

Monday Morning, October 20, 2008

MEMS and NEMS

Room: 206 - Session MN-MoM

Integrative Materials and Processes for MEMS/NEMS

Moderator: E. Gousev, Qualcomm

8:20am MN-MoM1 Integrated Piezoelectric RF MEMS Front-Ends, G. Piazza, University of Pennsylvania **INVITED**

This paper reports on the work performed in Dr. Piazza's laboratory for the realization of integrated piezoelectric RF MEMS front-ends. The work deals with three different aspects of the concept of MEMS integration: (i) integration of different piezoelectric devices such as resonators, filters and switches to create single-chip RF signal processors; (ii) integration of dissimilar materials such as thin film diamond and AlN piezoelectric films to enhance device quality factor and raise frequency of operation of resonators and (iii) integration or, more appropriately, considerations for integration with state-of-the-art CMOS electronics. The fundamental challenges faced from material, fabrication and design perspectives to attain the aforementioned three levels of integration are highlighted. For example, stress control, material and processing compatibility are important components that need to be taken into account in the demonstration of AlN switched filter banks. Surface roughness and etching compatibilities pose limitation in the fabrication of thin film diamond/AlN micromechanical resonators and constrain the design space. Furthermore, the material stack, deposition temperature and etching techniques need to be selected so that they are compatible or available in CMOS foundries. Design and experimental results demonstrating, for the first time, switched piezoelectric resonators and initial steps towards the integration of thin film diamond/AlN resonators are presented. These preliminary demonstrations set the foundations for the development of new classes of devices that can disrupt the way we currently perform RF signal processing by enabling fast frequency hopping and low power frequency synthesis in a broad frequency spectrum that could not be previously covered by any other MEMS technology.

9:00am MN-MoM3 Chemically-Modified Graphene Nanomechanical Resonators, M.K. Zalalutdinov, SFA Inc., J.T. Robinson, E.S. Snow, Z. Wei, P.E. Sheehan, J.W. Baldwin, B.H. Houston, Naval Research Laboratory

High quality factor ($Q \sim 4000$) radio frequency (20-100 MHz) nanomechanical resonators are fabricated using suspended ultra-thin films of chemically modified graphene (CMG).¹ The films were prepared by spin-casting graphene oxide platelets onto SiO₂/Si substrates, reducing back toward graphene using chemical and/or thermal treatments, then lifting-off and transferring films to patterned substrates. Large-area continuous films can be deposited using this method, enabling batch fabrication of nanoelectromechanical devices. Membranes as thin as 4 nm can be successfully transferred and suspended over 2.7 μm diameter holes. The ability to withstand high in-plane tensile stress ($T \sim 10$ N/m, deduced from membrane resonant frequencies) as well as high quality factors show that the integrity of the film is NOT compromised by the inter-platelet bonding. The extremely small mass of these CMG resonators provides an estimate for the added mass sensitivity as low as $\delta m \sim 10^{-18}$ g. In-plane stress inherent to as-fabricated CMG membranes can be dynamically tuned over a wide range due to thermoelastic effects by applying a low power localized heat source. In conjunction with the short thermal relaxation time ($\tau \sim 10^{-8}$ sec) this enables techniques such as parametric pumping for further enhancement of the performance of CMG resonators. Thicker ($h > 15$ nm) suspended CMG films show similar quality factors, can withstand strain in excess of 0.3% and constitute a virtually unpenetrable barrier for water or vapor as confirmed by resonant frequency measurements. Membranes encapsulating water on one side and exposed to vacuum on the other side show no frequency dependence on a time scale of days, indicating perfect sealing. In addition, both the membranes themselves and the adhesion of the CMG film to the substrate are strong enough to withstand boiling of the encapsulated water ($T_{\text{anneal}} > 100^\circ\text{C}$). Finally, ultra-thin CMG films are optically transparent and feature only minor e-beam scattering thereby facilitating access to encapsulated objects for imaging and/or spectroscopy. We will describe mechanical and thermomechanical properties of CMG films extracted from the behavior of the nanoresonators and discuss possible applications in sensing and nanofluidics.

This work was supported by the Office of Naval Research.

¹ Ruoff, R. Nature Nanotechnology 3, 10-11 (2008).

9:20am MN-MoM4 Electrical Transduction of Multi-layer Polysilicon Resonators, J.D. Cross, B.R. Ilic, Cornell University, M.K. Zalalutdinov, SFA Inc., E. Yilmaz, Cornell University, J.W. Baldwin, B.H. Houston, Naval Research Laboratory, H.G. Craighead, J.M. Parpia, Cornell University

A straightforward means of electrical transduction of resonator motion is investigated using MHz-frequency micromechanical resonators made from multi-layer film stacks. Devices are fabricated using polysilicon films stacked on top of each other with intermediate layers of insulating material. Electrical or optical drive is used to induce motion in the resonators and electrical transduction allows for direct detection of the resonator motion. A variety of structure geometries are investigated, including cantilevers, double-clamped beams, mushrooms, and domes. Quality factors of 1000-10000 are routinely observed. We discuss the transduction mechanism as well as the ability to integrate these kinds of MEMS structures into a standard CMOS foundry process with no added or modified fabrication steps. We show that the multi-layer film stack can be delaminated in the release step, resulting in stacked resonator structures with thin gaps between each vibrating surface. This work was partially supported by the Office of Naval Research, DARPA, and fabrication was performed at the Cornell NanoScale Science and Technology Facility.

9:40am MN-MoM5 Selective Detachment of Microspheres using In-Plane Modes of Nanoelectromechanical Oscillators, B.R. Ilic, Cornell University, S. Krylov, Tel Aviv University, Israel, M. Kondratovich, H.G. Craighead, Cornell University

Manipulating dynamics of flexural and torsional vibrational modes of micro- and nanoelectromechanical systems (MEMS and NEMS) with external fields has long been a sought-after goal. A widely studied class of NEMS devices consists of surface micromachined mechanical oscillators made of thin film layers patterned into various shapes that operate by motion perpendicular to the plane of the thin film and substrate by bending in their thin direction. Conventional mechanical driving and motion transduction methods typically activate and detect only motion in this "out-of-plane", transverse direction. We previously demonstrated a robust method for driving and detecting the motion of micro- and nano-scale resonators by utilizing optical drive of resonant motion and interferometric detection of that motion by a separate laser. This technique allowed non-invasive activation and interrogation of individual oscillators or arrays of oscillators. We describe here an approach that can activate and detect the perpendicular, in-plane motion of such oscillators. We show that optical fields are efficient for excitation, direct control and measurement of in-plane motion of cantilever-type nanomechanical oscillators. Using optical excitation and interferometric detection, we dynamically analyzed surface micromachined 200nm and 250nm thick single crystal silicon cantilevers of varying lengths and widths. We also have demonstrated the controlled capture, detection and release of submicrometer particles by the application of forces imparted by the in-plane motion of the resonators. In contrast, the out of plane motion, even in the strong non-linear impact regime, was insufficient for the removal of bound polystyrene spheres. Our results suggest that optical excitation of in-plane mechanical modes provide a unique mechanism for controlled removal of particles bound on the surface of nanomechanical oscillators.

10:20am MN-MoM7 Synthesis and Characterization of Large Area Ultrananocrystalline Diamond (UNCD) Films using Microwave Plasma Chemical Vapor Deposition Process and Integration with CMOS, A.V. Sumant, O. Auciello, Argonne National Laboratory, V. Adiga, A. Koniczek, University of Pennsylvania, X. Zhong, B. Kabius, Argonne National Laboratory, H. Yuan, Z. Ma, University of Wisconsin-Madison, R. Carpick, University of Pennsylvania

Because of exceptional mechanical, chemical, electrical and tribological properties of ultrananocrystalline diamond (UNCD), it has great potential to be used in for the development of high-performance, harsh environment-compatible devices for MEMS and NEMS, such as resonators and switches. Recent work by our group has demonstrated fabrication of functional RF-MEMS switches and resonators based on UNCD. However, transition of this technology to the industry will critically depend on the ability to produce UNCD films on wafer scale with acceptable thickness and microstructure uniformity. We have achieved 4%, 7%, and 11% uniformity in UNCD film thickness across 100 mm, 150 mm, and 200 mm diameter Silicon substrates respectively using 2.45 GHz and 915 MHz microwave plasma chemical vapor deposition (MPCVD) process. All the films were grown in the temperature range of 400-800 oC. We report on the microstructure uniformity, phase, and impurity content of UNCD films by using atomic force microscopy (AFM), Near edge X-ray absorption fine

structure spectroscopy (NEXAFS), and forward recoil spectrometry (FRES) characterization techniques respectively. Additionally, we have developed a materials integration strategy to enable diamond-CMOS integration. Ultrananocrystalline diamond (UNCD), a novel material developed in thin film form at Argonne, is the only diamond film that can be grown at 400oC, and still retain exceptional mechanical, chemical, and tribological properties comparable to that of single crystal diamond. We have developed a process based on microwave plasma CVD to synthesize UNCD films on 150 and 200 mm CMOS wafers, which will open new avenues for building CMOS-driven devices for MEMS/NEMS based on UNCD. UNCD films were grown successfully on individual Si-based CMOS chips and on 200 mm CMOS wafers at 400 oC in a microwave-plasma-enhanced chemical vapor deposition (MPCVD) system with Ar-rich/CH₄ gas mixture. The CMOS devices on the wafers were characterized before and after UNCD deposition. All devices were performing to specifications with acceptable degradation after UNCD deposition and processing. A threshold voltage degradation in the range of 0.08-0.44V and transconductance degradation in the range of 1.5-17% were observed. We also report the on the cross-section TEM/EELS studies of the UNCD/CMOS interface and discuss the possible mechanisms responsible for the degradation of CMOS performance.

10:40am **MN-MoM8 {100}-Textured PZT Films Grown on Chemically Deposited PbTiO₃ Seed Layers for MEMS Applications**, J. Zhong, S. Kotru, H. Han, R.K. Pandey, The University of Alabama

Lead zirconate titanate (PZT)-based thin films are gaining increased interest in wide variety of applications in MEMS due to their large longitudinal and transverse piezoelectric coefficients, and the compatibility with microelectronic circuits. Both micro-machined sensors (such as accelerometer and gyroscope) and actuators (such as micro-motors, micro-pumps, and micro-switches) have been fabricated based on PZT films. The piezoelectric response of PZT films is the key factor for sensing and actuation function of a device; the higher the value, the better the device will act as sensor and/or actuator. Texture of the films plays a major role in determining the piezoelectric response. So far, the highest transverse piezoelectric coefficient (-12.0 C/m²) has been reported for {100}-textured PZT films grown on PbTiO₃ seed layers. These PbTiO₃ were sputtered at 500~600° C. High temperature sputtering limits the practical implementation of such films in MEMS devices due to process constraint and sample size limitations. In this work, highly {100}-textured PZT films have been grown with PbTiO₃ seed layers. However, the seed layers in our work were deposited by chemical solution deposition. The effect of both Pb content and solution concentration of PbTiO₃ on PZT films was investigated extensively. These PZT films show 97% of {100} texture and effective transverse piezoelectric coefficients of -13.3 C/m². Thus our films have higher effective transverse piezoelectric coefficient than the PZT films grown with sputtered PbTiO₃ seed layers. Our approach of obtaining {100}-textured PZT films with high piezoelectric response on chemically deposited seed layers has advantage of being much easy and low-cost. Such films are feasible for MEMS based device implementation.

