness. Thus, understanding the mechanical basis of homeostasis (health) and defective cell renewal function (disease) increasingly requires us to consider the role of mechanics. To study how cells and tissues integrate mechanical signals, we and others have developed specialized cell cultures systems and micromachined tools to stimulate and measure forces and displacements at the scale of proteins and cells. A key feature of such experiments is the ability to observe cell outputs such as morphological changes, protein expression, electrophysiological signaling, force generation and transcriptional activity in response to mechanical stimuli.

#### 8:40am MN+MG-TuM3 Introducing Students to MEMS: A Practical Process for the Fabrication and Testing of Piezoresistive Cantilevers, *Frederic Loizeau*, E. Sadeghipour, T. Larsen, J.Y. Sim, C. Roozeboom, E. Mazzochette, B.L. Pruitt, Stanford University

We present a laboratory course to introduce students to Micro-Electro-Mechanical Systems (MEMS) through fabrication and characterization of piezoresistive cantilevers. We developed a process flow comprised of only three photolithography steps to minimize time spent by the students in the cleanroom and workload of the teaching team. Students performed handson work on over 80% of the fabrication process and thus earned qualification status to operate the standard tools in our cleanroom. The course included practical experience with signal conditioning, noise, and sensitivity measurements. The lab component spanned six sessions of 4.5 hours each and is ideal for integration in a lecture course or a two-week standalone mini-course.

The hands-on laboratory component was paired with lectures covering cleanliness, process selection, and device design and characterization. Six lab sessions of 4.5 hours each covered the fabrication and characterization of piezoresistive cantilevers. In each session, teams of five students learned fundamental MEMS processes and equipment use while fabricating predesigned devices. Weekly homework reinforced design, process, and testing concepts, e.g., predicting device performance, completing lithography steps, or building a measurement circuit. Device fabrication was completed in the first four lab sessions. Modest process support was provided outside of class by the teaching team for batch processes such as wafer preparation, metallization, and final HF release. The yield of the fabrication process was >90%. Each team built their own Wheatstone bridge and amplifier circuit to readout the piezoresistor signal prior to lab sessions 5 and 6 for device characterization. In session 5 they learned to use a dynamic signal analyzer to measure the Hooge and Johnson noise. In session 6, the students measured the power spectral density of cantilever tip deflection due to thermomechanical noise using a laser-Doppler vibrometer (LDV). From these measurements, they estimated the spring constants, resonant frequencies and quality factors of the cantilevers. Using a piezoelectric shaker and the LDV, students simultaneously measured the cantilever deflection and the piezoresistor bridge output and then used this to calculate cantilever sensitivity and resolution. Finally, experimental measurements were compared with theoretical predictions.

9:00am MN+MG-TuM4 Deflection Control of an Electroactive Polymer Bimorph Actuator by Carrier Frequency Modulation, *Leeya Engel*, Tel Aviv University, Israel, *K. Van Volkinburg*, University of California Irvine, *Y. Shacham-Diamand*, Tel Aviv University, Israel, *G.N. Washington*, University of California Irvine, *S. Krylov*, Tel Aviv University, Israel

In microelectromechanical systems (MEMS), actuator deflections are typically controlled by varying the voltage used to drive the active element. In this work, we use the frequency sensitivity of the permittivity of relaxor ferroelectric polymer poly(vinylidene fluoride-trifluoroethylene chlorotrifluoroethylene (P(VDF-TrFE-CTFE)) as an additional parameter for controlling the deflections of an electroactive polymer bimorph actuator.

The amplitude of the tip deflection of the electroactive polymer bimorph actuator, whose active layer comprised a thin film of P(VDF-TrFE-CFE), increased with the voltage applied at constant frequency, as expected. When the peak-to-peak displacements of the beam were plotted as a function of frequency at constant peak-to-peak voltage, a nonlinear decrease in tip deflection with increasing frequency was observed, independent of the resonance of the device. Electrical characterization of the material shows that the real component of the permittivity is  $\sim$ 55.5 at 100 Hz, but at radio frequencies, it decreases to 4. Dielectric losses are high at frequencies on the order of kHz-GHz with a coefficient of loss above 60% around MHz frequencies. Thus, the decrease in magnitude of electromechanical displacement with frequency can be attributed to the decrease in the permittivity-dependent electric field related electrostrictive coefficient with frequency. Deflections were recorded using both a laser Doppler vibrometer (LDV) and by interpreting the potential difference that formed across an integrated layer of piezoelectric polymer PVDF during actuation. In addition to adding mechanical sensing capabilities to the device, the PVDF layer also functioned as the passive layer of the bimorph structure.

This work directly demonstrates the dependence of the electromechanical behavior of an electroactive polymer actuator on the dielectric properties of P(VDF–TrFE–CFE) and our ability to exploit that dependence for an additional control parameter of the device. Frequency modulation of polymer beam deflections and integration of sensing capabilities can benefit the developing field of polymer microactuators, in applications such as "smart" prosthetics and implants, targeted drug delivery, tools for less invasive surgery, microfluidics, and on-chip cooling.

# 9:20am MN+MG-TuM5 Solder Based Self-Assembly Method For 3D Integration Using Poly-Acrylic Acid, Connor Smith, Y. Feng, S.L. Burkett, The University of Alabama

The use of Solder Based Self-Assembly (SBSA) in fabricating 3D structures on the microscopic scale is a process with numerous potential applications. This method involves creating copper plated 2D flat patterns of various shapes on a silicon substrate. Then, upon dip soldering these patterns and re-flowing the solder with hydrochloric acid, surface tension pulls up on these shapes to form a 3D structure. However, the use of a SiO2 sacrificial layer in performing this method results in the need for hydrofluoric acid (HF) during the etching phases, which has many dangerous hazards associated with it. The goal of this research is to develop a new process in which a water-soluble polymer, poly-acrylic acid (PAA), may be used as a sacrificial layer instead of SiO2--thus making the microfabrication process much safer. By working through the original SBSA method, and overcoming the various obstacles created by needing to protect the PAA from being exposed to water earlier than desired, an effective procedure has been developed. Through completing this project, future attempts to fabricate microscopic 3D structures using the SBSA method will be safer and less prone to dangerous HF exposure. Furthermore, removing HF etching from the procedure will reduce the time required to move through the process as a whole, thus increasing its efficiency.

# 11:00am MN+MG-TuM10 Mechanics and Spins in Diamond, A. Bleszynski Jayich, Donghun Lee, University of California at Santa Barbara INVITED

Single crystal diamond mechanical resonators have recently emerged as a promising platform for hybrid quantum systems comprising spins and phonons. Diamond mechanical resonators exhibit exceptionally high quality factors<sup>1</sup> and diamond plays host to a highly coherent spin system: the nitrogen vacancy (NV) center. The NV center is an atom-sized defect in diamond that is a remarkably good sensor of magnetic, electric, thermal, and strain fields on the nanoscale. Because of its strain sensitivity, the NV

can be easily coupled to a mechanical degree of freedom. We have recently characterized the sensitivity of the NV's ground state spin to strain by controllably applying dynamical strain to NV centers embedded inside high quality factor diamond mechanical resonators<sup>2</sup>. We have also recently demonstrated strain-mediated coupling to the optical transitions of single NV centers. Through strain coupling, we show that coherent mechanical control of individual spins in diamond is possible. These results are encouraging for proposals to use such a spin-mechanical platform for spin-squeezing, phonon-mediated spin-spin interactions<sup>3</sup>, and phonon cooling of macroscopic mechanical resonators<sup>4</sup>. We discuss the necessary steps needed to reach these goals and current progress including improvements in diamond fabrication, NV formation, and readout techniques.

1. Ovartchaiyapong, P., Pascal, L. M. A., Myers, B. A., Lauria, P. & Bleszynski-Jayich, A. C. High quality factor single-crystal diamond mechanical resonators. *Applied Physics Letters***101**, 163505 (2012).

2. Ovartchaiyapong, P., Lee, K. W., Myers, B. A. & Jayich, A. C. B. Dynamic strain-mediated coupling of a single diamond spin to a mechanical resonator. *Nat Comms***5**, (2014).

3. Bennett, S. *et al.* Phonon-Induced Spin-Spin Interactions in Diamond Nanostructures: Application to Spin Squeezing. *Physical review letters***110**, 156402 (2013).

4. Kepesidis, K. V., Bennett, S. D., Portolan, S., Lukin, M. D. & Rabl, P. Phonon cooling and lasing with nitrogen-vacancy centers in diamond. *Physical Review B88*, 064105 (2013).

#### 11:40am MN+MG-TuM12 Nano-Optomechanical Fin Resonators Designed for Sensing in Liquid Environments, *Jocelyn Westwood-Bachman*, *W.K. Hiebert*, University of Alberta and The National Institute for Nanotechnology, Canada

Nanomechanical systems are well known for their mass sensitivity, and are often used as mass sensors [1]. However, nanomechanical sensors tend to operate poorly in liquid environments due to viscous damping by the surrounding fluid. This drawback is particularly challenging for biological and related clinical sensing applications, where it is ideal to detect molecules within a liquid environment [2]. Here, we show the design of a fin-like nanomechanical resonator specifically for use in liquid environments. This design features a cantilever pointing out of the plane of the silicon device layer. This is in contrast to typical cantilevers that are in the silicon plane. The length of the cantilever is determined by the thickness of the silicon layer used, and the thickness of the resonator is designed to achieve specific resonance frequencies. The motion of these fin-like resonators is read out by an adjacent photonic microring resonator [3]. This microring resonator also provides an avenue for optical actuation of the fin resonator. The benefit of this design over existing designs is twofold. Firstly, our integrated photonics detection and actuation scheme provides higher displacement sensitivity than interferometric techniques [4]. Secondly, the fin is designed to operate at high frequencies (above 500 MHz) but can still have comparable surface area to nanoscale cantilevers as the width can be made arbitrarily large. This increases the sensing area while reducing the overall dissipation [5]. We will illustrate our design methodology and show the first generation of devices. As the as-fabricated devices have larger-than-desired feature sizes due to the limitations of photolithography, we will also discuss potential methods of tuning the device size post-fabrication. Specifically, we explore the possibility of trimming the fin resonator using Ga and He ion milling.

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[2] J. Tamayo et al, Chem. Soc. Rev. 42, 1287 (2013)

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#### 12:00pm MN+MG-TuM13 Directed Magnetic Optical Resonator Microballoons for Particle Imaging Manometry in 3D Environment, *Niladri Banerjee*, University of Utah

Measurement of velocity and pressure field in microfluidic 3D environment is vital in complete characterization of any fluid flow for capillary networks, flow-based separators and microchips for different biological applications. Particle imaging velocimetry though is the gold standard for measuring in-flow velocity, there has been no equivalent technique to perform pressure mapping. Recently hollow spherical micro-particles were fabricated to perform pressure measurement inside microfluidic channels. But lack of control on the trajectory of these particles inside micro-channels resulted in the ability to perform on-the-fly in-flow pressure mapping by spectroscopic method at any arbitrary location.

In this paper, we present the design, fabrication and testing of engineered magnetic micro-balloon pressure sensor particles. These directed particles, when injected into the flow-stream of any microchip, can be localized at

any specific location of interest for dynamic pressure measurement. Each particle consists of a vacuum sealed spherical cavity along with a goldnickel-gold magnetic tail attached to a polymeric support stem. The hollow cavity sealed by a thin polymeric shell, behaving as a Fabry-Perot interferometer, changes in size due to external pressure variation, which is detected by spectroscopic technique. Moreover the magnetic tail enables temporary immobilization of these particles at any position in the channel by the application of external magnetic field. The fabrication of these particles is based on buried sphere technology (BST). The fabrication starts with patterning of circular holes on thermally oxidized silicon. A trench 10-15 µm deep into the substrate is etched by DRIE. Next we oxidize the sample to grow 100 nm of oxide in order to protect the sidewalls of the trench. On selectively etching oxide from trench-bottom wall, spherical cavity of 6 µm radius is etched by XeF<sub>2</sub>. Then the spherical cavity is smoothened and hole necked down by subsequent oxidation and polydeposition process steps. Al<sub>2</sub>O<sub>3</sub> is then deposited by ALD forming a gas leakage-stop layer followed by parylene-C, to form the micro-balloon wall. Then a gold-nickel-gold (0.2-0.5-0.2  $\,\mu\text{m})$  sandwich is sputtered and encapsulated using another 1 µm layer of parylene-C. Real-time in-flow pressure measurement using 0.1T permanent magnet is performed at 8 magnet-specified locations with particles dispersed in IPA inside a serpentine test-chip. Spectral reflectance measurement indicates a pressure sensitivity of 37nm/psi. The paper will discuss the fabrication and test of the magnetic particles in detail with additional internal pressure measurement examples.