11:00am **MN-MoM9 Suppression of Anelastic Effects in Micromechanical Resonators from Suspended Al-CNT Nanolaminate Thin-Films**, Y.D. Kim, J.H. Bak, S.W. Cho, B.Y. Lee, S.R. Lee, K. Char, S. Hong, Y.D. Park, Seoul National University, South Korea

We present evidence that the addition of Al-CNT lamina to suspended Al thin-film micromechanical resonators suppress anelastic effects. Addition of Al-CNT lamina to form a metallic-CNT nanolaminate has been shown to enhance mechanical properties, including elastic modulus as well as strengths, from dynamic and quasi-static flexural measurements of suspended doubly-clamped micromechanical beam resonator structures.¹ In this study, dynamic flexural measurements for long loading-cycles (>10¹¹) is presented. The micromechanical beam resonator structures, which are patterned by a combination of e-beam and photolithography methods, are fabricated from UHV sputter deposition of Al onto a self-assembled CNT network on a GaAs substrate, which is selectively removed to suspend the beam. The frequency response of the microresonators is periodically measured by a laser vibrometer-like set-up, while the beam is actuated electrostatically. For Al beam resonators, the resonance frequency (f₀), which is directly related to the its elastic modulus, varies during the duration of measurement ($\Delta f_0 / f_0 < 1.5\%$), while for Al-CNT beam resonators, f₀ is relatively unchanged for the duration. Such observations are consistent with the view that the CNTs mechanically reinforce and is well-incorporated in the Al thin-film, as anelastic effects are attributed to grain boundary sliding and are a contributor to stress relaxation in metallic thin-films.²

¹ J.H. Bak, Y.D.Kim et al., Nature Materials advance online publication, 20 April 2008 (doi:10.1038/nmat2181).

² S. Hyun et al., Appl. Phys. Lett. 87, 061902 (2005).

11:20am **MN-MoM10 An Opto-Thermo-Mechanical MEMS Sensor for Direct Thermal Imaging**, P. Apte, B. Seth, O. Karhade, S. Chiluveru, IIT Bombay, India

Infrared imaging plays a critical role in many applications ranging from night vision, environmental monitoring and astronomy. The paper describes a room-temperature compensated opto-thermo-mechanical un-cooled infrared imaging system with a direct color display. The sensor consists of an array of sensing elements suspended from the substrate using bimorph beam elements. Infrared radiation incident on a sensing element is absorbed and leads to a rise in temperature. The heat conducted from the sensing element to the bimorph beam elements leads to a deformation of the beam elements and results in a displacement of the sensing element in a direction perpendicular to the plane of the sensing element. Thus the lateral positions of the sensing elements is influenced by the infrared energy received by the sensing elements in addition to the room temperature. The lateral movement of sensing element is converted into a color image by interference of reflected light from the sensing element and another parallel element with an air gap suitable for creating a constructive interference in the visible spectral range. This second parallel element is also mounted using similar bimorph beam elements and is made of a material transparent to infrared radiation. Thus the transverse position of the transparent element does not depend on the incident infrared radiation but only the ambient temperature. This way it is possible to cancel the effect of room temperature on the interference pattern. The design considerations for the above device are described here. Various configurations of arranging the elements and the bimorph are discussed along with their relative merits. Simulations were conducted using Ansys© software. For an incident radiation of 100 W/m², the rise in temperature of 0.3mm x 0.3mm sensing element was about 5 degrees C. The sensitivity of the device was found to be of the order of 4 nm²/W. The time constant of the device was found to be about 2 seconds.

11:40am **MN-MoM11 An Optical MEMS Sensor for Catechol Detection**, P.H. Dykstra, J. Hao, S.T. Koev, University of Maryland, G.F. Payne, University of Maryland Biotechnology Institute (UMBI), L. Yu, R. Ghodssi, University of Maryland

Catechol is a widely studied phenol which is a common byproduct of factory waste. Its presence in drinking water and food poses a safety concern due to its toxic and possibly carcinogenic effects. We report the successful fabrication and testing of an optical MEMS sensor for the detection of catechol. Other reported sensors suffer from a lack of selectivity. This sensor marks the first time optical measurements have been utilized for catechol detection on chip. In addition, it provides improved selectivity over conventional detection methods. Typically used detection techniques involve electrochemically oxidizing catechol solution and measuring the current from the reaction over time. However, these methods are prone to false positives since any other easily oxidized chemicals present, such as ascorbic or citric acid, will create a current. Other studies involving catechol detection have shown that byproducts from catechol oxidation will induce a significant absorbance change in an aminopolysaccharide film of chitosan. Chitosan is derived from the biopolymer chitin and has been well characterized by our group in the past. This absorbance change in chitosan caused by catechol oxidation is shown to be the highest in the UV and near UV range of the spectrum. Our reported device takes advantage of this unique absorbance property to detect catechol by measuring the change in light intensity at 472 nm. The device consists of a single microfluidic channel patterned in SU-8 with perpendicular waveguides for guiding light through a deposited chitosan film. Indium Tin Oxide (ITO), a transparent conductor, is used as the cathode on the waveguide facet to facilitate the chitosan film deposition. Chitosan forms a solid film at pH higher than 6.3 which allows it to be selectively deposited onto a cathode during an electrochemical reaction. As catechol flows down the channel it is electrochemically oxidized via patterned electrodes and causes the absorbance change in the chitosan film. Blue laser light is coupled in and out of the device using multimode optical fibers and the intensity is measured by an external spectrophotometer. The higher concentration of catechol contributes to a higher absorbance as expected while oxidizing buffer solution and ascorbic acid display no measurable change in the absorbance through the chitosan film. The data displays a considerable response even for the lowest measured concentration (0.001 M).

MEMS and NEMS

Room: 206 - Session MN+NC-MoA

Fabrication at the Micro- and Nano- Scales for MEMS/NEMS

Moderator: A.V. Sumant, Argonne National Laboratory

2:00pm MN+NC-MoA1 Large Area Nanofabrication for MEMS Applications, L.E. Ocola, A. Imre, Argonne National Laboratory INVITED

As microelectromechanical systems (MEMS) shrink towards nano scale dimensions (or NEMS) the need for novel fabrication techniques increases. In this paper we revisit the use of two well-known techniques and propose new ways they can contribute toward the fabrication of next generation NEMS devices. The two techniques are super-high aspect ratio patterning using high-voltage electron beam lithography, and large area nanofabrication using a focused ion beam tool (FIB). In this paper we report on hydrogen silsequioxane HSQ nanopatterned into super-high aspect ratio structures (aspect ratio > 10) using 100 KV e-beam lithography, along with development in aqueous TMAH solution and DI water rinse, both performed at elevated temperatures (60 °C). Hot development allows for the rapid removal of low molar mass uncrosslinked molecules from the exposed regions while preserving the mechanical integrity of the nanopatterned structures. Raising the water rinse temperature to 60 °C also has the benefit of reducing the water surface tension by about 10%. Preliminary results of 90 nm structures using 1.2 micron thick HSQ, i.e. aspect ratios of 12, have been obtained. Such structures have immediate application in MEMS, Fresnel zone plate fabrication, and nanophotonics among others. We also have explored the use of focused ion beam (FIB) lithography in similar fashion to that of electron beam lithography. Although FIB has been employed extensively as a single write-field exposure tool for small device tailoring, mask repair, and sample characterization, it is rarely used for nanoscale patterning on a large area. This requires a high precision stage, and additional lithography software to handle complex and large pattern designs. In general, neither the hardware nor the software of FIB instruments is prepared for this task. We configured a FEI Nova 600 Nanolab Dual Beam FIB system with a 100 nm resolution X-Y stage, a Raith Elphy Lithography software interface and a Raith 16-bit DAC pattern generator for the X and Y deflectors. This provides us with the capability to expose more than 65,000 pixels/axis in one write-field, and allows us reading layout designs from files in GDSII format. Both capabilities are typical and standard in any medium-level e-beam lithography tool, but not found until recently in FIB systems. We present various large-area patterns milled in silicon and diamond thin films, and evaluated for stitching accuracy.

2:40pm MN+NC-MoA3 Large-scale Fabrication of Silicon Nanowire NEMS Devices Using a Top-down Approach, C.S. Roper, University of California, Berkeley, R.T. Howe, Stanford University, R. Maboudian, University of California, Berkeley

While bottom-up nanofabrication techniques, including vapor-liquid-solid (VLS) growth, can create single crystalline nanostructures,¹ the integration of a single nanowire into a functioning, addressable device is an extremely difficult task, and problematic on a large scale, owing to the stochastic nature of the growth process. The primary barriers to bottom-up integration are manifold. First, nanowire placement and alignment are difficult to control. VLS nanowire growth is catalyzed by molten eutectic alloy nanoclusters that wander erratically on the substrate upon heating, and often coalesce with one another, rendering even size control unacceptably loose. Furthermore, transmission of signals to and from a nanostructure is problematic due to difficulty forming either directly contacted or capacitively coupled electrodes. Electron-beam lithography can be used to pattern electrodes on a bottom-up grown nanostructure to create a device,² but its high cost and serial nature make it ineffectual for the realization of large arrays of interconnected devices. Unlike bottom-up techniques, top-down microfabrication techniques, including projection lithography, oxidation of silicon, chemical vapor deposition (CVD) of thin films, and plasma etching, readily lend themselves to precise placement, alignment, and ultra large scale integration. However, the minimum feature size and alignment error limitations of optical lithography preclude the direct patterning of nanoscale devices. We present a manufacturable fabrication process to realize large arrays of individually addressable silicon nanowire resonators using an entirely top-down approach that relies on optical projection lithography and multiple steps of controlled oxidation. Our fabrication process uses novel and elegant mask and process design to overcome the limitations of traditional top-down processes, yielding arrays

of precisely positioned, vertically aligned, and electrically connected silicon nanowires with diameters as small as 30 nm. With the aim of creating ultra-sensitive mass sensors, devices with a single vertically aligned silicon nanowire as the resonant mass and multiple electrodes spaced hundreds of nanometers from the nanowire are also fabricated with the top-down process.

¹J. Westwater et al., J. Vac. Sci. Technol. B., 15 (3) 554-557, 1997.

²H. T. Soh, et al. Appl. Phys. Lett. 75, 627-629, 1999.

3:00pm MN+NC-MoA4 Fabrication of Nanopore and Nanochannel Structures through E-Beam Lithography and Atomic Layer Deposition Processes, S.W. Nam, IBM T.J. Watson Research Center & Seoul National University, Korea, M.J. Rooks, R. Sirdeshmukh, IBM T.J. Watson Research Center, K.B. Kim, IBM T.J. Watson Research Center & Seoul National University, Korea, S.M. Rossnagel, IBM T.J. Watson Research Center

The reliable and reproducible fabrication of nanopore and nanochannel structures is essential for building nano-fluidic systems, such as a device to control bio-molecules or DNA. Here, we report on CMOS-compatible fabrication methods to generate electrode-embedded nanopore and nanochannel structures as small as 10 nm feature size. To fabricate a small size fluidic channel, we adopt a combined process of e-beam lithography and atomic layer deposition (ALD). E-beam lithography is used for generating few tens of nanometer sized hole and line structures and ALD is employed for further shrinking down the features less than 10 nm. In the case of a pore fabrication, we used a membrane of SiO₂ (100 nm)/Si₃N₄ (20 nm)/TiN (30 nm)/Si₃N₄ (20 nm) on Si wafer. PMMA (300 nm thickness) hole structures on top of the membrane were patterned by e-beam lithography, which were then transferred to the membrane layers through two-step RIE process, consisting of sequential CHF₃+O₂ and CH₄ steps to remove SiO₂ and Si₃N₄/TiN/Si₃N₄, respectively. This drilling process formed 50-80 nm diameter pore structures on membrane. In a parallel way, we fabricated trench structures by using a stack of Si₃N₄ (5 nm)/a-Si (20 nm)/SiO₂ (100 nm) films on a bulk Si substrate. PMMA (200 nm thickness) line patterns fabricated by e-beam lithography were transferred to the Si₃N₄, a-Si and SiO₂ layers through a multi-step RIE process, which generated ~50 nm width and ~60 nm depth trench structures. Then, a selective isotropic etching of SiO₂ by a dilute HF(1:500) induced a round-shaped etch profile along the trench line. Alternatively, the a-Si layer could be oxidized by furnace oxidation to partially close the trench. For both pore (70-80 nm diameter) and trench (~50 nm width) structures, we used an amorphous, conformal ALD TiO₂ film to reduce the pore or trench dimensions down to less than 10 nm. ALD film provided a self-limiting process by remaining void along the pore and the trench, which will serve as fluidic channels less than 10 nm.

3:20pm MN+NC-MoA5 Fabrication of Nickel-tipped Cantilevers for Magnetic Resonance Force Microscopy, S.A. Hickman, Cornell University, J. Van Delden, Eigenphase Technologies, L.E. Harrell, United States Military Academy, S.R. Garner, J.C. Ong, S. Kuenh, J.A. Marohn, Cornell University

Magnetic resonance force microscopy (MRFM) is a technique that may one day allow us to acquire magnetic resonance images of single molecules – an extremely exciting prospect. To date we have demonstrated that MRFM can achieve a sensitivity of ~10³ proton spins, using a custom-fabricated silicon cantilever with a hand glued, 9 μm diameter magnet tip. By making improved magnetic tips and mitigating surface dissipation, it may be possible to achieve single-proton sensitivity, leading to such exciting prospects as structural determination of large biomolecules. Achieving the attonewton force sensitivity necessary to image single proton spins requires custom-fabricating cantilevers with extreme aspect ratios. In MRFM the force exerted on the cantilever, per spin, is proportional to the field gradient from the cantilever's magnetic tip. Achieving single proton sensitivity therefore also requires dramatically reducing magnet size. Unfortunately, all MRFM tips produced to date have been made by manually affixing magnets one-at-a-time to a cantilever. Even if the tips are ion-beam milled, it is difficult to see how they can be made small enough to detect a single proton. Likewise, ion-beam deposited tips have not yet reached the purity to produce the high magnetic field gradients desired for MRFM. We have developed an electron-beam-lithography (EBL) process for batch fabricating nanoscale tip magnets on ultrasensitive silicon cantilevers. Research by our group has shown that surface induced dissipation is a major source of noise. This surface dissipation results from charge in the cantilever interacting with electric field fluctuations in the sample. This surface dissipation can be minimized by fabricating the magnets overhanging the end of the silicon cantilever. We will present 50-600 nm wide nickel overhanging magnets fabricated by EBL and isotropic plasma etching. We will also present work on producing overhanging magnets via

anisotropic potassium hydroxide etching, and by fabrication of magnets over sacrificial oxide pillars. Our current challenge appears to be preventing the transformation of the magnet metal to metal silicide during the fabrication process. With our designed cantilever, we expect a sensitivity of better than 10^5 protons.

4:00pm MN+NC-MoA7 A Process for Control of the Support Conditions of Nanomechanical Beam Resonators, and Evaluation of the Resulting Impact on Mechanical Dissipation, R. Barton, S.S. Verbridge, B.R. Ilic, H.G. Craighead, J.M. Parpia, Cornell University

We have developed a fabrication process for suspended nanomechanical beam structures which allows precise control over the clamping conditions, particularly the extent of the overhanging support which results from the usual sacrificial release of such devices. We localize the sacrificial silicon dioxide material to specified locations using a process involving a chemical mechanical polishing step, followed by a deposition and patterning of the structural silicon nitride material. With proper alignment of the beam structures, we have succeeded in controlling or entirely eliminating the overhang which was previously thought to potentially impact the overall dissipation in these resonant systems. We will demonstrate that for a moderate overhang, on the order of ten percent of the resonator length, dissipation measured in terms of resonator quality factor, Q , is not significantly affected either in vacuum or ambient air. This finding demonstrates that neither the intrinsic material damping at low pressure, or the gas-related damping that is dominant at higher pressures are affected by the support overhang that inevitably results from typical release steps. We anticipate that the control over the clamping conditions we present could prove useful for more complex nanosystems, for example suspended fluid-containing nanochannels which integrate both optical and mechanical means of biomolecular detection.

4:20pm MN+NC-MoA8 Dynamic-Mode Multidirectional UV Lithography with Liquid State Photoresist, J.K. Kim, Y.K. Yoon, University at Buffalo

Recently an advanced ultraviolet (UV) lithography scheme, where UV is applied to liquid state photoresist, has been introduced with several advantages such as short process time with quick develop/rinse, and in-situ surface modification.¹ The container with liquid photoresist remains in a horizontal position to vertically incident light, resulting in relatively simple micro structures. In this research, a multidirectional UV lithography process has been developed with liquid state photoresist for complex three dimensional (3-D) microstructures, where the UV source and a substrate containing liquid photoresist have an arbitrary angle each other. A leak free container has been devised to hold liquid photoresist and is capped with the photomask plate. The container set is attached to a computer controlled movable stage with tilting and rotational functionality.² A liquid state negative tone photopatternable polyurethane, LF55GN has been utilized for test since it shows fast realization of thick structures with high aspect ratio.³ While the liquid state photoresist is exposed to UV, the stage is dynamically tilted and rotated in slow speed, at which the friction force should be greater than the inertia effect for the given viscosity of the photoresist and therefore the container and the enclosed liquid move as a single body in a quasi-static mode. This developed scheme provides all advantages of both the high aspect ratio complex 3-D micropatterning of multidirectional UV lithography with $SU-8^2$ and the rapid process time of UV lithography with liquid state photoresist.¹ As the height of the container determines the height of the structure, several thousand micro meter thick film can be easily realized unlike the spin coating approach where the thickness is limited to several hundred micrometers. Thus complex 3-D structures which are more than 1000 micrometers tall structures have been successfully demonstrated in 30 minutes. Ray tracing with regard to the stage movement has been simulated. The shapes of the fabricated structures show good agreement with those of the simulated structures. A single vertical triangular slab and an array have been demonstrated as test structures.

¹A. Sayah et al JMEMS 2007, vol.16, no.3, pp.564-570, June

²J.K. Kim et al MEMS 2008. Conference, vol., no., pp.399-402, Jan.

4:40pm MN+NC-MoA9 Development of High Rate Etching for Deep Si Etching in Advanced NLD Plasma, T. Murayama, T. Morikawa, K. Suu, ULVAC, Inc., Japan

In Si etching such as wafer level packaging technology for MEMS and the formation vertical interconnection of three-dimensional integration LSI, to establish high rate is important problem. Especially under 10um space pattern on 200-mm-diam wafer, it is desired to etch Si at high rates achieving anisotropic features. Commonly, it is essential to produce huge amount of F radicals in Si etching mainly dominated by radical reaction in Si surface, ion assist effect is important to achieve anisotropic feature.¹ Our experiments are conducted using NLD-Si etcher which we introduced sputtering system for sidewall passivation to NLD (magnetic Neutral Loop Discharge) etcher for quartz deep etching.^{2,3} In this study, we tried to

improve etching rate using a novel antenna [Multi-Slits rf Antenna] for controlling both ion and F radical density. This antenna consisted of grid type structure in which several slits are set perpendicularly in same direction to RF current. In experiments we used 3 parallel slit antenna in other words 4 parallel current paths structure, expecting high-density plasma is generated because of ion and radical generation area extending in space compared with simple single-turn antenna. Next, for control anisotropic feature, we investigated the dependence of bias rf frequency in high-density NLD plasma using this multi slit rf antenna. We used SF₆ as process gas. Consequently, this antenna improved in etching rate more than two times compared with our conventional antenna. However etching feature shifted bowing. We considered that this result is caused by not only F radical increase but also sheath impedance decrease by high-density plasma. To overcome this sheath impedance decrease, we changed bias rf frequency to 2 MHz from 13.56 MHz. In low bias frequency we etched 4 um space pattern, then high rate and anisotropic feature were achieved etching rate is 10.5 um/min, selectivity (Si/SiO₂) is 60 over. It is considered that for high rate and high quality Si etching it is very important that the control of incident ion energy distribution in addition to high-density radical produce.

¹ J. W. Coburn and H. F. Winters: J. Appl. Phys. 50 (1979) 3189.

² W. Chen, et al.; J. Vac. Sci. Technol., A 19 (2001) 2936.

³ Y. Morikawa, et al.; Thin Solid Films 515 (2007) 4918.