### Tuesday Afternoon, October 20, 2015

#### Electronic Materials and Processing Room: 210E - Session EM+MN+PS-TuA

#### More than Moore: Novel Approaches for Increasing Integrated Functionality

**Moderator:** Andy Antonelli, Nanometrics, Sean King, Intel Corporation

#### 2:20pm EM+MN+PS-TuA1 Maintaining the Pace of Progress as we Approach the end of Moore's Law: *Heterogeneous Integration, New Materials, New Processes, New Architectures, Bill Bottoms,* 3MTS INVITED

The environment is rapidly changing as we approach the end of Moore's Law scaling. Scaling continues but benefits in performance, power and cost are reduced. At the same time drivers for the electronics industry are impacted by the emerging Internet of Things and Migration to the Cloud. Satisfying the requirements of these emerging drivers cannot be accomplished with the current technology. It will require innovative heterogeneous integration approaches to satisfy demands for power, latency, bandwidth, reliability and cost in an environment where transistors will wear out.

Overcoming the limitations of the current technology will require heterogeneous integration using different materials, different device types (logic, memory, sensors, RF, analog, etc.) and different components incorporating multiple technologies including electronics, photonics, and MEMS in new, 3D, system-in-package (SiP) architectures. New materials, manufacturing equipment and processes will be required to accomplish this and meet the market demand for continuous reduction in cost per function.

The requirements, difficult challenges and potential solutions will be discussed.

#### 3:00pm EM+MN+PS-TuA3 More than Moore - Wafer Scale Integration of Dissimilar Materials on a Si Platform, *Thomas Kazior*, J. LaRoche, Raytheon Company INVITED

Advances in silicon technology continue to revolutionize microelectronics. However, Si cannot do everything, particularly for high performance, high frequency RF and mixed signal applications. As a result circuits based on other materials systems, such as III-V semiconductors, are required. However, these other device technologies do not enjoy the integration density, cost benefit and manufacturing infrastructure of Si. So how can we get the 'best of both worlds'? What is the best way to integrate these dissimilar materials with Si? In this paper, we review different heterogeneous integration approaches and summarize our results on the successful wafer-scale, 3D heterogeneous integration (3DHI) of GaN HEMTs and Si CMOS.

Our Au-free GaN HEMTs have been successfully fabricated entirely in a Si foundry on semi-standard, 200 mm diameter Si wafers using Cu damascene interconnects. RF performance compares favorably with GaN on SiC devices fabricated in a III-V foundry with Au-based contact and interconnect metallurgy. Oxide bonding is being used to integrate these GaN on Si wafers with Si CMOS wafers. Through-dielectric-vias (TDVs) are used to interconnect the high performance GaN RF devices/circuits with high density CMOS control and logic circuits, resulting in ultra-short, widebandwidth interconnects enabling circuit optimization through intimate and arbitrary placement of CMOS logic and control circuitry relative to III-V devices. Through-substrate-vias (TSVs) are used for thermal management. This 'flexible' wafer-scale, integration platform is compatible with other III-V devices, other (non-Si) device/component technologies and any node of Si CMOS or SiGe BiCMOS. The 3DHI process is being used to fabricate cost effective, high performance, digitally enhanced, RF and mixed signal ICs such as 'intelligent' and adaptive/reconfigurable transceivers.

MEMS and NEMS Room: 211A - Session MN+BI-TuA

#### **BioMEMS/NEMS, Wearable and Implantable Devices**

**Moderator:** Wayne Hiebert, University of Alberta and The National Institute for Nanotechnology, Beth L. Pruitt, Stanford University

#### 2:20pm MN+BI-TuA1 Entrepreneurial Environment for Implantable and Wearable BioMEMS, Kurt Petersen, Silicon Valley Band of Angels INVITED

Several converging trends are transforming the entrepreneurial process for starting MEMS companies and for transitioning MEMS devices into production and into the market. First, it is well-known that recent market set-backs have caused traditional VC funds to view any hardware start-ups with renewed scrutiny and skepticism. Hardware, and particularly bioMEMS, start-ups typically require large amounts of capital (\$50-\$100M) and many years (7-10), before getting close to a reasonable exit. This large investment in money and time is on top of the already inherently risky prospects for such a start-up being commercially successful. Secondly, MEMS is recognized, by investors, by foundries, and by large consumer electronics companies, as a very successful new product area because of the huge up-take of MEMS components in mobile devices during recent years. Third, key strategic issues in huge upcoming new consumer markets, such as wearables and IoT, are sensors and contextual awareness; areas which are uniquely solved by MEMS devices. And fourth, the sheer number of successful, high volume MEMS devices currently on the market, has created a huge pool of skilled MEMS developers and manufacturers which can be drawn upon for new devices and new start-up companies. All these factors dramatically influence how such companies get funding and how they operate. We will discuss all these issues as they relate specifically to new implantable and wearable MEMS start-up companies. As examples, we will also discuss а number of current technical developments/devices/companies involving implantable and wearable bioMEMS.

3:00pm MN+BI-TuA3 MEMS Sensors Make Up the Frontline of Wireless Health Solutions: Tremendous Growth Prospects, *Mehran Mehregany*, Case Western Reserve University INVITED Use of sensor-enabled wearable wireless health solutions to monitor the health condition of chronic disease patients is key to the quality of life of the patient and to reduction of cost of health care—by keeping the patient out of the hospital and emergency rooms. Monitoring for early intervention is key to avoiding long-term adverse outcomes for those at risk of developing chronic diseases. This presentation will elaborate on the important role that MEMS sensors play in enabling wearable, health monitoring solutions. Capturing data is the key to such solutions, which requires sensors of various modalities. MEMS sensors have the advantage s of miniaturization, integration and batch fabrication—driving size,

Annual heath care expenditure in the United States was  $\sim$ \$2.7 trillion in 2011 (i.e., \$8,680 per person), well above other developed countries. Health spending grew 3.9% in 2011, the same as in 2009 and 2010; spending as a share of GDP has remained stable from 2009 through 2011, at 17.9%. The US health care system is built on fee-for-service, wherein the service is reactive to illness. An aging population, longer lives and increasing cases of chronic diseases are some of the key drivers escalating health care expenditures.

performance and cost advantages.

Chronic diseases account for 75%+ of the US health care expenditures, i.e., \$2 trillion. 141 million (45% of the population) have at least one chronic disease, 72 million of which have two or more. Top 10 significant chronic diseases are: hypertension, obesity, arthritis, asthma, chronic kidney disease, depression, chronic obstructive pulmonary disease (COPD), diabetes, sleep disorder and heart failure.

4:20pm MN+BI-TuA7 GC-MS to GC-NOMS: A Step Towards Portable Analysis, Anandram Venkatasubramanian, S.K. Roy, V.T.K. Sauer, W.K. Hiebert, National Institute for Nanotechnology and University of Alberta, Canada

The Gas Chromatography (GC) – Mass Spectrometer (MS) system is the industry benchmark in research and chemical analysis. However given that MS systems are large and complicated instrumentation, chemical analyses

have a long turnaround time. In this regard, portable GCs have carved a market niche but they have poor sensitivities. Recent demonstrations with Nanooptomechanical (NOMS) resonators at atmospheric pressure have proven that these kind of sensors have the breakthrough potential to improve the sensitivity of portable GCs. In this regard we have built an experimental rig to integrate the GC system with our NOMS device. The goal of this study is two-fold. One will be to replace the GC sensor with NOMS devices, integrate with the portable GCs for better sensitivity, and ultimately match the analytical power of conventional GC-MS. The other will be to demonstrate the NOMS sensing capabilities for next generation genomic applications like personalized medicine. In this regard, we have designed and developed a free space interferometry system. The probe laser is coupled in and out of the photonic waveguide using grating couplers. Using the evanescent field of the waveguide, the shift in resonant frequency of the nanoscale resonators is recorded using lock in amplifier. Here we have tracked the response of both the ring resonators using the photodetector output and the nanomechanical resonator using the phase locked loop (PLL). GC peak sensing can be done with either or both of the mechanical and the photonic sensors. During the initial testing with analyte standards we observed the ring resonator to respond faster than the nanomechanical resonator on par with the GCs flame ionization (FID) detector. We were also able to capture the analyte peaks effectively with the sensitivity of the resonators to be about 77 zg/Hz.

4:40pm MN+BI-TuA8 Label-Free Biosensing Platform Integrating a Nanofluidic Preconcentrator with Surface Plasmon Resonance Sensors, *Wei-Hang Lee*, *P.S. Chung*, National Taiwan University, Taiwan, Republic of China, *P.K. Wei*, Academia Sinica, Taiwan, Republic of China, *W.C. Tian*, National Taiwan University, Taiwan, Republic of China

For bioMEMS applications, the integration of preconcentration and sensing has been studying to detect low-abundance analytes without labelling. In the past few years, an electrokinetic trapping (EKT)-based nanofluidic preconcentrator had been reported for providing a million-fold concentration factors that enable the validation of concentration process and the detection of trace and fluorescence-labelled analytes. However, the use of fluorescence-labelled analytes has suffered several disadvantages, e.g., additional sample preparation, high cost of labeling reagents, and difficulty in analyzing trace analytes. To monitor the concentration process without labelling, previously we have presented a real-time dual-loop electric current measurement system for label-free EKT-based nanofluidic preconcentrators. In this work, we further demonstrate a label-free biosensing platform by integrating a label-free nanofluidic preconcentrator with label-free SPR sensors.

The label-free biosensing platform was realized by a nanofluidic preconcentrator and two nanograting-structured SPR sensors. The preconcentrator is consisted of two parallel microchannels, i.e., one concentration channel and one buffer channel, cast in PDMS and connected by nanochannels. The two SPR sensors, i.e., one for control group and the other for experimental group, are fabricated on glass slide by e-beam lithography, e-gun evaporation and lift-off process. Then, we patterned a Nafion thin film on glass and at the position adjacent to the SPR sensors by using a microflow patterning method. Finally, the PDMS-based microchannels were sealed onto the by oxygen plasma bonding process.

We have demonstrated the ultra-sensitive label-free biosensing platform by detecting the amplified redshift magnitude of a specific range of a SPR spectrum. First, before preconcentration process, several reference spectra were measured. Second, after ten-minute preconcentration process for the 20 ng/ml BSA in PBS, a 5 nm-redshift spectrum was measured. Comparing the experimental spectrum with the reference spectra, the redshift magnitude of 20 ng/ml BSA in PBS after preconcentration process is equivalent to that of the 200  $\mu$ g/ml BSA in PBS. Hence, we demonstrate a preconcentration factor of ten-thousand folds and a sensing limit of at least 20 ng/ml BSA in PBS in this label-free biosensing platform.

In summary, by utilizing the electric current measurement system and the commercial optical system, low abundance analytes can be preconcentrated and sensed by the developed biosensing platform, which enables a label-free approach on preconcentrating and detecting trace molecules with high sensitivity.

#### 5:00pm MN+BI-TuA9 Microparticle Patterning Using Multimode Silicon Carbide Micromechanical Resonators, *Hao Jia*, *H. Tang*, *P.X.-L. Feng*, Case Western Reserve University

In recent years, there have been increasing interests in manipulating and patterning microparticles and biological cells on microscale planar surfaces<sup>[1],[2],[3]</sup>, among which "Chladni figures"<sup>[4]</sup>, enabled by resonant microelectromechanical systems (MEMS)<sup>[5]</sup>, offer a noninvasive, fast, and highly-controllable approach by simply programming frequency.

In this work, we report experimental demonstration of manipulating microparticles in fluidic environment using multimode silicon carbide (SiC)

MEMS resonators, forming diverse microscale Chladni patterns. Silica microspheres with various diameters (0.96, 1.70, 3.62, 7.75µm) sprinkled onto suspended surfaces of SiC doubly-clamped beams ( $60 \times 10\mu$ m,  $100 \times 10\mu$ m and  $100 \times 20\mu$ m) and square trampolines ( $50 \times 50\mu$ m and  $90 \times 90\mu$ m) are quickly manipulated into one dimensional (1D) and two dimensional (2D) geometrical patterns, such as "dots (.)", "line (/)", "cross (×)" and "circle ( $\circ$ )" by piezoelectrically exciting those resonators at their flexural resonance modes.

SiC MEMS resonators, with its unique biocompatibility<sup>[6]</sup>(indicating biological applications), are fabricated based on a SiC-on-Si platform, with device structures patterned by the focused ion beam (FIB) and suspended by an isotropic Si etching (HNA, 10% HF: 70% HNO<sub>3</sub>=1:1). Multimode resonances in liquid (up to 5MHz) are characterized using laser interferometry<sup>[6]</sup>, based on which the piezoelectric driving frequencies are switched in real-time to strongly excite the microspheres and manipulate them into a series of Chladni patterns. Such SiC resonating platform, by taking advantage of its straightforward device fabrication and engineerable multimodes, offer new means for microparticle manipulation, and other biophysical and biomedical studies.

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### Wednesday Morning, October 21, 2015

**2D Materials Focus Topic** 

Room: 212C - Session 2D+MN+NS+SP+SS+TF-WeM

#### **Mechanical and Thermal Properties of 2D Materials**

**Moderator:** Oleg Yazev, Ecole Polytechnique Fédérale de Lausanne (EPFL), Petra Reinke, University of Virginia

# 8:00am **2D+MN+NS+SP+SS+TF-WeM1** Mechanical Properties of **Polycrystalline Graphene**, *Joseph Gonzales*, University of South Florida, *R. Perriot*, Los Alamos National Laboratory, *I.I. Oleynik*, University of South Florida

Experimental investigation of mechanical properties indicates that the polycrystalline graphene grown by chemical vapor deposition is as strong as pristine. The microscopic characterization of graphene samples using Atomic Force microscopy (AFM) nano-indentation is limited in the sense that the detailed mechanical characteristics such as stress and stain distributions under the indenter, elastic moduli and breaking strength are not available directly from experiment. 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 graphene under conditions mimicking nano-indentation AFM experiments. The atomically resolved characterization of the stress and strain distributions under indenter are used to understand detailed mechanisms of graphene strength and failure. The breaking strength, the crack initiation and propagation are investigated as a function of the grain boundary structure and the grain size distribution as well as the position of the indenter - at the center of the grain, at the a single grain boundary and at the junction of three or more grain boundaries.

# 8:40am **2D+MN+NS+SP+SS+TF-WeM3** Lévy Flights Found in **Freestanding Graphene**, *Paul Thibado*, University of Arkansas, *M. Neek-Amal, F. Peeters*, University of Antwerp, Belgium

Local, long-time evolution measurements of the height fluctuations of a 2D membrane allows examination of the fundamental foundations of statistical mechanics in soft condensed matter. However, such measurements have proved elusive, thereby forcing critical theoretical assumptions in the best models. We report sub-nanometer, high-bandwidth height measurements of freestanding graphene using constant-current, point-mode scanning tunneling microscopy, as a follow-up to our previous related works [1-2]. By tracking atoms directly, the ability to measure dynamic events is increased by a factor of 1000 over the present state-of-the-art membrane imaging technology. Surprisingly, the membrane velocities follow the Cauchy-Lorentz distribution consistent with a Lévy process, rather than the expected Maxwell-Boltzmann distribution. We introduce a new theoretical approach using fractional-stochastic calculus.

Acknowledgements:

This work was supported in part by Office of Naval Research (USA) under Grant No. N00014-10-1-0181 and National Science Foundation (USA) under Grant No. DMR- 0855358.

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#### 9:00am 2D+MN+NS+SP+SS+TF-WeM4 Multilayer Graphene Strength Characterization, Joseph Rowley, N. Boyer, K. Berry, R.C. Davis, Brigham Young University, R. Creighton, J. Abbott, S. Cornaby, M. Harker, Moxtek Inc., R. Vanfleet, Brigham Young University

Although there are many examples in the literature of multilayer graphene fabrication and electrical characterization, there is a lack of data on the mechanical properties of multilayer graphene, especially many layer. Conversely there is data about the mechanical properties of single layer graphene, and limited information about bilayer and few layer, but little about many layer. Multilayer Graphene was fabricated using chemical vapor deposition on a Nickel catalyst. Different flow rates and cooling rates were investigated to produce many layer films. Due to the high strength in graphene, these films were able to be suspended over millimeter size openings and have a differential pressure applied. This allowed for the characterization of the strength of these membranes using bulge testing.

#### 9:20am 2D+MN+NS+SP+SS+TF-WeM5 Nanoelectromechanical Systems Based on 2D Materials beyond Graphene -- Effects from Geometry, Nonlinearity, and Anisotropy, Zenghui Wang, Case Western Reserve University INVITED

Investigating and manipulating the mechanical degree of freedom in twodimensional (2D) nanostructures present unique challenges and opportunities: such effort demands advanced fabrication and measurement schemes, and offers new insight into the physical properties of 2D materials. I will present our explorations and findings in mechanical the nanoscale, through studying processes at resonant nanoelectromechanical systems (NEMS) based on 2D materials beyond graphene (e.g., molybdenum disulfide, black phosphorus, etc.). I will discuss the implications of geometrical irregularities on the nanomechanical responses of 2D-material-based resonators; impacts of device and material parameters on the mechanical nonlinearity and motional noise in 2D resonant transducers; and effects of material anisotropy in nanomechanical resonators based on new types of highly anisotropic 2D materials. These findings open new pathways towards nanomechanical coupling and tuning of the physical properties in 2D nanomaterials, and offer opportunities for building novel devices with new multimode functions.

11:00am **2D+MN+NS+SP+SS+TF-WeM10** Phonon Spectroscopy of Graphene Field Effect Devices with the STM, Fabian Natterer, Y. Zhao, J. Wyrick, NIST/CNST, W.Y. Ruan, Y.-H.C. Chan, M.-Y.C. Chou, Georgia Institute of Technology, N.B. Zhitenev, J.A. Stroscio, NIST/CNST

Phonon spectroscopy of graphene by inelastic electron tunneling spectroscopy with the STM has been elusive in previous measurements [1-3]. The difficulty lies within the weak phonon signatures that are buried by other dominant spectral features that inhibit a clear distinction between phonons and miscellaneous excitations. Utilizing a back gated graphene device that permits continuous adjustment of the global charge carrier density, we employ an averaging method where individual tunneling spectra at varying charge carrier density are condensed into one representative spectrum [4]. This method improves the signal for inelastic transitions that appear at constant threshold, while it broadens and thereby suppresses dispersive spectral features. We use this method to demonstrate the mapping of the total graphene phonon density of states, in good agreement with density functional calculations. Using the knowledge about the phonons thusly obtained, we closely examine our gate resolved spectra and observe a surprising and abrupt change in the phonon intensity when the graphene charge carrier type is switched through a variation of the back gate electrode potential. This sudden variation in phonon intensity is asymmetric in carrier type, depending on the sign of the tunneling bias. We invoke a resonance mediated tunneling process that relies on the presence of tipinduced quasi-bound state resonances in graphene, resembling whispering gallery modes for electrons and holes [5]. Our tip-sample system thereby mimics a giant molecular state and shares analogies with resonant enhanced excitations of molecular vibrational or rotational modes [6-9].

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11:20am **2D+MN+NS+SP+SS+TF-WeM11 Edge-state-induced Stabilization of Dopants in Graphene**, *Yuuki Uchida*, *A. Akaishi, J. Nakamura*, The University of Electro-Communications (UEC-Tokyo) and JST CREST, Japan

Impurity doping is an efficient way to modify electronic properties of graphene. Several groups have reported the stability of dopants in graphene, especially near edges of graphene: Impurity atoms prefer to locate at the zigzag edge of graphene rather than the armchair one[1]. It has also been

reported that the electronic properties are strongly dependent upon the location of dopants, which is derrived from the non-equivalence of the two sublattice[2]. It is well-known that the edge-localized state emerges at the zigzag edge[3], which is specific for the so-called bipartite lattice. However, it has not been clarified yet how the edge-state affects the dopant stability depending on the sublattice. In this study, we investigate the role of the sublattice-dependent edge-state on the stabilization of impurities. We evaluate the dependence of the structural stability on the distance of impurity atoms from the zigzag edge using first-principles calculations within the density-functional theory. We have employed two types of graphene nanoribbons (GNRs) with the armchair- (AGNR) or the zigzag- (ZGNR) edge.

For AGNR, the formation energy of dopants does not change neither systematically nor monotonically as a function of the distance from the edge. On the other hand, for ZGNR, the formation energy is lower than that for AGNR and decreases with decreasing distance from the edge. In addition, two types of tendencies are confirmed for odd- and evennumbered sites from the zigzag edge, corresponding to the different sublattices of the bipartite lattice.

Such peculiar behavior as for of the formation energy can be explained as follows : The doped N atom donates its electron to the unoccupied-edgestate just above the Fermi level, resulting in the lowering of the one-electron energy of this state. The smaller the distance of N atoms from the zigzag edge is, the larger the electrostatic attraction between electrons of edge-localized states and positively-charged ion-shell at the N site becomes. Further, N atoms are much more stabilized at the odd-numbered site, because the edge-state has finite amplitude only at the odd-numbered sites.

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# 11:40am **2D+MN+NS+SP+SS+TF-WeM12** Exploring the Thermal Stability of Two-Dimensional Black Phosphorus, *Xiaolong Liu, J.D. Wood, K.-S. Chen, E. Cho, M.C. Hersam*, Northwestern University

Two dimensional (2D) black phosphorus (BP) has attracted significant attention due to its superlative electronic and optical properties. Unlike graphene, its intrinsic and thickness-dependent band gap makes it feasible for direct application in electronic and optoelectronic devices.<sup>1</sup> However, before 2D BP can be effectively employed in such applications, it is necessary to establish the thermal stability of 2D BP since annealing is a key element in most device fabrication processes. Towards this end, we have utilized in situ scanning/transmission electron microscopy and spectroscopy methods to characterize the thermal decomposition process of mechanically exfoliated 2D BP.<sup>2</sup> The decomposition is observed to occur at ~400 °C in the form of sublimation, compared to the 550 °C of bulk BP. This decomposition initiates via eye-shaped cracks along the [001] direction and then continues until only a thin, amorphous red phosphorous-like skeleton remains. In situ electron energy loss spectroscopy, energydispersive X-ray spectroscopy, and energy-loss near-edge structure changes provide further quantitative insight into this chemical transformation process

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12:00pm 2D+MN+NS+SP+SS+TF-WeM13 Gas Permeation Through 1 nm Thick Carbon Nanomembranes, A. Beyer, M. Ai, Bielefeld University, Germany, S. Shishatskiy, J. Wind, Helmholtz-Zentrum Geesthacht, Germany, X. Zhang, V. Chinaryan, Y. Yang, Armin Gölzhäuser, Bielefeld University, Germany

The gas permeation characteristics of 1 nm thick carbon nanomembranes (CNMs) from self-assembled monolayers are reported. The assembly of CNMs onto polydimethylsiloxane (PDMS) support membranes allows determination of gas permeation characteristics. Single layer and triple layer CNMs were investigated in respect to permeation of hydrogen, helium, carbon dioxide, oxygen, nitrogen, argon, methane and ethane. In addition, the CNM-PDMS composites were characterized by X-ray photoelectron spectroscopy, helium-ion microscopy as well as atomic force microscopy. A careful analysis about the contribution of the PDMS support membranes to the gas permeation allowed an estimate of the intrinsic CNM permeances. These values indicate a molecular sieve-like property of CNMs which is attributed to molecular-sized channels in CNMs. As an example, hydrogen and carbon dioxide gas molecules display an order of magnitude higher permeance values for single layer CNMs in comparison to oxygen and nitrogen, which possess larger kinetic diameters.

#### MEMS and NEMS Room: 211A - Session MN+AM-WeM

#### Emerging Materials & Fabrication Technologies toward Scalable & Additive Nanomanufacturing I

**Moderator:** Philip Feng, Case Western Reserve University, Roya Maboudian, University of California at Berkeley

#### 8:00am MN+AM-WeM1 The Why, the What and the How of Nanomanufacturing, *Khershed Cooper*, National Science Foundation (NSF) INVITED

In 2010, at the conclusion of the review of the NNI, PCAST recommended greater emphasis on commercialization by doubling investment of the Federal Government in nanomanufacturing R&D. Federal agencies responded in various ways. The NNI Signature Initiative: Sustainable Nanomanufacturing was announced, OSD's MURI topic call included nanomanufacturing as a high-priority theme, and NSF announced the Scalable Nanomanufacturing solicitation. The private sector also responded to the need for nanotechnology commercialization through interest groups such as the NanoBusiness Alliance and, as the Institutes for Manufacturing Innovation multiply, nanoscience and nanotechnology appear to play an increasing important role. This is the 'why' of nanomanufacturing. Nanomanufacturing is the fabrication of nano-scale building-blocks, their assembly into higher-order structures and the integration of these into larger scale systems such that both heterogeneity and complexity are achievable with manipulation and control at the nano-scale. R&D activities address the nanomanufacturing value chain and manufacturability challenges such as scale-up and cost. This is the 'what' of nanomanufacturing. This talk will these aspects of nanomanufacturing and 'how' discuss the nanomanufacturing programs (NM, SNM, NSEC, NERC, SBIR) at NSF are meeting these challenges through support of basic and applied research involving novel ideas. Proposed ideas encompass a wide range of materials, processes and applications and are usually based on strong fundamental foundations. Research outputs are nanomanufacturing fundamentals that will help overcome barriers to large-scale, high quality production of nanoenabled products for the benefit of society.