5:00pm MN+NC-MoA10 XeF₂ Etching of Si and Mo for MEMS Manufacturing, J.-F. Veyan, Y.J. Chabal, University of Texas at Dallas, X.-M. Yan, A. Londergan, E. Gousev, Qualcomm

XeF₂ is a widely used isotropic etchant in MEMS and NEMS fabrication because it selectively removes a large variety of pure compounds (e.g. Si, Ge, Mo, W) but not their stoichiometric oxides. Understanding the etching mechanisms is crucial to achieve highly selective etching. We have studied and compared the XeF₂ etching of Silicon oxide-Silicon and Molybdenum Oxide -Molybdenum systems, under typical pressure conditions used in industry (~ few Torr). Using in-situ Infrared Absorption Spectroscopy (IRAS) we have investigated the reaction kinetics and characterized the gas phase, surface and subsurface species after and during the etching process. To reduce side effects due to reaction with the reactor walls and sample holders, an all aluminum chamber has been designed, with Teflon gasket and Kalrez O-ring for sealing. The length of stainless steel tubing for gas transport has also been reduced. To minimize reactions with contaminants such as water and hydrocarbon, the reactor is pumped to 10^{-5} Torr prior any XeF₂ introduction. The sample temperature prior and during XeF₂ exposure is controlled by heaters made with Ta filaments inserted in a ceramic frame imbedded inside the sample holders. A chromel-alumel thermocouple is placed directly in contact with the sample surface. With the sample out of the IR beam path the reaction products are monitored during and after etching process. Transmission is used to probe SiO₂/Si, and grazing angle reflection to probe MoO₃/Mo, and surface and subsurface species are detected/identified during and after etch reactions. XeF₂ induces a strongly exothermal reaction with both Si and Mo, producing gaseous SiF₄ (1030 cm⁻¹) and MoF₆ (741 cm⁻¹), and incorporating Fluorine species into the subsurface region. No reactions have been observed with stoichiometric Silicon Oxide and Molybdenum Oxide, but both oxides are completely removed when overlaying the pure material. The removal of the oxide overlayers is believed to be due to fluorine migration through the oxide and reaction with the elemental Si or Mo at the oxide-element interfaces.

5:20pm MN+NC-MoA11 A Versatile, Bilayer Resist Method for Creating Silica Microstructures, B.R. Cipriany, B.R. Ilic, H.G. Craighead, Cornell University

The rapid and widespread acceptance of polydimethylsiloxane (PDMS) in the microfabrication community illustrates the growing importance of versatile, simple, and inexpensive fabrication techniques, particularly for lab-on-a-chip applications. In this context, we demonstrate a new method for creating silica microfluidic networks over nanophotonic structures using a bilayer resist process involving Hydrogen silsesquioxane (HSQ) and a single photolithographic step. Ridge waveguides 180nm tall were formed in silicon nitride using electron beam lithography (JEOL9300FS) and reactive ion etching. A 630nm thick layer of HSQ was spun conformally over the structures and then exposed to oxygen plasma to cross-link a 10nm thin barrier layer. This barrier was robust against photoresist solvents, allowing a bilayer stack to be formed without altering the underlying HSQ bulk. Photoresist was then spun, patterned with optical lithography, and used as a mask layer. An HF based chemical etch was used to transfer the pattern into the barrier layer, followed by development to isotropically dissolve the HSQ bulk. Microfluidic networks formed with this developer-based transfer were self-terminated on the photonic structures without inducing structural damage. We studied the formation of HSQ channels with widths of 1.5-3.1 micrometers and heights of 80-520nm, respectively. Cross-sectional electron micrographs of these channels revealed a sponge-like film composition, which was compacted into a dense, amorphous silica film

during a subsequent high-temperature anneal. Post-anneal measurements with a variable-angle spectroscopic ellipsometer reveal a 17% reduction in film thickness and a negligible complex refractive index over the entire visible spectrum, relative to non-annealed films. Using an inverted microscope with photon counting modules, laser-induced autofluorescence of annealed HSQ was found to be over an order of magnitude less than PDMS, suitable for ultra-sensitive fluorescence spectroscopy. Unlike PDMS, annealed HSQ demonstrated chemical resistance in both aqueous solutions and common solvents. Within our sealed waveguide-microfluidics network, we directly observed flow of fluorophore-labeled deoxyribonucleic-acid (DNA) using fluorescence videomicroscopy. Future applications of this fabrication method include microfluidics integration with MEMS/NEMS, nanowire sensors, or other integrated optical elements.

Tuesday Morning, October 21, 2008

BioMEMS Topical Conference

Room: 309 - Session BM+MN+BI+BO-TuM

MEMS/NEMS for Biology and Medicine

Moderator: E. Meng, University of Southern California

8:00am **BM+MN+BI+BO-TuM1 Microfluidic Systems for Cell Growth and Analysis**, *K.F. Jensen, A. Adamo, L. Ye, Z. Zhang*, Massachusetts Institute of Technology **INVITED**

We present microfluidic systems for cell growth, including instrumented microreactors in which the main process parameters (e.g., optical density, dissolved oxygen and pH) are monitored optically and controlled. The system accommodates bioreactors in different operational modes, batch and continuous. The systems are disposable and consist of layers of poly(methyl methacrylate) for structural integrity and poly(dimethyl siloxane) (PDMS) layers for aeration. We also combine cell growth with analysis of protein responses underlying cell signaling. Analysis of these potentially fast transient events requires very short treatment times and well-controlled and reproducible stimulus conditions. Consequently, such pathways can be difficult to probe reproducibly with conventional laboratory techniques that are susceptible to small fluctuations in manual handling – in particular at short times. Microfluidic systems provide for reproducible and automated analysis with excellent control over experimental conditions. We describe microfluidic based methods for investigating signaling pathway ways of adherent cells with the overall aim of controlling cell culture, cell stimulation, and the subsequent protein analysis. The devices, which are fabricated in PDMS by soft lithography, enable dynamic studies of cell signaling by taking advantage of the equivalence between distance travelled along a microfluidic channel and treatment time. They perform all the necessary steps needed in stimulus-signal response analysis of signaling pathways by a fluorescent immunocytochemical assay including cell culture, cell stimulus, cell fixation, and antibody analysis. Average cell population data are obtained by scanning and imaging the entire device, while high resolution microscopy moving along the channel allows responses to be collected at the single cell level. Finally, we present microfluidic devices for quantitative microinjection of macromolecules and nanoparticles into living cells. These approaches overcome limitations with traditional manual manipulation of microinjection needles.

8:40am **BM+MN+BI+BO-TuM3 High-Throughput pMHC Microarrays for Characterizing Diverse T Cell Populations**, *M. Paulaitis*, Ohio State University and Johns Hopkins University, *C. Yue, N. Guzman*, Ohio State University, *J. Schneek, M. Oelke*, Johns Hopkins School of Medicine **INVITED**

We are developing protein microarrays for rapidly scanning and screening diverse T cell populations to characterize human adaptive immune responses. An important early molecular recognition event that triggers an immune response is the interaction of a T cell receptor (TCR) on the surface of the T cell with its complementary major histocompatibility complex (MHC) on the surface of antigen-presenting cells. This interaction is mediated by a small peptide (the antigen) 8-10 amino acids in length bound to the MHC, such that the amino acid sequence of the peptide antigen determines the specificity of the TCR/peptide-MHC (pMHC) interaction. Surface plasmon resonance studies of TCR/pMHC interactions have established that the overall range of binding affinities of stimulatory pMHC ligands is low relative to that for anti-body-antigen interactions. Yet, these interactions have remarkably high specificity/sensitivity leading to T cell activation and different immune responses depending on the nature of the peptide. Individual T cells are also characterized by a unique TCR; therefore, pMHC microarrays printed with peptides having different amino acid sequences serve to distinguish T cells by their characteristic TCR/pMHC interactions. In addition, co-printing antibodies against cytokines secreted by the captured T cells enables an antigen-specific functional analysis of T cell activation across this population. We show that pMHC microarrays can selectively capture and enumerate antigen-specific T cells in diverse populations at high sensitivity, and that this information provides insights into the general principles governing early molecular recognition events in human immune responses. Results on the functional diversity of the human immune response will also be presented. This work is supported by the National Science Foundation (BES-055281) and the National Institute of Allergy and Infectious Diseases of the National Institutes of Health (1R21AI077097-01).

9:20am **BM+MN+BI+BO-TuM5 Tunable Microeddies for Microfluidics: Non-contact Single-cell Trapping using Gentle Fluid Flow**, *B.R. Lutz, J. Chen, D.T. Schwartz*, University of Washington, *D.R. Meldrum*, Arizona State University

Cells that normally live in suspension typically exhibit strong biological responses to physical contact. Microfluidic devices have been very successful for studying single adherent cells in controlled chemical environments, but tools for manipulating single cells in suspension are extremely limited. We developed a non-contact microfluidic single-cell trap that creates strong trapping forces using only gentle fluid flow. The traps are based on steady streaming flow, which is the steady flow generated when oscillating fluid interacts with any boundary that causes the fluid to turn (e.g., obstacles, cavities, bends). Steady streaming was first identified over a century ago, but its remarkable ability to trap cells was not known. A key feature of this approach is that traps are insensitive to differences in cell shape, cell density, and fluid medium. We demonstrate the ease of trapping for bubbles, spheres, rod-like debris, non-spherical motile phytoplankton, macrophages, and monocytes in different fluid media. The approach is remarkably simple to implement and control, in fact, early work used hand-built flow channels and a home stereo amplifier. The flow is created by audible-frequency fluid oscillation in a microchannel containing a cylindrical post. The back-and-forth motion creates four eddies around the cylinder, and each eddy traps a cell and holds it in place at a predictable location within the fluid. We use capture and release of swimming phytoplankton to estimate the trap strength; strong trapping forces capable of holding the strongest swimmers are easily generated (>30 piconewtons), while gentle shear conditions in the traps are comparable to arterial blood flow. By using flow to displace trapped spheres under different conditions, we determine a simple scaling relationship that quantitatively describes the trapping force for common cell sizes (5-50 microns). The traps withstand net flows as large as 1 cm/second, which enables medium exchange and chemical treatment of single cells in suspension. Posts can be arrayed with little effect on trapping behavior, providing the potential for high-throughput screening of suspension cells based on dynamic measurements. The combination of strong, tunable trapping forces and gentle trapping environment makes this an appealing new alternative for manipulating single cells in microfluidic devices.