#### 8:40am MN+AM-WeM3 Large-Area Nanoimprinting and Nanoplasmonics for Energy Harvesting, LEDs & Biosensing, Stephen Y. Chou, Princeton University INVITED

This talk will present (1) the technology advances and applications of nanoimprinting, a revolutionary nanofabrication method that not only allows the fabrication of nanostructures that could not be fabricated before, but also offers a viable way to mass manufacturing of nanostructures and hence commercialization; and (2) new nanoplasmonic structures for high efficiency solar cells and LEDs and ultrasensitive biosensing that have unprecedented properties and performances.

In nanoimprint, the presentation will address the advances in (a) planar as well as roll-to-roll nanoimprinting, (b) methods of making large nanostructure nanoimprint molds without using direct-write (e.g. electronbeam lithography), (c) self-perfection technologies, and applications in electronics, optics, optoelectronics, magnetics, biotechnologies, displays, and energy harvesting.

In nanoplasmonics, the presentation will discuss the advances in design large-area, high performance nanoplasmonics used for solar cell, LEDs and biosensing, in particular, two new nanoplasmonic structures: disk-coupled dots-on-pillar antenna-array (D2PA) and plasmonic cavity with subwavelength hole-arrays (PlaCSH). Significant enhances in energy efficiency and detection sensitivity have been achieved using D2PA and PlaCSH.

#### 9:20am MN+AM-WeM5 Scalable Nanomanufacturing of Plasmonic and Metasurfaces, *Regina Ragan*, *F. Capolino*, University of California Irvine INVITED

Nanodisks and nanoparticles serve as meta-molecule building blocks to tune plasmonic and metamaterial properties when arranged in planar and threedimensional geometries. For example, the ability to control nanomaterial interfaces via colloidal synthesis allows for tuning of the plasmon resonance as well as mitigating losses affecting extinction spectra. In addition, colloidal assembly is beneficial as a high-throughput, wafer scale deposition method. We have achieved robust surface enhanced Raman scattering (SERS) sensors approaching single molecule detection limits *reproducibly* over large areas using colloidal assembly. Transmission electron microscopy data shows that by varying driving forces for assembly, diffusion versus electrophoresis, nanoparticle clusters with gaps between nanoparticles of 4 nm down to 1 nm, respectively, are obtained. Corresponding orders of magnitude decreases in detection limit allow for identification of fermentation products present in the parts per billion range in cystic fibrosis patients.

Arrays of tightly coupled metal and metal- dielectric nanoparticles also support narrow band resonances, Fano resonances, based on "dark" electric and/or magnetic resonances. We will discuss how material interfaces can be used to mitigate losses that eliminate Fano resonant features. For example, the extinction and absorption efficiencies resulting from an array of linear trimers of Au nanoshells in homogeneous environment show that efficiency is affected by changing dye concentration in nanoshells. The use of dyes as gain media induces sharpened Fano resonance features (attributed to the meta-molecule nature of the linear trimers) and increased maximum absorption efficiency at 422 THz. Using similar methods, circular nanoclusters (CNC) of metal nanoparticles can support a magnetic Fano resonance at 472 THzvia dipole moments forming a current loop underoblique TE-polarized plane wave incidence. In particular, arrayinduced resonances are narrower than single-CNC-induced ones and also provide even larger field enhancements, in particular generating a magnetic field enhancement of about 10-folds and an electric field enhancement of about 40-folds for a representative metasurface.Natural magnetism fades away at infrared and optical frequencies and artificial magnetism is cumbersome to achieve in these regimes, as conventional split ring resonators are difficult to scale down to optical wavelengths. Nanoparticles assembled from colloids are a scalable approach to engineer materials' electromagnetic properties.

#### 11:00am MN+AM-WeM10 Roll to Roll Processes at the University of Michigan: Continuous Patterning, Flexible OPVs, and Growth of Carbon Nanomaterials, Jay Guo, University of Michigan, Ann Arbor INVITED

Roll to roll fabrication is regarded as a high-throughput and cost-effective method for future manufacturing of flexible electronics, large area photonic elements and functional surfaces for a variety of applications. This talk will discuss a few examples of roll to roll processes developed at the University of Michigan. First, roll-based patterning processes will be introduced. These include roll to roll nanoimprint lithography (R2RNIL) based on mechanical deformation of polymers and capable of tens of nm resolution, photo-rollerlithography (PRL) by using flexible photomasks to pattern sub-micron and larger features continuously on a moving web. These techniques can be used in many applications, such as metal wire grid based transparent conductors. Next, fabrication of organic optoelectronics OLED and OPV by roll to roll coating processes such as blade coating, blade-slit coating will be discussed. The coating process should be selected properly by considering the thickness requirement for each active layers in the devices. Finally I will report some progress made by the UM team in the Scalable NanoManufacturing program in the roll based continuous growth of carbon nanotubes and graphene.

# 11:40am MN+AM-WeM12 Nanotube Templated Manufacturing of Hierarchically Structured High Throughput Fluid Filters, Andrew Davis, R.C. Davis, R. Vanfleet, Brigham Young University, N. Morrill, Precision Membranes

High throughput microfilters for use in water purification have been developed to address filtration challenges by dramatically decreasing flow resistance and filter size while improving filter strength, longevity, and affordability. The microfilters were fabricated out of a high strength carbon-nanotube composite material using a process for precise hierarchical patterning on both the micro and nano scales.

#### 12:00pm MN+AM-WeM13 Improved Vacuum Deposition of Small Patterned Features Using Precision Shadow Masks and a Novel Low Pressure Sputtering Source, *Rob Belan*, Kurt J. Lesker Company, *V. Heydemann*, Advantech U.S. Inc, *S. Armstrong*, Kurt J. Lesker Company, *T. Fisher*, *B. Brocato*, Advantech U.S. Inc

A novel, low pressure sputter source has been used in conjunction with a precision shadow mask to deposit crisp features on glass substrates. The low pressure sputter source (LPSS) exhibits high-rate omnidirectional deposition and can form crisp lined features that are typically 5  $\mu$ m to 50  $\mu$ m in size when combined with precision mask technology. These feature sizes are a factor of 5x smaller compared to traditional magnetron sputtering at typical sputtering pressures (~1 Pa). The LPSS operates at pressures lower than 0.1 Pa which increases the mean free path of the sputtered atoms and reduces the spread of the deposited pattern through a shadow mask that is often associated with magnetron sputtering at normal pressures.

The LPSS sputters at rates (up to 3 Å/s) with precise rate control in the range of 1 Å/s to 3 Å/s for target/substrate distances from 76 mm to 101 mm. The first generation LPSS utilized a 137 mm x 5.4 mm aluminum cathode. The second generation LPSS employed a 826 mm x 32.5 mm high purity (10 ppm) aluminum cathode demonstrating the scalability of this novel deposition source.

Patterned thin films with thicknesses between 300 Å to 2,000 Å were deposited using the first and second generation low pressure sputter sources. The impact of ambient pressure, power and source/substrate distance on the resulting thin film was investigated. The deposition runs were conducted under static conditions, with stationary source and substrate, as well as with a scanning source.

Patterned features were deposited on display-grade borosilicate glass substrates by placing custom manufactured nickel shadow masks with apertures in the 20  $\mu$ m to 50  $\mu$ m size range between the low pressure sputter source and the substrates. The shadow masks are manufactured by an electroforming process that allows precise control of the mask thickness. Typical shadow mask materials are nickel and invar (FeNi36). As well as studying the sputtering process parameters of the LPSS the quality of the deposited patterned features were investigated using a variety of mask thicknesses and aperture dimensions.

The low pressure operation of the LPSS source enables thin film deposition of precision nanostructured patterns via shadow masks and sputtering, an area traditionally kept to evaporation methods. Results and conclusions of this work will be presented for this emerging fabrication technology.

## Wednesday Afternoon, October 21, 2015

In-Situ Spectroscopy and Microscopy Focus Topic Room: 211B - Session IS+SS+NS+BI+VT+MN+AS-WeA

#### In situ Imaging of Liquids using Microfluidics

**Moderator:** Xiao-Ying Yu, Pacific Northwest National Laboratory, Stephen Nonnenmann, University of Massachusetts - Amherst

2:20pm IS+SS+NS+BI+VT+MN+AS-WeA1 In Situ Multimodal Biological Imaging using Micro- and Nanofluidic Chambers, James Evans, C. Smallwood, Pacific Northwest National Laboratory INVITED Biological organisms have evolved a number of spatially localized and highly orchestrated mechanisms for interacting with their environment. Since no single instrument is capable of probing the entire multidimensional landscape, it is not surprising that one of the grand challenges in biology remains the determination of how dynamics across these scales lead to observed phenotypes.

Therefore, there is a need for in-situ correlative multimodal and multiscale imaging to fully understand biological phenomena and how chemical or structural changes at the molecular level impact the whole organism. We have been advancing new methods for both cryogenic and in-situ correlative analysis of biological samples using electron, ion, optical and x-ray modalities. Central to this work is the development of new micro- and nanofluidic chambers that enable in-situ observations within precisely controlled liquid-flow environments. In this talk I will review the design of these new chambers, highlight current science applications and outline our future goals for adding additional functionality and expanding the versatility of the devices to other disciplines.

3:00pm IS+SS+NS+BI+VT+MN+AS-WeA3 Glyoxal Aqueous Surface Chemistry by SALVI and Liquid ToF-SIMS, *Xiao Sui*, *Y. Zhou*, *Z. Zhu*, Pacific Northwest National Laboratory, *J. Chen*, Shandong University, China, X.-Y. Yu, Pacific Northwest National Laboratory

Glyoxal, a ubiquitous water-soluble gas-phase oxidation product in the atmosphere, is an important source of oxalic acid, a precursor to aqueous secondary organic aerosol (SOA) formation. Many recent laboratory experiments and field observations suggest that more complex chemical reactions can occur in the aqueous aerosol surface; however, direct probing of aqueous surface changes is a challenging task using surface sensitive techniques. The ability to map the molecular distribution of reactants, reaction intermediates, and products at the aqueous surface are highly important to investigate surface chemistry driven by photochemical aging. In this study, photochemical reactions of glyoxal and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) were studied by a microfluidic reactor, System for Analysis at the Liquid Vacuum Interface (SALVI), coupled with Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS). Aqueous surfaces containing glyoxal and hydrogen peroxide were exposed to UV light at variable lengths of time and were immediately analyzed in the SALVI microchannel by in situ liquid ToF-SIMS. In addition, various control samples were conducted to ensure that our findings were reliable. Compared with previous results of bulk solutions using ESI-MS, our unique liquid surface molecular imaging approach provided observations of glyoxal hydrolysis (i.e., first and secondary products, dimers, trimers, and other oligomers) and oxidation products (i.e., glyoxylic acid, oxalic acid and formic acid) with submicrometer spatial resolution. We potentially provide a new perspective and solution to study aqueous surface chemistry as an important source of aqueous SOA formation of relevance to atmospheric chemistry known to the community.

3:20pm IS+SS+NS+BI+VT+MN+AS-WeA4 Investigating Shewanella Oneidensis Biofilm Matrix in a Microchannel by In Situ Liquid ToF-SIMS, Yuanzhao Ding, Nanyang Technological University, Singapore, X. Hua, Y. Zhou, J. Yu, X. Sui, J. Zhang, Z. Zhu, Pacific Northwest National Laboratory, B. Cao, Nanyang Technological University, Singapore, X.-Y. Yu, Pacific Northwest National Laboratory

Biofilms consist of a group of micro-organisms attached onto surfaces or interfaces and embedded with a self-produced extracellular polymeric substance (EPS) in natural environments. The EPS matrix, like the "house of the cells", provides bacteria cells with a more stable environment and makes them physiologically different from planktonic cells. *Shewanella oneidensis* MR-1 is a metal-reducing bacterium, forming biofilms that can reduce toxic heavy metals. This capability makes *S. oneidensis* biofilms very attractive in environmental applications. To better understand the biofilm EPS matrix composition at the interface, in situ chemical imaging

with higher spatial resolution and more molecular level chemical information is strongly needed. Traditionally, electron microscopy and fluorescence microscopy are common imaging tools in biofilm research. However, the bottlenecks in these imaging technologies face the limitations that it is difficult for them to provide chemical information of small molecules (e.g., molecule weight <200). In this study, we use an emerging technology liquid Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) to observe S. oneidensis biofilm cultured in a vacuum compatible microchannel of the System for Analysis at the Liquid Vacuum Interface (SALVI) device. Chemical spatial distributions of small organic molecules that are considered to be the main building components of EPS in live biofilms are obtained. Principal component analysis is used to determine differences among biofilms sampled along the microchannel. This new approach overcomes previous limitations in live biofilm analysis and provides more chemical information of the EPS relevant to biofilm formation. Better understanding of the biofilm matrix will potentially fill in the knowledge gap in biofilm surface attachment and detachment processes and improve the engineering and design of S. oneidensis biofilms with high efficiencies in heavy metal reduction.

IS+SS+NS+BI+VT+MN+AS-WeA7 Ultrafast Proton and 4:20pm Electron Dynamics in Core-Level Ionized Aqueous Solution, Bernd Helmholtz-Zentrum Winter. Berlin für Materialien und Energie/Elektronenspeicherring BESSY II, Germany INVITED Photo- and Auger electron spectroscopy from liquid water reveals a novel electronic de-excitation process of core-level ionized water in which a pair of two cations forms, either  $H_2O^+ \cdot H_2O^+$  or  $OH^+ \cdot H_3O^+$ . These reactive species are the delocalized analogue to  $H_2O^{2+}$ , formed in a localized on-site Auger decay, and are expected to play a considerable role in water radiation chemistry. Both cationic pairs form upon autoionization of the initial ionized water molecule, and we are particularly interested in the situation where autoionization occurs from a structure that evolves from proton transfer, from the ionized water molecule to a neighbor molecule, within a few femtoseconds. The actual autoionization is either through intermolecular Coulombic decay (ICD) or Auger decay. Experimental identification of the proton dynamics is through isotope effects. A question that arises is whether such so-called proton-transfer mediated charge separation (PTM-CS) processes occur in other and similarly hydrogenbonded solute molecules as well. This is indeed the case, and is illustrated here for ammonia and glycine in water, as well as for hydrogen peroxide in water, where characteristic differences are detected in the Auger-electron spectra from the light versus heavy species, i.e., NH<sub>3</sub> in H<sub>2</sub>O versus ND<sub>3</sub> in D<sub>2</sub>O, glycine(H) in H<sub>2</sub>O versus glycine(D) in D<sub>2</sub>O, and H<sub>2</sub>O<sub>2</sub> in H<sub>2</sub>O versus D<sub>2</sub>O<sub>2</sub> in D<sub>2</sub>O. The important spectral feature here is the high-kinetic energy tail of the Auger spectrum, which has no gas-phase analogue, and hence reflects the participation of solvent water in the relaxation process. The probability of the proton dynamics, judged from the intensities of the electron signal and inferred from methods of quantum chemistry and molecular dynamics, is found to depend on hydrogen-bond strength and hence on the specific hydration configuration. Favorable configurations for hydrogen peroxide(aq) occur due to the molecule's flexible structure. In ammonia(aq) the PTM processes are found to be less probable than for water(aq), which is attributed to the planarization of the ammonia molecule upon core-level ionization. The effect is smaller for the neutral -NH<sub>2</sub>(aq) group of glycine at basic pH, where intramolecular dynamics is less likely. Nature and chemical reactivity of the initial transient species and their role for radiation chemistry and for local reactions relevant for biological molecules in an aqueous environment are discussed for the different molecular hydrogen-bonded systems.

5:00pm IS+SS+NS+BI+VT+MN+AS-WeA9 Water Dissociation in Metal Organic Frameworks with Coordinatively Unsaturated Metal Ions: MOF-74, *Kui Tan*, The University of Texas at Dallas, *S. Zuluaga*, Wake Forest University, *E. Fuentesf*, The University of Texas at Dallas, *H. Wang*, Rutgers University, *P. Canepa*, Wake Forest University, *J. Li*, Rutgers University, *T. Thonhauser*, Wake Forest University, *Y.J. Chabal*, The University of Texas at Dallas

Water dissociation represents one of the most important reactions in catalysis, essential to the surface and nano sciences. However, the dissociation mechanism on most oxide surfaces is not well understood due to the experimental challenges of preparing surface structures and characterizing reaction pathways. To remedy this problem, we propose the metal organic framework MOF-74 as an ideal model system to study water reactions. Its crystalline structure is well characterized; the metal oxide node mimics surfaces with exposed cations; and it degrades in water. Combining *in situ* IR spectroscopy and first-principles calculations, we explored the MOF-74/water interaction as a function of vapor pressure and

temperature. Here, we show that, while adsorption is reversible below the water condensation pressure (~19.7 Torr) at room temperature, a reaction takes place at ~150 °C even at low water vapor pressures. This important finding is unambiguously demonstrated by a clear spectroscopic signature for the direct reaction using D<sub>2</sub>O, which is not present using H<sub>2</sub>O due to strong phonon coupling. Specifically, a sharp absorption band appears at 970 cm<sup>-1</sup> when D<sub>2</sub>O is introduced at above 150 °C, which we attribute to an O-D bending vibration on the phenolate linker. Although H<sub>2</sub>O undergoes a similar dissociation reaction, the corresponding O-H mode is too strongly coupled to MOF vibrations to detect. In contrast, the O-D mode falls in the phonon gap of the MOF and remains localized. First-principles calculations not only positively identify the O-D mode at 970 cm<sup>-1</sup> but derive a pathway and kinetic barrier for the reaction and the final configuration: the D (H) atom is transferred to the oxygen of the linker phenolate group, producing the notable O-D absorption band at 970 cm<sup>-1</sup>, while the OD (or OH) binds to the open metal sites. Experimental data and theoretical modeling further shows that the reaction is facilitated by a cooperative effect of several H<sub>2</sub>O molecules. This finding explains water dissociation in this case and provides insight into the long-lasting question of MOF-74 degradation. Overall, it adds to the understanding of molecular water interaction with cation-exposed surfaces to enable development of more efficient catalysts for water dissociation.

Ref: K. Tan, S. Zuluaga, Q. Gong, P. Canepa, H. Wang, J. Li, Y. J. Chabal and T. Thonhauser, *Chem. Mater.*, 2014, **26**, 6886-6895.

5:20pm IS+SS+NS+BI+VT+MN+AS-WeA10 Competitive Co-Adsorption of CO<sub>2</sub> with H<sub>2</sub>O, NH<sub>3</sub>, SO<sub>2</sub>, NO, NO<sub>2</sub>, N<sub>2</sub>, O<sub>2</sub>, and CH<sub>4</sub> in M-MOF-74 (M= Mg, Co, Ni): The Role of Hydrogen Bonding. K. Tan, The University of Texas at Dallas, *Sebastian Zuluaga*, Wake Forest University, H. Wang, Rutgers University, Y. Gao, The University of Texas at Dallas, J. Li, Rutgers University, T. Thonhauser, Wake Forest University, Y.J. Chabal, The University of Texas at Dallas

The importance of co-adsorption for applications of porous materials in gas separation has motivated fundamental studies, which have initially focused on the comparison of the binding energies of different gas molecules in the pores (i.e. energetics) and their overall transport. By examining the competitive co-adsorption of several small molecules in M-MOF-74 (M= Mg, Co, Ni) with in-situ infrared spectroscopy and ab initio simulations, we find that the binding energy at the most favorable (metal) site is not a sufficient indicator for prediction of molecular adsorption and stability in MOFs. Instead, the occupation of the open metal sites is governed by kinetics, whereby the interaction of the guest molecules with the MOF organic linkers controls the reaction barrier for molecular exchange. Specifically, the displacement of CO2 adsorbed at the metal center by other molecules such as H2O, NH3, SO2, NO, NO2, N2, O2, and CH4 is mainly observed for H<sub>2</sub>O and NH<sub>3</sub>, even though SO<sub>2</sub>, NO, and NO<sub>2</sub>, have higher binding energies (~70-90 kJ/mol) to metal sites than that of CO<sub>2</sub> (38 to 48 kJ/mol) and slightly higher than water (~60-80 kJ/mol). DFT simulations evaluate the barriers for  $H_2OaCO_2$  and  $SO_2aCO_2$  exchange to be ~ 13 and 20 kJ/mol, respectively, explaining the slow exchange of CO<sub>2</sub> by SO<sub>2</sub>, compared to water. Furthermore, the calculations reveal that the kinetic barrier for this exchange is determined by the specifics of the interaction of the second guest molecule (e.g., H<sub>2</sub>O or SO<sub>2</sub>) with the MOF ligands. Hydrogen bonding of H<sub>2</sub>O molecules with the nearby oxygen of the organic linker is found to facilitate the positioning of the H2O oxygen atom towards the metal center, thus reducing the exchange barrier. In contrast, SO<sub>2</sub> molecules interact with the distant benzene site, away from the metal center, hindering the exchange process. Similar considerations apply to the other molecules, accounting for much easier CO<sub>2</sub> exchange for NH<sub>3</sub> than for NO, NO<sub>2</sub>, CH<sub>4</sub>, O<sub>2</sub>, and N<sub>2</sub> molecules. In this work, critical parameters such as kinetic barrier and exchange pathway are first unveiled and provide insight into the mechanism of competitive co-adsorption, underscoring the need of combined studies, using spectroscopic methods and ab initio simulations to uncover the atomistic interactions of small molecules in MOFs that directly influence co-adsorption.

Ref: K. Tan, S. Zuluaga, Q. Gong, Y. Gao, N. Nijem, J. Li, T. Thonhauser and Y. J. Chabal, *Chem. Mater.*, 2015, **27**, 2203-2217.

#### 6:00pm IS+SS+NS+BI+VT+MN+AS-WeA12 In Situ STM Observation of Pd(110) Under the Hydrogen Pressure Between 10<sup>-6</sup> Pa and 10<sup>-3</sup> Pa, Jun Yoshinobu, H. Kikuchi, T. Koitaya, K. Mukai, S. Yoshiomoto, University of Tokyo, Japan

Hydrogen adsorption and absorption on/in Pd and Pd alloys are vital processes for the hydrogen storage and hydrogen permeation materials. We investigated the Pd(110) surface under the hydrogen pressures between  $10^{-6}$  Pa and  $10^{-3}$  Pa at room temperature using in-situ atom-resolved scanning tunneling microscopy (STM). We observed missing-atom, missing-row and added-row structures and the number of atoms in these structures were quantitatively analyzed as a function of exposure time. Note that adatoms were not detected probably because they were mobile in the present

experimental conditions. At  $10^{-6}$  Pa, the numbers of missing-row and addedrow atoms increased up to ~20 L (langmuir) and after that they were gradually reaching the saturation (steady-state). On the other hand, the number of missing-atoms decreased gradually from the initial stage. With increasing the hydrogen pressures the number of missing-row atoms and added-row atoms increased, and the whole surface was covered with these reconstructed structures after large exposures (>1000 L). It has been known that not only hydrogen adsorption but also hydrogen absorption occur in such conditions. Thus, the missing-row and added-row reconstructed structures are inevitable for hydrogen absorption on Pd(110).

#### MEMS and NEMS

Room: 211A - Session MN+AM-WeA

#### **Emerging Materials & Fabrication Technologies toward Scalable & Additive Nanomanufacturing II**

**Moderator:** Susan Burkett, The University of Alabama, Philip Feng, Case Western Reserve University

#### 2:20pm MN+AM-WeA1 Scalable Laser-Assisted Three Dimensional Printing of Nanomaterials, Costas Grigoropoulos, University of California at Berkeley INVITED

Nanomaterials and nanotechnology offer unique opportunities for fabricating devices of novel architecture and enhanced performance and can overcome system integration issues challenging current nanomanufacturing methods that are suited to planar geometries and are confined to top-down architectures. The central motivation of our work is to develop a new manufacturing method that offers scalability and flexibility enabling nanoscale device fabrication and integration in truly three-dimensional architectures over large areas and with arbitrary densities.