9:40am **BM+MN+BI+BO-TuM6 Automated on-Chip Rapid Microscopy, Phenotyping, and Screening of C. elegans**, *H. Lu*, Georgia Institute of Technology

Microscopy, phenotyping, and visual screens are critical methods frequently applied to model organisms in combination with genetics. Although widely used, these techniques for multicellular organisms have mostly remained manual and low-throughput. We report the complete automation of sample handling, high-resolution microscopy, phenotyping, and screening of *C. elegans* using a custom-designed microfluidic system. The engineered system, coupled with customized software, enables high-throughput diffraction-limited imaging and sorting of samples with no human intervention with any microscopy setup. The robustness and automation of our system relies greatly on integrated closed-loop control software as well as engineered hardware design of the microchip. The chip has six salient features that ensure a consistent and reliable operation for an extended period of time. First, it automatically self-regulates the loading of nematodes by a simple passive loading-regulator design. Constant pressure drives the flow, so that no feedback or intervention is necessary for the microchip to allow one and only one animal to occupy the imaging area at a time. Second, the setup automatically positions the samples in an identical position in the chip, so as to minimize the travel of the motorized stage and thereby reduce the processing time and increase the throughput. Third, the device has an integrated local temperature control system whereby animals are cooled to ~4 °C and completely immobilized briefly (~ a few seconds) for imaging and manipulation without the use of anesthetic drugs. Cooling provides an alternative to anesthetics, potentially minimizing the adverse developmental effects. Fourth, the microchip and the setup are compatible with any standard microscopy setup with no modification necessary, including simple compound epifluorescence microscopy, as well as more expensive multiphoton or confocal microscopy. Fifth, the microchip has no permanent small features (<20 μm), and therefore is easy to fabricate, less likely to be clogged by debris, and can operate very robustly. Lastly, losses through our system are minimal (~3%) and the device design is gentle on the animals as the viability of all the sorted animals is ~100%. We show that compared to standard manual operation, time for phenotyping and visual screens can be reduced by ~2 orders of magnitude in our system with no human intervention, which has not been demonstrated before. Moreover, we show the ability to perform multiple sensitive and quantitative genetic

screens with real biological samples based on cellular and subcellular features with over 95% accuracy per round.

10:40am **BM+MN+BI+BO-TuM9 BioMEMS Challenges and Opportunities – A Department of Defense Perspective**, *D. Polla*, Defense Advanced Research Projects Agency, *S. Barker*, System Planning Corporation **INVITED**

Considerable progress has been made over the last 15 years in realizing a great variety of BioMEMS devices and systems. The field of BioMEMS can be approximately subdivided into three categories: (1) bioanalytical systems, (2), surgical systems, and (3) therapeutic systems. All three areas have numerous commercial and defense applications, but in many cases progress is inhibited by fundamental scientific and technological challenges. This paper presents the authors' perspective on the top 10 challenges facing BioMEMS today. Bioanalytical systems, which are also commonly referred to as "lab-on-a chip," have not realized their full potential for numerous reasons: (1) Autonomous sample processing with minimal human intervention has yet to be achieved. (2) Sample clean-up and pre-processing pose significant challenges that often limit the performance of a bioassay. (3) The ability to take a biological sample and obtain a result or set of results is still a long process, often taking several hours; obtaining a microfluidics-based PCR bioassay result in less than one minute persists as a grand challenge for the BioMEMS community. (4) The development of size-scaled microinstruments for bioanalysis presents an enormous opportunity toward the realization of remote site-derived information that can be conveniently communicated to a physician's office and correlated with a patient's stored medical record. The potential of surgical MEMS has often meant "micro-invasive" surgery that provides significant benefit to the patient. (5) But non-invasive surgery enabled by MEMS has the potential for providing even better patient outcomes. (6) Both sensors and actuators with the capability for more accurately and more reliably reproducing the skill of a surgeon's hands still need to be realized. (7) Developments that enable in vivo imaging of cells and organs using MEMS devices may also play an important role in enabling more effective precision surgeries. Therapeutic systems based on MEMS technology have yet to be made smart. (8) This means effectively integrating sensors, electronics, and actuators in a controlled feedback system designed to provide therapy only when the body needs it. These systems are often implantable and are limited by (9) battery size and lifetime. And finally, (10) neural prosthesis represents an exciting new domain where MEMS may provide an effective interface between nerves and electronics.

11:20am **BM+MN+BI+BO-TuM11 CD Based Sample Preparation and Pathogen Screening**, *M.J. Madou*, University of California, Irvine **INVITED**

We have demonstrated the feasibility of a multiplexed microfluidic CD apparatus for sample preparation of a wide variety of clinical samples and the subsequent detection of viruses, bacteria and fungi through fast DNA hybridization on the same platform. As the CD slowly rotates, a free moving magnetic disc in a lysis chamber is moved back and forth in the radial direction by the magnetic force of stationary magnets located below the rotating CD. The movement of the magnetic disk causes mechanical shear that disrupts cell membranes. This CD does not only process multiple samples simultaneously, but can also be used for the centrifugal precipitation of solids from each sample liquid. After precipitation, the resulting clarified liquid is transferred through a solid phase extraction membrane to capture the DNA. This step is followed by subsequent automated washing, elution, and detection by hybridization and fluorescence detection on an embedded DNA array. Recent sample to answer results and modeling of ice valves and coriolis valves will be detailed.

MEMS and NEMS

Room: 206 - Session MN-TuM

Materials Processing and Characterization for MEMS/NEMS

Moderator: B.R. Ilic, Cornell University

8:00am **MN-TuM1 Weighing of Biomolecules, Single Cells, and Single Nanoparticles in Fluid**, *S. Manalis*, Massachusetts Institute of Technology **INVITED**

Recent advances towards developing biomolecular and single cell applications for a mass-based biosensor known as the suspended microchannel resonator (SMR) will be presented. In SMR detection, target molecules or cells flow through a vibrating suspended microchannel and are captured by receptor molecules attached to the interior channel walls. What

separates the SMR from the existing resonant mass sensors is that the receptors, targets, and their aqueous environment are confined inside the resonator, while the resonator itself can oscillate at high Q in an external vacuum environment, thus yielding extraordinarily high sensitivity. This approach solves the problem of viscous damping that degrades the sensitivity of cantilever resonators in solution. We have achieved a resolution of approximately 1 femtogram (1 Hz bandwidth) which represents an improvement of six order of magnitude improvement over a high-end commercial quartz crystal microbalance. This gives access to intriguing applications such as mass based flow cytometry and real-time monitoring of single cell growth.

8:40am **MN-TuM3 Development of Superhydrophobic Biomimetic Surfaces with Hierarchical Roughness**, *B. Bhushan*, The Ohio State University, *K. Koch*, Nees Institute for Biodiversity of Plants, Germany, *Y.C. Jung*, The Ohio State University

Superhydrophobic plant surfaces, e.g. the Lotus leaves, and theoretical calculations show that a hierarchical surface roughness is beneficial for superhydrophobicity. Biomimetic hierarchical surface structures were produced by multiple replication of a microstructured silicon master surface. Replicas are made in a two step process, in which a two-component silicon molding mass was applied into the micropatterned Si surfaces, followed by a filling of the mold with an epoxy resin. On these different nanostructures have been applied by physical vapor deposition of hydrophobic n-hexatriacontane (C₃₆H₇₄), and octacosyl-1-ol. These organic molecules are able to self assemble on the substrates into three-dimensional crystals, and their shape, size and chemistry is comparable to those structures, found on water repellent surfaces. The surfaces created are fully characterized by SEM and AFM and attempted to separate out the effect of hierarchical structures on the hydrophobicity. We show how static contact angles, hysteresis and tilt angles vary with microstructure, nanostructure and hierarchical structure. We also study the effect of droplet size on contact angle by evaporation using droplets on the surfaces.

9:00am **MN-TuM4 Nanomechanical Resonators for Specific Detection of Proteins**, *C. Guthy*, *L.M. Fischer*, *V.A. Wright*, *A. Singh*, *J.M. Buriak*, *S. Evoy*, University of Alberta and National Institute for Nanotechnology, Canada

Rapid, sensitive and inexpensive analysis of biological molecules is vital to disease detection, monitoring, drug discovery. For instance, the detection and identification of biomarker proteins of diseases such as metabolic disorders, multiple sclerosis and cancer have gained considerable attention over the recent years. Detection of such proteins with high specificity at very low concentrations would greatly facilitate diagnostic and help predict disease progression. Current analytical technologies such as DNA microarray, mass spectrometry and nuclear magnetic resonance spectroscopy are expensive and technically challenging for clinical applications. Mechanical resonators have been demonstrated as highly sensitive transducers for the detection of molecular systems. The sensitivity of resonators scales favorably as their dimensions are reduced, offering a compelling path for the development of sensors with exceptional mass sensitivities. To enable the specificity of detection, various analyte-specific functional layers need to be immobilized onto the surface of resonators. Such resonators could be then used as sensing arrays for the analysis of complex protein mixtures. As a proof of concept, we recently demonstrated the specific detection of streptavidin using doubly-clamped nanomechanical resonators (bridges) functionalized with biotin. Bridges with widths down to 300 nm were realized from silicon carbonitride (SiCN) thin films using a novel fabrication method. Based on the shift of resonant frequency, a mass of 3.6 fg/ μm^2 was attributed to the added streptavidin, corresponding to one molecule per 27 nm². We have since further scaled down the dimensions of our devices and have demonstrated the surface machining of resonators of world record lateral dimensions of 40 nm with a yield approaching 100%. These 15 μm long resonators show resonance frequencies of ~ 11 MHz with quality factors of ~ 5000 in the mTorr pressure range. We will present a thorough investigation of the resonant behavior of these novel sub-100nm NEMS devices of various dimensions. We are also developing the attachment of antibodies onto these resonators. The specific detection of human interferon gamma (IFN- γ) protein was chosen as target system. Our ultimate goal is to use similar immobilization procedures for the detection of disease biomarkers, including but not limited to multiple sclerosis biomarkers.

9:20am **MN-TuM5 Experimental Determination of the Dynamic Spring Constants of Higher Flexural Modes of Microcantilevers**, *G. Hähner*, *G.V. Lubarsky*, University of St Andrews, UK

Cantilever based technologies have seen an ever increasing level of interest since the atomic force microscope (AFM) was introduced two decades ago. Most recent developments employ microcantilevers as stand-alone sensors by exploiting the dependence of their oscillating properties on external

parameters such as adsorbed mass, or the density and the viscosity of a liquid environment. In this context higher flexural modes have attracted significant attention due to their high sensitivity towards forces and mass changes. While some effort has been devoted to the determination of the static and the first dynamic spring constant, there are currently no equivalent simple yet reliable methods to determine the values of higher modes experimentally. We demonstrate how the spring constants of higher flexural modes of microcantilevers can be determined experimentally with high precision. We recently presented a fast and simple method to measure the dynamic spring constant of the first mode in a non-destructive and non-invasive fashion.¹ The method is based on comparing the spring constants of the cantilever to a known spring constant by measuring the change in the resonance frequency of the flexural modes as a function of the fluid flow through a microchannel. Here we will show that the same approach can also be applied to higher flexural modes. Results for both rectangular and V-shaped cantilevers will be presented and compared to theory.

¹ G.V. Lubarsky and G. Hähner Rev. Sci. Instr. 78, 095102 (2007).