The core research strategy takes advantage of ultrafast laser beam processing for generating the scaffold multi-scale structures with 100 nm feature resolution. Two-photon polymerization is used to fabricate structures of tunable properties that are sensitive to pressure, light, heat and electrical stimulation. This technique, together with ultrafast laser micro/nanomachining will be adapted to multiple beam configurations in order to increase the processing throughput. Once the template is constructed, the directed self-assembly of block copolymers will be used to produce three-dimensional materials with tailored functionality where pattern amplification will be used to push the length scale to finer dimensions.

#### 3:00pm MN+AM-WeA3 Material Requirements and Challenges for NEM Logic Relays, *Tsu-Jae Liu*, University of California at Berkeley INVITED

The proliferation of mobile electronic devices and the emergence of applications such as wireless sensor networks and the Internet of Things have brought energy efficiency to the fore of challenges for future information-processing devices. The energy efficiency of a digital logic integrated circuit is fundamentally limited by non-zero transistor off-state leakage current. Mechanical switches have zero leakage current and potentially can overcome this fundamental limit. Contact adhesive force sets a lower limit for the switching energy of a mechanical switch, however, and also directly impacts its performance.

Stable operation with high endurance is a key requirement of switches for digital logic applications, and generally is a challenge for mechanical devices. The reliability of a miniature relay is limited not by structural fatigue or dielectric charging, but by contact oxidation or stiction.

This invited talk will begin with a review of recent progress toward the development of a reliable nano-electro-mechanical (NEM) relay technology for digital logic applications. It will then discuss the influence of contacting electrode material properties on relay performance and reliability. Opportunities for ultra-low-power computing with relays will be described.

#### 4:20pm MN+AM-WeA7 Microplasma-based Direct-write Patterning Processes for Additive Microfabrication, Christian Zorman, Case Western Reserve University INVITED

Metals comprise the most versatile and widely used class of materials in micro- and nanosystems, serving as electrical contacts, interconnects, electrodes, and even mechanical components. The most commonly used method to fabricate metallic structures involves physical vapor deposition combined with photolithography. This subtractive approach is effective in producing device structures with high pattern fidelity; however, such processes are limited by modest throughput, use of aggressive chemicals and high material wastage. Reliance on vacuum-based deposition processes

limits process scalability and can hinder adoption in cost-sensitive applications. The emergence of flexible electronics has stimulated the development of additive approaches such as ink-jet printing for depositing patterned metal structures. Printing is attractive for such applications because it is performed under ambient conditions and can be integrated into large-scale roll-to-roll systems. However, the inks can be expensive and the variety of materials that are available as inks is limited. In addition, the organic capping agents that are used to stabilize nanoparticle-based inks are difficult to remove, which can compromise conductivity and mechanical integrity. Removal of the organics requires post-deposition processes that structures to the substrates can also be a significant issue, especially in flexible applications.

This paper describes two novel microplasma-based processes under development at Case Western Reserve University to fabricate patterned metallic structures with micro- to nanoscale dimensions on rigid and flexible substrates. Our principal process utilizes electrons extracted from an atmospheric pressure microplasma to electrochemically reduce metal ions within a polymer substrate, selectively forming continuous metallic structures within that polymer. Recently, we have developed a microsputtering process that uses ions generated by an atmosphericpressure microplasma. This process capitalizes on a physical vapor that is generated within a small capillary by Ar ion bombardment of small diameter metal wires. Forced Ar flow aids in the ejection of the resulting physical vapor through the orifice, which is positioned in close proximity to the substrate. Both processes are performed under ambient conditions thereby offering the same advantages as ink-jet printing, including potential scale-up to roll-to-roll processing. This presentation will detail the two processes and summarize most recent results in creating and characterizing micropatterned metal structures on a variety of substrates.

5:00pm MN+AM-WeA9 Ni-induced Graphitization for Enhanced Long-term Stability of Ohmic Contact to Polycrystalline 3C-SiC, S. Chen, L.E. Luna, University of California at Berkeley, Z. You, Tsinghua University, C. Carraro, Roya Maboudian, University of California at Berkeley

Micro- and nano-electromechanical systems (M/NEMS) technology enables a diverse range of physical and chemical sensing under conditions close to ambient. However, there is a growing interest in sensors that can operate under harsh environments, including high temperature, high pressure, extreme radiation and corrosive. Sensing within these environments necessitates a robust semiconductor platform, different from those employed in traditional Si-based M/NEMS. A robust material, such as silicon carbide (SiC) provides compelling advantages not achievable with Si-based devices. SiC is a wide bandgap semiconductor with excellent mechanical, chemical and electrical stability, and thus is well suited for designing devices capable of operation in many harsh environments. Yet, harsh-environment stable metallization remains one of the key challenges with SiC technology. Here, we present a novel metallization scheme, utilizing solid-state graphitization of SiC, to improve the long-term reliability of Pt/Ti contacts to polycrystalline n-type SiC at high temperature. The metallization scheme includes an alumina protection layer and exhibits low stable contact resistivity even after long-term (500 hr) testing in air at 450 °c. This study provides a feasible fabrication method and discusses the role of induced graphitic layer on contact stability.

#### 5:20pm MN+AM-WeA10 Fabrication of High Aspect Ratio Millimeter-Tall Free Standing Post Arrays using Carbon-Nanotube-Templated Microfabrication with a Sacrificial Hedge, *Guohai Chen, R. Vanfleet, R.C. Davis*, Brigham Young University

Carbon-nanotube-templated microfabrication (CNT-M) has shown precise high aspect ratio features in interconnected geometries.<sup>1</sup> However, the feature of isolated posts remains challenging.<sup>2</sup> Here we developed a process which involves fabrication of CNT posts connected by supporting hedges using CNT-M followed by oxygen plasma etching to remove the sacrificial hedges. We have explored the fabrication of posts with diameters from 10-40 um and heights up to 1.3 mm using sacrificial hedges of 1-5 um in width. With the CNT template, isolated free standing posts from a variety of materials can be made. For example, silicon or silicon nitride posts can be fabricated by infiltrated with silicon or silicon nitride. The creation of hybrid carbon/metal (copper, nickel) posts can also be realized through pulse electroplating.

1. J. Song, et al. Carbon-Nanotube-Templated Microfabrication of Porous Silicon-Carbon Materials with Application to Chemical Separations. Adv. Funct. Mater., 2011, 21, 1132.

2. K. Moulton, et al. Effect of iron catalyst thickness on vertically aligned carbon nanotube forest straightness for CNT-MEMS. J. Micromech. Microeng., 2012, 22, 055004.

#### Thin Film Room: 111 - Session TF+AS+EM+EN+MN-WeA

# CV Infiltration Methods and Energetic and Thermal Properties of Thin Films

**Moderator:** Richard Vanfleet, Brigham Young University, David Allred, Brigham Young University

#### 2:20pm TF+AS+EM+EN+MN-WeA1 The Many Avatars of PVD, Murali Narasimhan, Applied Materials, Inc. INVITED

Physical Vapor Deposition has been used for many years for depositing thin film coatings for diverse uses ranging from jewelry to industrial cutting tools. PVD has found usage in the manufacture of advanced semiconductor manufacturing for depositing various metals and some specialty dielectrics as well. The majority of high purity metal deposition for semiconductor use has been done using PVD although the use of CVD and ALD has increased over the years because of requirements of conformality and gap fill where conventional planar PVD has not been adequate. However, breakthroughs in PVD technology have been successful in extending the use of PVD to advanced semiconductor manufacturing nodes by changing the geometry of PVD sources and reactors and the nature of the plasma involved. Collimated and long-throw sources developed by the semiconductor equipment industry in the early '90s enabled the deposition of high-purity Ti to lower contact resistance for transistors. Reactive sputtering of TiN enabled a robust barrier for CVD W plugs used at the 0.5um node. Further, use of electromagnetic fields to ionize and then guide the plasma and sputtered ionized atoms has been successful in improving the conformality of PVD Ti films. Ionized Metal Plasma (IMP), Hollow-Cathode Magnetron (HCM) and Self-Ionized Plasma (SIP) were innovations in ionized PVD reactor design that led to widespread adoption of PVD TaN and PVD Cu for Cu interconnect barrier and seed layer production from the 90nm node to the present. The application of thermal energy on the substrate during PVD Al and Cu has been useful in improving the flow of deposited material and subsequent gap-fill of sub-micron features. The use of Radio Frequency (RF) energy to power the target has allowed for more efficient ionization at lower power levels. The application of a capacitive tuner to modulate the ion bombardment on the wafer and tailor the film properties of TiN for hard mask applications has enabled the realization of etched features at the 22nm node. Pulsed DC magnetrons enable sputtering of dielectric materials, thus opening up the controlled deposition of thin films of insulating films for various applications such as improving the brightness of high-efficiency LEDs. Multi-cathode off-axis PVD magnetrons have enabled the deposition of multi-layers of ultra-thin films for magnetic devices such as advanced inplane and out-of plane MRAM and the manufacture of EUV mask blanks for sub 10nm manufacturing. This talk will present the above listed progression of PVD technology over the years and its use for many applications in semiconductor manufacturing.

3:00pm TF+AS+EM+EN+MN-WeA3 Reactive Foil Ignition by Laser Irradiation: Experimental and Modeling Results, Ryan Murphy, C.D. Yarrington, Sandia National Laboratories, R.V. Reeves, Lawrence Livermore National Laboratory, D.P. Adams, Sandia National Laboratories It has been shown that forced mixing of reactive layers (foils) leads to an exothermic release of energy after initiation by pulsed laser irradiation. In order to understand the ignition of foils initiated by laser irradiation, we study the interaction of laser pulses with Al/Pt multilayer reactive foils prepared by sputter deposition. It will be shown that the single-pulse ignition threshold and dynamics are dependent on the length of the laser pulse as the pulse length is varied from 150 fs to 100 ms. The dependence of the ignition threshold on pulse length is a combination of laser-material interactions such as the size of the heat affected zone and the onset of ablation for ultrafast irradiation. Simulations of single-pulse laser heating were performed with Aria, the thermal package of the SIERRA finite element computational framework. Three-dimensional geometries were subjected to laser flux boundary conditions equal to those measured from the experimental conditions. Modeling and experimental results are correlated to show the effects of the heat affected zone size and shape on ignition thresholds and onset times.

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

#### 3:20pm TF+AS+EM+EN+MN-WeA4 The Effects of a Heat Sink on Self-Sustained Propagating Reactions in Sputter-Deposited Bimetallic Multilayers, David Adams, R.V. Reeves, M. Hobbs, Sandia National Laboratories

Reactive multilayers grown by sputter deposition have recently attracted interest for applications including material joining (soldering, brazing) and energy sources. For these applications, a metal-metal multilayer is typically designed to have many discrete reactant layers and a composition that corresponds to the peak enthalpy for a given material system. A thickness of reactive multilayers as small as 1.6 microns has recently been demonstrated for microelectronics joining (Braeuer et al. ECS Transactions, 2012). However, little is known about the minimal multilayer thickness required for ensuring a self-sustained, high temperature synthesis (SHS) reaction.

With this presentation, we describe the behavior of thin reactive Al/Pt multilayers tested as freestanding foils and as adhered films. For multilayers having a total thickness of 1.6 microns, self-sustained, high temperature reactions readily occur when the multilayer is tested as a freestanding foil. When coupled to a semi-infinite substrate, the likelihood of reaction is reduced depending on the multilayer design.

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#### 5:00pm TF+AS+EM+EN+MN-WeA9 Beyond Deep Silicon Etching – Generating High Aspect Ratio Microstructures by Infiltration of Carbon Nanotube Frameworks, *Robert Davis*, Brigham Young University INVITED

In addition to being the anchor material for microelectronics, silicon is widely used as the basis of high aspect ratio microfabrication for MEMS with applications ranging from inertial sensors to neural probe arrays. Carbon nanotube templated microfabrication (CNT-M), extends the palate of materials and structures for high aspect ratio microfabrication beyond those achievable with vertically etched bulk silicon. In CNT-M, 3-D forests of patterned vertically-aligned carbon nanotubes are grown as a high aspect ratio framework and then the "forests" are infiltrated with a secondary material by chemical vapor deposition. Precision structures (including nanoporous structures) with very high aspect ratios (greater than 400:1) can be generated with CNT-M. The infiltration materials range from ceramics to metals and include silicon dioxide, silicon nitride, carbon, nickel, and yes silicon. We are using CNT-M to fabricate functional structures for applications including mechanical actuation, chemical separations and detection, and electrochemical energy storage.

5:40pm TF+AS+EM+EN+MN-WeA11 The Influence of Thin Binder Films on Reaction Behavior in Reactive Powder Complexes, Robert Reeves, K.T. Sullivan, A.E. Gash, Lawrence Livermore National Laboratory With the recently renewed interest in additive manufacturing (AM), there has been a recent upswell in the number of AM processes available. One such process that could be useful for reactive materials utilizes a curable liquid binder to adhere loose powders into coherent solid forms. In this process, tap-density powders are nearly saturated with binder, so the resulting film of binder present on each particle can represent a significant contaminant to the reaction system. In this work, the effect of the binder on reaction behavior in the Ni-Al system is explored. First, the distribution of binder and its elemental constituents are studied by electron microscopy and energy dispersive spectroscopy for powders with varying levels of binder saturation. Then, the effect of binder on the reaction kinetics and overall behavior is investigated. The change in overall heat release and apparent activation energy are quantified through differential scanning calorimetry, and the bulk reaction propagation rate is measured by high speed photography as a function of the weight fraction of binder in the compact. Finally, the reaction products are identified through x-ray diffraction. In all tests, comparisons are made to the neat Ni-Al system.

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

6:00pm **TF+AS+EM+EN+MN-WeA12** Carbon Nanotube Sheets from Horizontally Aligned Carbon Nanotubes, *Nathan Boyer*, *D.B. Syme, J.T. Rowley*, Brigham Young University, *M. Harker, R. Creighton, S. Cornaby*, Moxtek Inc., *R. Vanfleet, B.D. Iverson*, Brigham Young University, *L. Pei*, Johns Hopkins University, *R.C. Davis*, Brigham Young University

Carbon sheets comprised of horizontally aligned carbon nanotubes (CNT) were prepared by rolling vertically aligned CNTs into a thin-film. A subsequent infiltration step to coat the rolled CNTs with amorphous carbon or polymer has also been performed to improve adhesion of neighboring CNTs. Amorphous carbon infiltration was achieved using chemical vapor deposition and polymer infiltration was performed by dipping the sheet into

a solvent-mediated, polymer solution. The typical failure mode of the CNT thin-films is to tear parallel to the alignment of the CNTs. Infiltration of the aligned CNT film with additional materials strengthens the film against tearing and increases burst pressure. Non-infiltrated CNT thin-films have sustained a differential pressure of 1.4 atm over a circular area of 7 mm2 on a bulge test apparatus. Both carbon and polymer infiltrated sheets could be used in many applications including micromechanical sensing and actuation.

## Thursday Morning, October 22, 2015

#### MEMS and NEMS Room: 210B - Session MN-ThM

#### Atomic Layer Nanostructures and 2D NEMS

**Moderator:** Tse Nga (Tina) Ng, PARC (Palo Alto Research Center), a Xerox Company

8:00am MN-ThM1 Piezoelectric and Phase Change Properties of Two-Dimensional Materials, *Evan Reed*, Stanford University INVITED Some of the most dramatic accomplishments with 2D materials have been enabled by properties that emerge only at the single or few-layer limit and are not found in bulk forms. Using and developing a variety of atomistic modeling methods, we have predicted that many of the commonly studied single-layer and few-layer transition metal dichalcogenide (TMD) materials (e.g. MoS<sub>2</sub>) exhibit substantive electromechanical coupling in the form of piezoelectric and flexoelectric-like effects, unlike their bulk parent crystals.<sup>1</sup> I will describe the first recent observations of some of these effects in the laboratory by several research groups.

Single-layers of two-dimensional Mo- and W-dichalcogenide compounds differ from graphene in an important respect: they can potentially exist in more than one crystal structure. Some of these monolayers exhibit hints of a poorly understood structural metal-to-semiconductor transition with the possibility of long metastable lifetimes. If controllable, such a transition could bring an exciting new application space to monolayer materials. We have discovered that mechanical deformations provide a route to switching thermodynamic stability between a semiconducting and a metallic crystal structure in some of these monolayer materials.<sup>2</sup> Our DFT-based calculations reveal that single-layer MoTe<sub>2</sub> exhibits a phase boundary at a few percent tensile strain. The potential application space for this work ranges from information and energy storage to electronic and optical electronic devices.

<sup>1</sup>Karel-Alexander N. Duerloo, Mitchell T. Ong, and Evan J. Reed, Journal of Physical Chemistry Letters **3** (19), 2871 (2012); Karel-Alexander N. Duerloo and Evan J. Reed, Nano Letters (4), 1681 (2013).

<sup>2</sup>Karel-Alexander Duerloo, Y. Li, and E. J. Reed, Nature Communications **5**, 4214 (2014).

#### 8:40am MN-ThM3 Novel Graphene Devices Based on Laser-Scribing Technology, H. Tian, Qian-Yi Xie, N.-Q. Deng, L.-Q. Tao, X.-F. Wang, W.-T. Mi, Y.-X. Li, H.-M. Zhao, Y.-T. Li, Y. Shu, Y. Yang, T.-L. Ren, Tsinghua University, China INVITED

Wafer-scale graphene devices could be fabricated by one-step laser-scribing technology. Six kinds of novel graphene devices have been developed, including in-plane transistor, resistive memory, photo detector, earphone, strain sensor and pressure sensor. The in-plane graphene transistor has a large on/off ratio up to 5.34 with simpl e structure. The graphene resistive memory has a Fin-like structure with forming-free, stable switching, reasonable reliability and potential for 2-bit storage. The 1D and 2D arrays of graphene photo detectors were achieved with photo responsivity as high as 0.32 A/W. The graphene earphone realizes wide-band sound generation from 100 Hz to 50 kHz, which can be used for both human and animals. The strain sensor based on graphene micro ribbon has the gauge factor up to 9.49. The sensitivity of the graphene pressure sensor is as high as 0.96 kPa-1 in a wide pressure range (0~50 kPa). These results demonstrated that the laser-scribed technology could be used as a platform to develop novel graphene devices.

Keywords: Graphene Devices; Laser-Scribing; Wafer-scale; Transistors; Memory; Sensors and Actuators

Acknowledgments: This work was supported by National Natural Science Foundation (61434001), 973 Program (2015CB352100), National Key Project of Science and Technology (2011ZX02403-002) and Special Research Fund of Agricultural Science and Technology for Public Interest (201303107) of China.

#### 9:20am MN-ThM5 Ultrathin Hexagonal Boron Nitride (h-BN) Nanomechanical Resonators, *Xu-Qian Zheng, J. Lee, P.X.-L. Feng*, Case Western Reserve University

Among recently emerged two-dimensional (2D) materials, hexagonal boron nitride (h-BN) possesses some unique properties, including a wide bandgap (5.9eV) [1] and electrical insulation, excellent thermal stability, and superb inertness [2]. It also has very high elastic modulus ( $E_{\rm Y} \sim 810$ GPa) which is on the same order with that of graphene [3]. Similar to graphene, h-BN also has very high fracture strain limits [4] that are far beyond achievable values

in conventional 3D crystals. In particular, h-BN offers piezoelectricity in an ultrathin 2D platform [5]. Therefore h-BN is an attractive structural material for 2D nanoelectromechanical systems (NEMS), especially as resonant transducers in harsh environment applications.

In this work, we describe the construction of h-BN drumhead structures and the first demonstration of vibrating h-BN 2D NEMS resonators. We investigate the elastic properties and resonant characteristics of such devices, by measuring flexural-mode resonances using ultrasensitive laser interferometry. We first fabricate circular drumhead h-BN resonators with thickness ranging from 9nm to 292nm and diameter of ~10µm using a completely dry transfer technique [6]. Then, by conducting both undriven and driven measurements on h-BN resonators, we observe and study the multimode resonances up to 7 modes, in high frequency (HF) and very high frequency (VHF) range. We further conduct spatially resolved measurements to attain the mode shapes of the multimode resonances. We then investigate the multimode frequency scaling of h-BN nanomechanical resonators with different thickness and analyze the experimental results to extract the elastic properties, structural and geometric effects. Finally, in comparison with the previously studied graphene and MoS<sub>2</sub> 2D NEMS resonators, we identify potential applications of these new h-BN devices in the 2D NEMS family.

- [1] Y. Kubota, et al., Science **317**, 932-934 (2007).
- [2] A. Pakdel, et al., Chem. Soc. Rev. 43, 934-959 (2014).
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- [4] L. Song, et al., Nano Lett. 10, 3209-3215 (2010).
- [5] K.-A. Duerloo, et al., J. Phys. Chem. Lett. 3, 2871-2876 (2012).
- [6] R. Yang, et al., J. Vac. Sci. & Tech. B 32, 061203 (2014).

9:40am MN-ThM6 Microfabricated MoS<sub>2</sub> and MoS<sub>2</sub>/Graphene Aerogel based Sensor for NO<sub>2</sub> Gas Detection, *Hu Long*, University of California at Berkeley, *M. Worsley*, Lawrence Livermore National Laboratory, *A. Harley-Trochimczyk*, *C. Carraro*, *R. Maboudian*, University of California at Berkeley

Two dimensional layered materials, such as graphene and transition metal dichalcogenides (TMD) have great potential for gas sensing applications. Ultrasensitive chemical detection using single-layer graphene and singleand few-layer MoS<sub>2</sub> has been reported. However, many of these sensors suffer from slow response and recovery. The difficulty of the fabrication process also hinders the broader application of these materials. In this paper, we report on MoS<sub>2</sub> and MoS<sub>2</sub>/graphene aerogels for chemical sensing applications. The aerogels are integrated onto a microfabricated transducer and a ppb level detection limit for NO<sub>2</sub> at room temperature is achieved. The performance of the sensors is comparable to the results reported for single or few-layer MoS<sub>2</sub> devices but follows much more scalable synthesis and device fabrication processes.

#### Nanometer-scale Science and Technology Room: 212B - Session NS+MN-ThM

# Nanopatterning and Nanolithography/Nanoscale Mechanics

**Moderator:** Stephane Evoy, University of Alberta, Jeremy Robinson, Naval Research Laboratory

#### 8:00am NS+MN-ThM1 Electron Stimulated Desorption and Raman Investigations of HafSOx Inorganic Resists, *Ryan Frederick*, *G. Herman*, Oregon State University

Hafnium oxide hydroxide sulfate (HafSOx) materials are under investigation as inorganic photoresists due to their high patterning fidelity, which is important for next-generation nano-lithography. In order to develop materials with better patterning sensitivity and higher resolution it is critical to understand the underlying mechanisms that result in the soluble/insoluble transition after exposure to radiation. Prior studies have shown that the incorporation of hydrogen peroxide into HafSOx is necessary to provide radiation sensitivity. In this presentation we will demonstrate that electron stimulated desorption (ESD) is a very useful technique for the characterization of radiation-induced mechanisms in HafSOx, and potentially other inorganic photoresists. These ESD studies are being performed with low energy electrons,  $E_{kin} = 50 - 500eV$ , which includes the range of photoemitted and secondary electron energies expected during extreme ultraviolet (EUV) lithography. In the case of HafSOx films we found significant O<sub>2</sub> desorption during ESD for films containing hydrogen peroxide, and found no O2 desorption during ESD for films not containing hydrogen peroxide. These studies suggest that O2 is the primary ESD species, and this desorption product can be correlated with the radiation sensitive hydrogen peroxide that is incorporated in the HafSOx films. We have used the Menzel-Gomer-Redhead desorption model to determine the O2 ESD cross-sections over a range of electron energies and doses, and determined that the cross-sections range from  $2 \times 10^{-15}$  to  $8 \times 10^{-15}$ To confirm that these low energy electrons can drive the cm<sup>2</sup> soluble/insoluble transition in HafSOx we have measured contrast curves using ellipsometry to measure film thickness after exposing HafSOx to 100eV electrons for various electron doses and after development. The soluble/insoluble transition was found to occur in the electron dose range between 100 to 250  $\mu$ C/cm<sup>2</sup>. We have also used Raman spectroscopy to better understand the radiation-induced processes in HafSOx by monitoring the metal coordinated peroxide species after various electron doses. There were very similar reaction kinetics when comparing the increase in O<sub>2</sub> desorption signal during ESD and the decrease in the peroxide Raman signal for increasing electron dose. Finally, we will discuss the radiationinduced mechanisms in HafSOx and relate these to other inorganic photoresists being developed for EUV lithography.