9:40am MN-TuM6 Heterodyne Optical Detection of Mechanical Resonances of Ultra High Frequency Silicon Micro-Resonators, L.J. Klein, T. Barwicz, S. Guha, H.F. Hamann, IBM TJ Watson Research Center

Recent advancement in microscale electromechanical systems fabrication and operation demonstrated ultra high mass and force sensitivity. Here we investigate the parameters affecting the ultimate sensitivity of suspended mechanical resonators, in particular the resonance frequency and quality factor scaling as the resonators dimensions are reduced. The silicon resonators are fabricated by e-beam lithography with length ranging from 20 μm down to 500 nm and having a width of 500 nm. The suspended resonators are actuated using electrostatic force acting between the suspended beam and side gates and an optical heterodyne interferometry is used to detect the resonant oscillations. The heterodyne interferometry has a large bandwidth allowing resonance frequency detection above GHz and has a detection sensitivity of 10 pm. For fundamental oscillation modes up to 220 MHz, the quality factor of resonance were over 20000 in high vacuum at room temperature and increased in the explored range. We present experimental approaches to detect both flexural and transversal oscillation modes for silicon micro-resonators using our optical detection scheme.

10:40am MN-TuM9 The Effect of Substrate Material and Metallization Layers on the Mechanical Properties of Micromachined Amorphous Silicon Carbide Structures, R.J. Parro, Case Western Reserve University, M.C. Scardelletti, N.C. Varaljay, NASA Glenn Research Center, C.A. Zorman, Case Western Reserve University

Amorphous SiC (a-SiC) films are attractive for micromachined structures requiring the properties of SiC but whose substrates cannot tolerate the high deposition temperatures associated with the conventional CVD methods used to deposit the crystalline forms. The preponderance of data in the literature focuses on the properties of a-SiC films deposited directly onto Si; much less is known about the properties of these films when deposited onto silicon dioxide sacrificial layers. Even less is known about how metallization (required for electrostatic actuation) affects the mechanical behavior of a-SiC micromachined structures. This study examines the roles that the substrate and metallization layers play on the mechanical properties of a-SiC structures. Test specimens were fabricated from 300 nm-thick a-SiC films deposited by PECVD on bare (100) Si wafers and (100) Si wafers coated with a 3.2 μm -thick PECVD silicon dioxide film. After deposition, the wafers were annealed at 450C for 30 min to convert the as-deposited compressive film stress to a low tensile stress. Suspended membranes with areas of 750x750 μm^2 were bulk micromachined by anisotropic etching in KOH at 50C. For the oxide-coated wafers, a 35 min immersion in BOE at 25C was performed to remove the silicon dioxide beneath the SiC membranes. The membranes were subjected to load-deflection testing at differential air pressures between 0 and 138 kPa. From the resulting pressure versus deflection data, it was found that the average Young's modulus for a-SiC films deposited on Si was 129 GPa with a residual stress of 162 MPa, while films deposited on silicon dioxide had a Young's modulus of 116 GPa with a residual stress of 154 MPa. For films deposited on oxide-coated wafers, the effect of metallization on the residual stress of the membranes was characterized. Membranes were first subjected to load-deflection testing, then coated with a Cr adhesive layer and Au structural layer by e-beam evaporation and again subjected to load-deflection testing. For a total metal thickness of 60 nm (10 nm/50 nm Cr/Au), the average residual stress increased by 52 MPa. For a total metal thickness of 125 nm (25 nm/100 nm Cr/Au), the average residual stress increased by 43 MPa, and for 250 nm (50 nm/200 nm Cr/Au) the average residual stress increased by 35 MPa.

11:00am MN-TuM10 Effects of Actuation Voltage and Relative Humidity on Polycrystalline Silicon Corrosion, F. Liu, C.S. Roper, C. Carraro, R. Maboudian, University of California at Berkeley

The effects of relative humidity and actuation voltage in MEMS have been investigated using polysilicon electrodes. The results indicate occurrence of anodic oxidation under positive bias and absence of cathodic protection under negative bias that leads to the precipitation of dissolved species. We will also report on the effect of electrode geometry and surface termination. Our results show that corrosion can be the dominant failure mechanism of polysilicon MEMS when driven by large electrostatic voltages in humid environments.

11:20am MN-TuM11 Gold-Tantalum Nanocomposites as Structural Material for Nanomechanical Sensors, N. Nelson-Fitzpatrick, C. Ophus, E. Luber, University of Alberta, Canada, Z. Lee, V. Radmilovic, Lawrence Berkeley National Laboratory, D. Mitlin, S. Evoy, University of Alberta, Canada

Micro and nanomechanical cantilevers and resonators have received significant attention as a technological solution for ultra-sensitive mass detection.¹ One promising approach to enable specific nanoresonator-based biosensing would be to coat the devices with a thin film of gold in order to exploit thiol-based chemistries for the functionalization of these surfaces. However, such metal coatings would significantly lower its resonance quality, impairing its mass sensitivity.² Alternatively, we are currently developing of a novel Au/Ta alloy that would allow the machining of micro and nanomechanical devices directly out of the metal.³ Metals have been largely overlooked as a structural material for NEMS since most deposition methods tend to yield films with large grain structures complicating reliable machining at the nanometer scale, and containing differential stresses which result in the deformation of released devices. To remedy these problems, we must be able to deposit a metal that is either amorphous or has extremely small grain sizes in order to fabricate a device with nm size critical dimensions. Previously we reported on the deposition of Au/Ta nanocomposites for the purpose of making NEMS resonators.³ We reliably realized 50nm thick Au/Ta nanoresonators with low stress (~20MPa), reduced grain size and RMS roughness. The devices also retained the gold's <111> texture important for the formation of thiolized SAMs. We have now moved on to the development of micro-cantilevers operating in the static regime with this material. The fine grain size of this alloy enables the realization of ultra-thin, ultra-compliant, released cantilevers directly out of Au/Ta composite. The distinctive grain structure of this material, as well as the inclusion of tantalum impacts the dynamics of molecular attachment, which will affect the response of static cantilevers to the target analyte. To that end, we intend to present a full study on the impact of the alloy nanostructure and its composition on the surface stresses induced by the chemical attachment of a thiolized SAM.

¹ Ilic B, Czaplewski D, Craighead H G, Neuzil P, Campagnolo C and Batt C, 2000 Appl. Phys. Lett. 77 450

² Sekaric L, Carr D W, Evoy S, Parpia J M and Craighead H G, 2002 Sens. Act. A 101 215

³ Nelson-Fitzpatrick N, Ophus C, Luber E, Gervais L, Lee Z, Radmilovic V, Mitlin D, and Evoy S, 2007 Nanotechnology 18.

11:40am MN-TuM12 Theoretical and Experimental Investigation of Electrostatically Actuated Bistable Micro and Nano Structures, S. Krylov, Tel Aviv University, Israel, B.R. Ilic, Cornell University, D. Schreiber, Tel Aviv University, Israel, S. Seretensky, Smart Team inc., H.G. Craighead, Cornell University

In this work, theoretical and experimental study of initially curved electrostatically actuated micro and nanobeams with clamped ends was performed. Due to unique combination of non-monotonous stiffness-deflection dependence typical for mechanically bistable structures and of nonlinear electrostatic force abundant in micro and nano systems the structure exhibits sequential snap-through buckling and electrostatic pull-in instability as well as multiple stable configurations at the same voltage (bistability). Reduced order Galerkin and consistently constructed lumped models were built and verified by numerical analysis and experimentally. The minimal initial elevation required for appearance of the bistability in the electrostatically actuated beam is smaller than in the case of an uniform deflection independent loading; closed form approximation of this elevation was evaluated. The devices were fabricated from silicon on insulator (SOI) wafer using deep reactive ion etching and in-plane responses were characterized by means of optical and Scanning Electron microscopy (SEM). In addition, out-of-plane moving nano scale devices made of a intrinsically pre-stressed polysilicon were fabricated and characterized. Characterization inside a SEM was found to be a useful experimental approach providing stable operational in-vacuo conditions while higher magnification improves the quality of data processing. Model results obtained for the actual dimensions of the device were in good agreement with the experimental data. Designs incorporating bistable beams have clear functional advantages and may result in improved performance of switches,

capacitive based sensors and MEMS/NEMS based nonvolatile memory devices.

Tuesday Afternoon Poster Sessions

MEMS and NEMS

Room: Hall D - Session MN-TuP

MEMS and NEMS Poster Session

MN-TuP1 Effects of Gate Fabrication Processes on Electrical Characteristics in Suspended Gate FET, T. Kumada, H. Kasai, M. Edo, Y. Ichikawa, Fuji Electric Advanced Technology Co., Japan

Suspended gate FET's (SG-FET) are expected to have a number of applications such as switching devices and acceleration sensors. A key process in these devices is how to fabricate the suspended gate. We have studied influence of the gate materials, the gate structure and sacrifice layer etching on the electrical characteristics of the SG-FET. The SG-FET we have developed has a conventional planar type MOS FET except for the gate placed apart from the silicon oxide gate insulator; the gate has a bridge structure with supports put outside the gate insulator. The gap between the gate and the gate insulator was designed to be 0.5 - 1.0 μ m. In these devices, the stress of the gate materials deforms the gate and changes the gap from the designed value; it affects the electrical characteristics of the SG-FET. Thus we have studied the gate materials and the gate structure to reduce deformation of the gate. Another important key in the SG-FET is the fabrication process of the suspended gate. To make its bridge structure, we deposit a sacrifice layer on the gate insulator first, and then deposit the gate material on it. After patterning it to form the gate, we remove the sacrifice layer by etching. This etching process affects the electrical characteristics of the SG-FET because of contamination and damage of the surface of the gate insulator. We applied polyimide to the sacrifice layer, and studied the influence of the etching method and the etching condition on the characteristics. In this presentation, we will discuss the relationship between the fabrication process and the materials of the suspended gate and the electrical characteristics in detail.

MN-TuP2 Q-Factors of Suspended Al and Al-CNT Thin-Film Micromechanical Resonators as Function of Tensile Stress, Y.J. Yi, J.H. Bak, Y.D. Kim, B.Y. Lee, S. Hong, Y.D. Park, Seoul National University, Republic of Korea

We report on the Quality (Q) factor of micromechanical resonators, realized from suspended Al and Al-CNT nanolaminate thin-films,¹ as function of tensile stress. The doubly-clamped beam micromechanical resonators with varying dimensions are realized on GaAs substrate, which is selectively removed. Suspension of microresonators is preceded by UHV sputter deposition of Al and carbon nanotube (CNT) network assembly and patterning by standard e-beam and photolithography techniques. The frequency response of micromechanical resonators are characterized by a laser vibrometer-like set-up while the microresonators are actuated electrostatically. With significant difference in coefficients of thermal expansion, the suspended metallic thin-films have significant intrinsic internal stress as evident from fits to resonant frequency as function of beam dimensions measurements. The internal stress is further varied by post-fabrication thermal treatments as well as by chip-bending method.² Increase in Q-factors ultimately improves the force sensitivity of micromechanical resonator devices (i.e., mass sensing applications). Comparing the dependence of Q-factor to tensile stress of Al and Al-CNT thin-film microresonators allows for another avenue to study the mechanical enhancements due to CNT networks in the Al-CNT lamina.