#### 8:20am NS+MN-ThM2 Fundamental Limits of Material Toughening with Molecularly Confined Polymers, *Scott Isaacson*, Stanford University, *K. Lionti, W. Volksen, T.P. Magbitang*, IBM Almaden Research Center, *R.H. Dauskardt*, Stanford University, *G. Dubois*, IBM Almaden Research Center

Low-density hybrid molecular materials with organic and inorganic components engineered at molecular length scales can be made to exhibit diverse mechanical, thermal, and optical properties. We present a novel class of hybrid nanocomposites created through a unique backfilling approach in which selected polymers are homogeneously infiltrated into the pores of a sol-gel nanoporous glass scaffold, leading to uniform mixing at unprecedentedly small length-scales (~1 nm) and confinement of polymer chains to dimensions far smaller than their bulk radius of gyration [1-3]. The second-phase material may be chosen from an extensive library of polymers, allowing for the development of composites with novel electrical, optical, and mechanical properties. This synthesis technique is versatile and can produce uniform, high-quality films over large areas.

We show that it is possible to dramatically improve the mechanical and fracture properties of a nanoporous organosilicate matrix by filling the porosity with a polymeric second phase. The degree of toughening is shown to increase with the polymer molecular weight, and is also found to depend on synthesis conditions. These studies of confined polymers enable us to explore the fundamental limits of nanocomposite toughening in terms of molecular strength, molecular size, and degree of confinement. We describe a novel toughening mechanism based on the molecular bridging and pullout of individual confined polymer chains from the porous matrix, distinct from the more common entanglement-based crazing mechanisms exhibited by bulk polymers. This mechanism is supported and quantified with a model that describes the nanomechanical processes occurring on the length scale of individual polymer chains. The toughening model is further leveraged to calculate the tensile strength of individual polymer chains and find it in agreement with our own independent estimates of molecular strength. This study provides new insight into the mechanical behavior of polymer chains under nanoscale confinement and suggests potential routes for increasing the cohesive strength of multifunctional nanocomposites, where the traditional bulk toughening mechanisms may be absent.

[1] T. Frot, W. Volksen, S. Purushothaman, R. Bruce, G. Dubois, Adv. Mater. 2011, 23, 2828-32.

[2] T. Frot, W. Volksen, S. Purushothaman, RL. Bruce, T. Magbitang, DC Miller, VR. Deline, G. Dubois, *Adv. Funct. Mater.* 2012, 22, 3043-3050.

[3] W. Volksen, K. Lionti, T. Magbitang, G. Dubois, *Scripta Mater.*, 2014, 74, 19-24.

8:40am NS+MN-ThM3 Nanomanufacturing from Silicon to DNA, James Liddle, Center for Nanoscale Science and Technology, National Institute of Standards and Technology, Gaithersburg, MD 20899-6203 INVITED

The production of integrated circuits using silicon fabrication technology is the dominant nanofabrication technology in the world today. However, the industry is maturing, and the technology used for integrated circuit manufacturing, although extraordinarily impressive, is suited, economically, only for that function. At the same time, the nanotechnology revolution has delivered an array of novel structures and materials with fascinating and useful properties, but has presented us with several challenges. These include scaling up production and reducing costs to levels that are commercially interesting, and finding ways of integrating heterogeneous nanostructures into fully functional systems. I will illuminate these issues with a brief description of the strengths and weaknesses of the semiconductor manufacturing paradigm, a comparison with the possibilities offered by DNA-mediated assembly, and an illustration of how a deep understanding of nanoscale physics can turn optical metamaterials from a laboratory curiosity into a manufacturable product.

#### 9:20am NS+MN-ThM5 Pattern Formation by Ion Beam Sputtering on Thermally Treated Ge Surfaces Implanted with Si Ions, *AngélicaGuadalupe Hernández, Y. Kudriavtsev*, CINVESTAV-IPN, Mexico

We have investigated formation of patterned surfaces after low energy oxygen  $(O_2)$  ion sputtering on germanium (Ge) crystals implanted with silicon (Si) ions and subsequent thermal annealing by using Atomic Force Microscopy (AFM).

A commercially available Ge crystal wafer was implanted with Si<sup>+</sup> ions. The energy of implantation was 25 keV. This low energy and the high dose regime  $(2x10^{16} \text{ cm}^{-2})$  results a relatively high Si concentration obtaining at the implanted maximum of  $5.3x10^{21}$  atoms/cm<sup>3</sup>. Distribution and depth near-surface of implanted ions were calculated by Montecarlo simulations using TRIM software as 15 nm and confirmed by the Secondary Ion Mass Spectrometry (SIMS).

After ion sputtering a study of the surface morphology was performed by AFM for comparison of the obtained results with the Ge surface without implantation. In other hand, Raman spectra of the as implanted samples shows the vibration modes of  $\alpha$ -Ge, c-Ge, Ge-Si,  $\alpha$ -Si and Si-Si, which is an indicator of Si clusters formation in to the Ge bulk due to the ion implantation.

In order to recover the crystallinity of the Ge lattice, the thermal annealing at 700 °c during 5min under nitrogen ( $N_2$ ) atmosphere was performed. The corresponding Raman spectra shows the transition from amorphous to crystalline Ge.

After the thermal process, ion beam sputtering was performed by using  $O_2$  ions and energy beam of 0.5, 1 and 2 keV at different ion doses under ultrahigh vacuum conditions (base pressure ~10<sup>9</sup> mbar). Due to the differences in the sputtering yield between Ge and Si, the preferential sputtering of Ge results in an enrichment of Si on the surface. The Si nano-crystals produces an effect of "shadow" leading to a formation of nano-structured surfaces. This results were compared with our previous work, where Ge surfaces (not implanted) were bombarded under the same sputtering conditions (ion specie, energy and ion dose).

Morphology of the surface was investigated by AFM of the original surface, as implanted samples, thermally treated and after ion bombardment. Finally, we compared the experimental morphology with ones obtained previously by the oxygen sputter Ge crystal.

9:40am NS+MN-ThM6 Poor Q-factor? - No Problem: Nano-Optomechanical Mass Sensing in Ambient Conditions, *Swapan Roy*, *V.T.K. Sauer, A. Venkatasubramanian, W.K. Hiebert*, University of Alberta and The National Institute for Nanotechnology, Canada

It has been demonstrated that optimum dynamic range (DR) and high quality factor (Q) of NEMS resonators provides unprecedented mass sensitivity [1]. The mass sensitivity and frequency stability of these devices are limited by their thermomechanical (TM) noise. TM noise goes down with Q as pressure increases, at the same time enhanced critical amplitude leads to a higher DR value with better sensitivity. However, detecting TM noise signal at ambient condition is always challenging. Optomechanical transduction successfully resolved this challenge with high displacement sensitivity and high bandwidth of NEMS devices [2]. Previously we have demonstrated robustness of our optical racetrack resonator transduction scheme in detecting TM noise signal [3]. Taking advantage of this measurement protocol we have found zeptogram level mass sensitivity at atmospheric pressure for a double clamped beam. This is similar to the sensitivity in high vacuum, even though Q-factor drops 300 fold from vacuum to ambient pressure. These intriguing experimental results challenge assumptions about fundamental limits of mass sensitivity of NOMS sensors and open the door for ultrasensitivity in ambient conditions.

1. K. L. Ekinci, Y. T. Tang and M.L. Roukes, "Ultimate limits to inertial mass sensing based upon nanoelectromechanical systems.", J. Appl. Phys. Vol. 95. No.5, March 2004.

2. M. Li, W. H. P. Pernice, C. Xiong, T. Baehr-Jones, M. Hochberg, and H. X. Tang, "Harnessing optical forces in integrated photonic circuits." Nature, Vol.456, pp.480-4, Nov.2008.

3. V. T. K. Saur, Z. Diao, M. R. Freeman, and W. K. Hiebert, "Optical racetrack resonator transduction of nanomechanical cantilevers." Nanotechnology, Vo.25, 05522, 2014.

11:00am NS+MN-ThM10 Frequency Division Using a Micromechanical Resonance Cascade, K. Qalandar, M. Sharma, B. Gibson, Kimberly Turner, University of California, Santa Barbara INVITED

A coupled micromechanical resonator array demonstrates the first successful realization of multi-stage frequency division. Frequency converters, dividers and multipliers, are necessary over a wide range of frequencies for a variety of applications, including vibration energy harvesters, RF transceivers, phase-locked loops, and quantum cascade lasers. In the VHF and UHF frequency range, solid-state and electromagnetic devices have traditionally dominated on-chip signal processing activities due to their wide bandwidth operation, programmability, and ease of implementation. However, design becomes more complicated and noise increases when cascading electronic dividers, due to buffers, amplifiers, and complex impedance matching circuits. Increasing constraints in size, power, and phase noise have led microelectromechanical systems (MEMS) to be considered in place of many traditional electronic elements. In particular, MEMS resonators are of interest since these high Q narrow bandwidth devices can provide better phase noise characteristics than similar devices that utilize amplifier technology. Coupled vibratory modes in micro-resonators have been shown to provide phase noise improvements in frequency sources, and have been considered in other contexts. This frequency divider combines the benefits of cascading, internal resonance, and mechanical coupling in a single microdevice. The operation is based on nonlinear dynamics and exploits the robustness of parametric resonance. This narrow-band approach uses a subharmonic resonance cascade in a chain of internally resonant subsystems with specific coupling that allows energy exchange between successive divide-by-two stages. The mechanical structure consists of a set of N sequentially perpendicular microbeams that are connected by relatively weak elastic elements such that the system vibration modes are localized to individual microbeams and have natural frequencies with ratios close to 1 : : 2N. Conservative (passive) nonlinear inter-modal coupling 2 : provides the required energy transfer between modes and is achieved by finite deformation kinematics. When the highest frequency beam is excited, this arrangement promotes a cascade of subharmonic resonances that achieve frequency division of 2j at microbeam j for j = 1; :: :; N. Results are shown for a capacitively driven three-stage divider in which an input signal of 824 kHz is passively divided through three modal stages, producing signals at 412 kHz, 206 kHz and 103 kHz. The system modes are characterized and used to delineate the range of AC input voltages and frequencies over which the cascade occurs. This narrow band frequency divider has simple design rules that are scalable to higher frequencies, and can be extended to a larger number of modal stages.

#### 11:40am NS+MN-ThM12 Single-Molecule Analysis with Nanomechanical Systems, Michael Roukes, California Institute of Technology INVITED

Mechanical resonators based on NEMS (nanoelectromechanical systems) now enable measurement of the inertial mass of individual atoms and molecules [1]. We have employed NEMS to realize a new form of mass spectrometry (MS) with single molecule sensitivity, and have demonstrated the analysis of individual large-mass biomolecular complexes, one-by-one, in real-time [2]. In this talk I will survey progress and prospects in this emerging field. In particular, NEMS-MS offers transformational capabilities for the field of native MS, which focuses upon the topological investigation of intact protein complexes with high sensitivity and a theoretically unrestricted mass range. Recently, we have developed an approach that greatly enhances the capabilities of NEMS-MS by enabling imaging the spatial mass distribution of individual analytes - in real time, and with molecular-scale resolution - when they adsorb onto a NEMS resonator [3]. This new approach, which we term inertial imaging, employs the discrete, time-correlated perturbations induced by each single-molecule adsorption event to the ensemble of modal frequencies of a NEMS resonator. The spatial moments of each adsorbing analyte's mass distribution are deduced from the discrete, time-correlated shifts it induces to a multiplicity of vibrational modes. The lowest moment of the measured mass distribution function yields the total analyte mass; higher moments reveal the center-ofmass position of adsorption, the analyte's average diameter, and its spatial skew and kurtosis, etc. Together, these higher moments completely characterize the analyte's molecular shape. Once acquired, these moments can subsequently be inverted to yield an "inertial image" of each analyte. Unlike conventional imaging, the minimum analyte size detectable through nanomechanical inertial imaging is not limited by wavelength-dependent diffraction phenomena; instead frequency fluctuation processes determine the ultimate attainable resolution. Advanced NEMS devices are capable of resolving molecular-scale analytes.

 Naik, A. K., Hanay, M. S., Hiebert, W. K., Feng, X. L. & Roukes, M. L., Towards Single-molecule Nanomechanical Mass Spectrometry. Nature Nanotechnology 4, 445–450 (2009). [2] Hanay, M. S., Kelber, S. I., Naik, A. K., Chi, D., Hentz, S., Bullard, E. C., Colinet, E., Duraffoug, L. & Roukes, M. L., Single-protein Nanomechanical Mass Spectrometry in Real Time. Nature Nanotechnology, 7, 602-608 (2012).

[3] Hanay, M. S., Kelber, S. I., O'Connell, C. D., Mulvaney, P., Sader, J. E. & Roukes, M. L., Inertial Imaging with Nanomechanical Systems. Nature Nanotechnology 10, 339-344 (2015).

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