¹ J.H. Bak, Y.D.Kim et al., Nature Materials advance online publication, 20 April 2008 (doi:10.1038/nmat2181).

² S.S. Verbridge et al., Nano Letters 7, 1728 (2007).

MN-TuP3 Test Instrument for the Tensile Fracture Strength of Micro-Nano Materials, A. Kasahara, H. Suzuki, M. Goto, H. Araki, Y. Pithosh, M. Tosa, NRIM, Japan

Recent nano-technology researches have created various advanced micro-nano materials. In particular, there have been many reports on nano-meter-scale tubes and wires such as carbon nanotubes and silicon wires. We have prepared long crystal silicon wires with a diameter of several tens of nano meters at a temperature lower than 523k by using the low-pressure low-temperature CVD method. To use these as materials for application to micro-nano electromechanical system, we need to fully understand their electric, chemical and mechanical properties. As for the electric properties, current-voltage characteristics have been measured with the multi probe system using STM measurement units, although but there is almost few technique established for the measurement of mechanical properties. We have been therefore developing a strength measurement equipment for micro-nano materials to determine the mechanical properties of micro-nano wires in a vacuum for SEM observation. We will report this result on the

development of test instrument to measure the tensile strength that can be observed with a desk-top simple SEM.

MN-TuP4 Functionalization of Micro Mechanical Cantilever Sensors with TiO₂ and γ -Fe₂O₃ Nanocrystals, C. Ingrosso, M. Striccoli, CNR-IPCF Sez. Bari c/o Dip. di Chimica, Italy, E. Sardella, IMIP-Bari, Italy, A. Voigt, G. Gruetzner, Micro Resist Technology GmbH, Germany, A. Agostiano, CNR-IPCF Sez. Bari c/o Dip. di Chimica, Italy, S. Keller, G. Blagoi, A. Boisen, Technical University of Denmark, M.L. Curri, CNR-IPCF Sez. Bari c/o Dip. di Chimica, Italy

The outstanding flexibility of the surface chemistry of colloidal nanometer-sized particles¹ allows their reliable manipulation as building blocks and opens the access to the development of original supramolecular approaches, devoted to the mesoscale organization of nanoparticles in hierarchical structures. In this work, 3D arrays of organic-capped colloidal NCs were covalently immobilized on the surface of micro mechanical cantilever sensors made of a negative tone epoxy photoresist matrix. Mechanical cantilever sensors surface functionalized with a sensing active layer are very active in the detection of target molecules.² Q-sized colloidal NCs were immobilized at the surface of the miniaturized responsive components by means of the surface reactive residual epoxy at crosslinked photoresist matrix, which provide good accessibility to external interacting molecules, with an high immobilization capacity. The transferring of Q-sized colloidal NC properties to mechanically responsive miniaturized components represents an intriguing challenge, which disclose a great potential for extending the field of application of the ultrasensitive microfabricated mechanical cantilever sensors by exploiting novel transduction processes in molecule detection. Here, a simple solution-based method was adopted to modify the epoxy photoresist made cantilever surface with oleic acid (OLEA)-capped anatase TiO₂ nanorod (NR) and nearly spherical maghemite γ -Fe₂O₃ NC building blocks. The morphological evolution of the native epoxy surface upon the attachment of NCs was monitored by Atomic Force Microscopy (AFM), while the effective covalent anchoring of the nanoparticles was demonstrated by means of X-Ray Photoelectron Spectroscopy (XPS). The photo/catalytic, magnetic and optical functionalities of NCs³ transferred to the high dense NC layout open the access to the development of novel MEMS/BIO-MEMS devices based on new bio/molecular recognition processes for bio/sensing or environmental purposes. Acknowledgements: The work was partially supported by the 7th FP EU project NOVOPLY (STRP 013619)

¹ Yin, Y. et al. Nature 437, (2005), 664.

² Gfeller, K. Y.; Nugaeva, N.; Hegner, M. Biosens. Bioelectron. 21, (2005), 528.

³(a) Narazaki, A.; Kawaguchi, Y.; Niino, H.; Shojiya, M.; Koyo, H.; Tsunetomo, K. Chem. Mater. 17, (2005), 6651. (b) Detlef, M. S.; Thomas, S. R. J. Magn. Magn. Mater. 302, (2006), 267.

Wednesday Morning, October 22, 2008

Tribology Focus Topic

Room: 205 - Session TR+MN+NC-WeM

Surfaces and Interfaces in MEMS/NEMS

Moderator: J.A. Harrison, United States Naval Academy

8:00am **TR+MN+NC-WeM1 A Study of Au and Ru RF MEMS Contacts in Controlled Vacuum Environments, M. Walker,** North Carolina State University, *N. McGruer*, Northeastern University, *J. Krim*, North Carolina State University

Studies of RF MEMS switch performance under ultra clean and controlled environmental conditions have to date been extremely limited.¹ Such studies are highly valuable however, as they provide an opportunity to separately examine the impact of various factors such as contamination films, creep, deformation and stiction that plague current RF MEMS switch reliability. We have constructed a custom ultra high vacuum chamber with in situ surface cleaning, variable temperature and gas dosing capabilities in an effort to isolate the multiple variables that impact RF MEMS contact resistance and longevity. This chamber allows us to study switches in atmosphere followed by studies performed in the cleanest environment possible. We have investigated cantilever MEMS devices with both Au on Au and Ru on Ru contacts. Au on Au switches have so far been investigated by the vast majority of studies, and provide baseline material for our studies. Ru on Ru switches are far less studied. Ru is currently our material of interest on account of its harder properties that resists creep and deformation. In atmosphere we observe the resistance of a closed switch over time then open the switch. We pump the chamber to a base pressure of 9×10^{-10} torr followed by measuring the resistance over time then opening the switch. Surface cleaning is performed via in situ oxygen plasma. This is followed by closing the switch and observing the resistance over time. We have observed switches that have initially had infinite resistance in both atmosphere and UHV exhibit tens of ohms contact resistance after oxygen plasma cleaning. The resistance values after oxygen cleaning are closer to the theoretical values for clean contact. We compare these resistance changes to theoretical models² that account for creep and deformation of the switch contacts. A study of contact resistance as a function of hydrocarbon uptake is in progress.³ This work was supported by the DARPA Center for RF MEMS Reliability and Design Fundamentals Grant # HR0011-06-1-0051 and the AFOSR Extreme Friction MURI Grant #FA9550-04-0381.

¹ C. Brown, A. Morris, A. Kingon, J. Krim, submitted to J. MEMS

² O. Rezvaniyan, C. Brown, M. Zikry, A. Kingon, J. Krim, D. Irving, D. Brenner, submitted to J. Applied Physics

³ H. Koidl, W. Rieder, Q. Salzman, vol. 22, No. 3, 1999.

8:20am **TR+MN+NC-WeM2 Bimetallic Nanoparticles as Surface Coatings in MEMS Switch Contacts, M.L. Jespersen,** Air Force Research Laboratories, *S.T. Patton*, University of Dayton Research Institute, *J. Slocik*, *R. Naik*, *A. Campbell*, *A.A. Voevodin*, Air Force Research Laboratories

Microelectromechanical systems (MEMS) switches have a broad range of applications in the aerospace, communications, and electronics industries. However, contact failure, especially during hot switching, prevents widespread implementation of the next generation MEMS devices in new technologies. Few studies have investigated physical and chemical processes that occur on modified MEMS contact interfaces, although one published study used self-assembled monolayers (SAMs) as a switch lubricant.¹ These SAMs thermally decompose in the contact.¹ We also have investigated nanoparticle liquids (NPLs) deposited onto MEMS contacts as nanomaterial-based lubricants, which improved the performance and durability of MEMS contact switches by orders of magnitude.² In this study, we investigated bimetallic (Au/Pd) nanoparticles (NPs) as surface lubricants for MEMS contact switches. Bimetallic systems offer enhanced properties for MEMS by taking advantage of the physical characteristics of the individual components. For example, Au has a low contact resistance, while Pd exhibits higher melting temperatures and lower adhesion. Performance of bimetallic NP-lubricated contact surfaces were investigated, using a micro/nano-adhesion apparatus as a MEMS switch simulator with in-situ monitoring of contact resistance and adhesion force. Ex-situ analyses of the chemical and physical processes at the contact interfaces were carried out using SEM, TEM, XPS, and scanning Auger spectroscopy. Bimetallic NPs exhibited orders of magnitude improvement in electrical performance and durability as compared to uncoated and SAM-coated contacts. The observed improvement in performance and reliability results from nanoscale surface roughness extending across multiple nanocontact regions, enhanced thermal and electrical conductivity over SAM coatings, and self-limited nanowire growth that prevents shorting failure in the contact regions, as determined

from physical and chemical analyses. Based on these results, bimetallic nanoparticles are promising candidates as surface lubricants for MEMS switch contacts.

¹S. T. Patton, K. C. Eapen, J. S. Zabinski, J. H. Sanders, and A. A. Voevodin, "Lubrication of MEMS RF switch contacts using self-assembled monolayers," J. Appl. Phys., vol. 102, pp. 024903-1 – 024903-5, 2007.

²A. A. Voevodin, et al. "Nanoparticle-Wetted Surfaces for Relays and Energy Transmission Contacts." Small, vol. 3, pp. 1957-1963, 2007.

8:40am **TR+MN+NC-WeM3 Contact Mechanics and Lubrication of MEMS Switches: Insights from Atomic and Multiscale Modeling, D.W. Brenner,** North Carolina State University **INVITED**

We have been using a combination of molecular modeling and continuum analysis to understand and predict a range of dynamic processes that occur during the contact of RF and capacitive MEMS switches. The results of these studies are being used in the rational design of new materials and lubrication strategies for enhancing the lifetimes of these devices. In the case of closed RF-MEMS switches, it will be shown that the time-dependent resistance is well described by a power law, and using an asperity creep model that the prefactor and exponent in the power law can be related to the surface roughness and creep coefficient, respectively. For capacitive switches we have used molecular modeling to explore the efficacy of a "bound+mobile" lubrication scheme involving tricresylphosphate molecules diffusing on an octadecylchlorine self-assembled monolayer. Temperature-dependent diffusion coefficients calculated from the simulations have been used in a scaling relation for liquid lubrication that depends on the ratio of the contact area to the product of the lubricant diffusion coefficient and the switch cycle time. This combination of atomic modeling and multiscale analysis predicts that this molecule-surface combination will only be effective for temperatures greater than $\sim 200\text{K}$ and up to $\sim \text{MHz}$ oscillation frequencies.

This work was done in collaboration with D. Irving, O. Rezvaniyan, C. Brown, M. Zikry, A. Kingon, C. Padgett and J. Krim. This work was supported by the Extreme Friction MURI program, AFOSR grant FA9550-04-1-0381 and the Office of Naval Research.

9:20am **TR+MN+NC-WeM5 Sidewall Tribometer Study of Vapor Phase Lubricants for MEMS, D.A. Hook, B. Vlastakis, B.P. Miller,** North Carolina State University, *J. Rutledge*, University of California, Irvine, *M.T. Dugger*, Sandia National Laboratories, *J. Krim*, North Carolina State University

Long hydrocarbon and fluorocarbon based monolayers have been widely used in MEMS applications to prevent release related stiction and adhesion. These and similar monolayers, however, have proven ineffective as MEMS lubricants. Indeed, even the most robust of SAM layers fails to protect devices from tribological failure for either normal or sliding cyclic contact.¹ Alternate schemes, such as vapor phase lubrication, must therefore be developed if progress is to occur.² The vapor phase of pentanol has recently been reported by Seong et al to extend the lifetime of a MEMS device in a mixture of dry nitrogen and various concentrations of pentanol. Macroscale friction experiments have meanwhile shown the build up of long carbon chain reaction films in identical conditions.³ In order to probe the effectiveness of pentanol and related alcohols, we have employed a ringdown measurement technique with a specially designed MEMS sidewall tribometer to compare the coefficients of friction of a device before and after introduction of pentanol into a vacuum chamber at one monolayer of coverage. Initial measurements show no change in the coefficient of friction, but subsequent measurements show a progressive decrease. This reflects a formation of a reaction film extremely quickly upon rubbing. Lifetime measurements using the sidewall tribometer were also taken with shorter chain alcohols, namely trifluoroethanol and ethanol, at one monolayer coverage to determine whether amount of carbon present affects the lubricating properties as well as the role of methyl versus trifluoromethyl termination. It has been found that ethanol's ability to lubricate is dependant upon the initial state of the device whereas trifluoroethanol and pentanol will lubricate a device that has previously failed. Work funded by the AFOSR Extreme Friction MURI #FA9550-04-0381.

¹Hook, D.A., Timpe, S.J., Dugger, M.T., Krim, J., "Tribological Degradation of Fluorocarbon Coated Silicon Microdevice Surfaces in Normal and Sliding Contact" Journal of Applied Physics, in press

²Krim, J., Abdelmaksoud, M., "Nanotribology of Vapor-Phase Lubricants" Tribology Issues and Opportunities in MEMS, B. Bhushan, ed. 1998 pp. 273-284

³Asay, D.B., Dugger, M.T., Ohlhausen, J.A., Kim, S.H., "Macro- to Nanoscale Wear Prevention via Molecular Adsorption", Langmuir 2008, 24, 155-159.

9:40am **TR+MN+NC-WeM6 Effects of Organic Vapor Adsorption on Nanoasperity Adhesion and Friction – From Fundamentals to MEMS Applications, S.H. Kim,** Pennsylvania State University

As the contact size involved in mechanical device operations decreases, the adsorption of gaseous molecules on the contact surface – which normally

ignored in macroscopic measurements – becomes more important and dominant factors governing the contact properties such as adhesion and friction. Water adsorption can cause high adhesion and severe wear of silicon oxide surfaces. In contrast, alcohol vapor adsorption from the ambient can provide unprecedentedly efficient lubrication effects for operation of microelectromechanical systems (MEMS) with sliding contacts. Atomic force microscopy (AFM) is an ideal tool for studying the adhesion and frictional behavior of nanoscale asperity contacts. The tribological response of a silicon nanoasperity contact was studied with AFM with alcohol vapors as the VPL. Alcohol vapor adsorption on silicon oxide surface readily forms a thin organic film on the surface which mitigates the adhesion and friction forces between the AFM tip and substrate surfaces. The origin of adhesion and friction changes in the presence of alcohol vapor is elucidated through vibrational spectroscopic investigation of the thickness and structure of the adsorbed layers as well as theoretical calculations of their tribological responses.

10:40am **TR+MN+NC-WeM9 Dynamics and Spreading of Pentanol and Other Alcohols for MEMS Applications**, *B.P. Miller, J. Krim*, North Carolina State University

Microelectromechanical Systems (MEMS) have the potential to revolutionize widespread technologies, but friction and other tribological issues are currently preventing commercialization of devices that contain surfaces in sliding contact. Self-assembled monolayers (SAMs), while highly effective against release related stiction, have proven ineffective as MEMS lubricants. Indeed, even the most robust of SAMs fail to protect devices from tribological failure for either normal or sliding cyclic contact.¹ Alternative MEMS lubrication schemes must therefore be developed if progress is to occur. Vapor phase lubrication has been proposed as a solution to the issue of tribological device failure in Micro-Electro-Mechanical Systems (MEMS) with TCP and alcohol vapors attracting much interest as candidate materials.² In an effort to understand the basic mechanisms of lubrication we have performed a quartz crystal microbalance (QCM) study of the uptake, sliding friction, and spreading rates of adsorbed ethanol, trifluoroethanol (TFE) and pentanol films on silicon, aluminum and perfluorodecyltrichlorosilane (PFTS) treated substrates.³ In response to the oscillatory motion of the QCM, pentanol, and also ethanol, exhibit viscoelasticity and/or interfacial slippage when adsorbed on silicon or PFTS, implying that enhanced tribological performance may be expected in MEMS devices. TFE exhibited slippage on silicon but not PFTS. Significantly lower mobility levels were observed for all three alcohols adsorbed on aluminum. This work is funded by AFOSR Extreme Friction MURI Grant #FA9550-04-1-0381.

¹D. A. Hook, M. T. Dugger, and J. Krim. *J. Applied Physics*, in press.

²J. Krim and M. Abdelmaksoud, in *Tribology Issues and Opportunities in MEMS*, B. Bhushan, ed. (Kluwer Academic, Dordrecht, 1998), pp. 273-284; W. Neeyakorn et al., *Trib Lett.* 27 (2007) 269-276; D. B. Asay, M. T. Dugger, S. H. Kim. *Trib Lett.* 29 (2008) 67-74.

³B.P. Miller and J. Krim, Submitted to *Langmuir*.

11:00am **TR+MN+NC-WeM10 Effect of Fluid Flow on the Sensitivity of Microcantilever Sensors**, *R. Desikan, D. RangaPrasad, A. Passian, R.H. Datar, T.G. Thundat*, Oak Ridge National Laboratory

Microcantilever arrays are emerging as an attractive platform for detection of biomolecules because of their high sensitivity, miniature size, and their ability to work under solution. Selectivity in detection is accomplished by immobilizing receptor molecules on one surface of the cantilever. Interaction of biomolecules with the immobilized receptors results in cantilever bending. In general, the cantilevers are operated under constant flow of the buffer solution. Since cantilevers are sensitive to fluid flow, the flow rate is kept constant during injection of analytes in the flowing buffer solution. In some cases, reference cantilevers are used to eliminate the effect of fluid flow rate. However, we have observed that the interaction of analytes on receptors on cantilever surface is affected by the variations in the flow rate. More analyte molecules tend to bind the receptors on cantilever surface in static condition when molecular interaction is influenced by diffusion, compared to dynamic condition where analyte molecules flow across the cantilever using a flow control system. This work addresses the issues associated with biomolecular adsorption kinetics, flow rate dependence, and cantilever geometry for increasing the sensor sensitivity.

11:20am **TR+MN+NC-WeM11 Self-Affine Fractal Analysis of MEMS Surfaces for Minimizing Adhesion**, *D.-L. Liu*, Worcester Polytechnic Institute, *J. Martin*, Analog Devices Incorporated, *N.A. Burnham*, Worcester Polytechnic Institute

Differing approaches to studies of the influence of surface roughness on adhesion have recently appeared in the literature. Molecular dynamics has been used to simulate the contact of two surfaces and found that atomic-scale roughness can have a large influence on adhesion, causing the breakdown of continuum mechanics models.¹ Yet a simple continuum

model predicted the qualitative behavior of adhesion as a function of root-mean-square surface roughness in the nanometer to tens-of-nanometers range.² Although a useful first-order approximation, the assumptions in the latter work were severe; a more descriptive approach is necessary in order to design surfaces that either maximize or minimize adhesion. Self-affine fractal analysis provides a reasonable framework in which to move forward. In addition to the root-mean-square (RMS) roughness, it characterizes surfaces with two more parameters, the roughness exponent and the correlation length. A high roughness exponent and a small correlation length should minimize adhesion for two rough surfaces, as predicted by Chow.³ Our adaptation of his work shows similar results for the case of a smooth tip of an atomic force microscope (AFM) and a rough surface. Specifically, the surfaces had the same RMS roughness, 0.2 μm , and the same lateral correlation length, 3.0 μm , but their roughness exponents ranged from 0.1 to 1.0. The height-height correlation functions and the height distribution functions were calculated from the surface height data, and the three fractal parameters were extracted for all the surfaces. The adhesion between a smooth AFM tip and the fractal rough surfaces were then calculated based on both the height distribution and the force-distance relationship between one molecule in the AFM tip and the fractal rough surface. The adhesion was found to decrease linearly as the roughness exponent increased. Furthermore, experimental data of the adhesion between AFM tips and MEMS surfaces as a function of the three fractal parameters will be shown and compared with the theoretical predictions. The work presented here should help minimize adhesion in future MEMS devices and progress the understanding of adhesion between the atomic- and macro-scale.

¹B. Luan and M.O. Robbins, *Nature* 435, 929-932 (2005).

²D.-L. Liu, J. Martin, and N.A. Burnham, *Appl. Phys. Lett.* 91, 043107 (2007).

³T.S. Chow, *Phys. Rev. Lett.* 79, 1086 (1997).

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Voevodin, A.A.: TR+MN+NC-WeM2, 11
Voigt, A.: MN-TuP4, 10

— W —

Walker, M.: TR+MN+NC-WeM1, 11
Wei, Z.: MN-MoM3, 1
Wright, V.A.: MN-TuM4, 7

— Y —

Yan, X.-M.: MN+NC-MoA10, 4
Ye, L.: BM+MN+BI+BO-TuM1, 6
Yi, Y.J.: MN-TuP2, 10
Yilmaz, E.: MN-MoM4, 1
Yoon, Y.K.: MN+NC-MoA8, 4
Yu, L.: MN-MoM11, 2
Yuan, H.: MN-MoM7, 1
Yue, C.: BM+MN+BI+BO-TuM3, 6

— Z —

Zalalutdinov, M.K.: MN-MoM3, 1; MN-MoM4, 1
Zhang, Z.: BM+MN+BI+BO-TuM1, 6
Zhong, J.: MN-MoM8, 2
Zhong, X.: MN-MoM7, 1
Zorman, C.A.: MN-TuM9, 8