MEMS and NEMS

Room: 615 - Session MN-MoM

Materials Processing, Characterization and Fabrication Aspects

Moderator: A.V. Sumant, Argonne National Laboratory

8:00am **MN-MoM1 Materials for the Realization of High Performance Radio Frequency MEMS Devices**, ***S.P. Pacheco***, Freescale Semiconductor, Inc., *G. Piazza*, University of Pennsylvania **INVITED**

RF MEMS technology has rapidly evolved and matured over the last decade. More than 60 companies are currently involved in RF MEMS development with around 25% shipping commercial products or samples to customers. According to industry projections, by 2009, the RF MEMS market will break the $1 billion barrier with about 40% of the total market dominated by Bulk Acoustic Wave (BAW) devices.[1] Major opportunities for other RF MEMS devices will continue to expand as the rest of the market hits its stride in terms of both high-volume and high-end applications as issues with reliability, packaging, and CMOS integration are solved. The remaining market will be split between micro-mechanical resonators and oscillators for consumer and IT applications and RF MEMS switches for military applications and RF Test and Automated Test Equipment (ATE). This paper will describe proven material systems that are being presently commercialized as well as examine innovative materials that are starting to gain popularity for RF MEMS micro-resonators and switches. Benefits and challenges associated with each of these material systems will be presented. Topics such as CMOS/MEMS monolithic integration as well as the use of high acoustic velocity materials such as silicon carbide and diamond-like films for the realization of high performance, compact frequency references will be discussed. Additionally, the introduction to CMOS-compatible, low-loss GHz-range bandpass filters based on piezoelectric aluminum nitride contour-mode MEMS resonators will be covered. Piezoelectricity is also being investigated as an actuation mechanism for RF MEMS switches that would allow handset front-end compatible bias voltages in the 2-4 V range. Lastly, packaging breakthroughs using wafer-level techniques, including 3-D integration and surface micromachining, have the potential to enable low-cost, high-reliability, high-performance RF MEMS devices.

[1] J. Bouchaud, B. Knoblich, RF MEMS Market and Industry Overview, WTC - Wicht Technologie Consulting, Munich, http://www.wtc-consult.de.

8:40am **MN-MoM3 Nano-Scale, Directional Contact Metal Transfer in Hot-Switched MEMS Actuators**, ***Z. Yang***, *D. Lichtenwalner, A. Kingon*, North Carolina State University

We have investigated failure mechanisms of metal-contact Micro-electro-mechanical Systems (MEMS) switches using a new accelerated lifetime test facility. The facility utilizes a double-contact upper cantilever from commercial MEMS contact switches, and tests these against bottom gold contact pads within a modified AFM. After a number of switching cycles, both the upper contacts and bottom electrode were characterized by atomic force microscopy (AFM). In this paper, a new phenomenon we term "nano-scale, directional contact metal transfer" during hot switching is reported. The material (gold) in the contact area transfers between the bottom contacts and upper contacts during hot-switching, following the electric field direction. The field-directional transfer was confirmed by DC signal (open circuit voltage, Voc = 6 V) test and reversed DC signal (Voc = -6 V) test. It has been found that the material transfer process is accelerated with the increase of cycling number. Volume analysis of the damaged contact area shows that using an AC signal (with the amplitude of Voc = 6 V) yields an order of magnitude less material transfer damage than DC current, and the material transfer under AC does not have any directional characteristics. This indicates that under the condition of "hot-switching", signal type (AC/ DC; biased or not) may have a significant effect on MEMS switches' failure mechanism. Different material transfer related theories and models are reviewed and examined. A new micro-contact degradation mechanism is proposed.

9:00am **MN-MoM4 Growth of AlN by Pulsed Laser Deposition and Reactive Sputtering Techniques and Fabrication of RF MEMS Resonators**, ***S. Hullavarad***, University of Alaska Fairbanks

AlN is a desirable piezoelectric material for MEMS and NEMS resonators and micro-switches for high frequency filtering applications. The theoretical maximum frequency of AlN is >180 GHz, as opposed to <60 MHz for PZT. The Young's modulus of bulk ceramic AlN at 25°C is 345 GPa, and the density is 3260 kg m-3. This compares to a Young's modulus of 56 GPa for thin film PZT, and a density of 7600 kg/m3 measured for bulk PZT. Additionally, AlN is better suited for the integration of MEMS devices into silicon-based electronics due to its complete compatibility with conventional silicon technologies.[1] In this work, the growth of AlN by Pulsed Laser Deposition method and comparison of properties of AlN thin films with reactive sputter deposition method will be presented. The fabrication and performance of MEMS resonators on AlN thin films deposited by both Pulsed laser deposition and sputtering techniques on substrates consisting of Pt/SiO2/Si structures will be discussed. Special emphasis will be given to the quality (thermal stability, stress) of SiO2 (thermal or PECVD) used as a support layer in fabrication of MEMS resonators. The results obtained by Rutherford Back Scattering Spectroscopy and X-Ray Diffraction techniques to understand the structural stability, composition, crystalline quality of SiO2, Pt and AlN films will be discussed. The quality factor of the resonators in air and vacuum will be compared. It will be shown that the crystalline quality of the films affects the actutation properties of the resonator beams.

[1] S.S.Hullavarad, B. Nagaraj, V.N.Kulkarni, S.Dhar, R.D.Vispute, T. Venkatesan, K.A. Jones, M. Derenge, T. Zheleva, M. Ervin, A. Lelis, C.J. Scozzie, D. Habersat, A.E. Wickenden, L. Currano, M. Dubey, J. Electronic Materials 35, 777 (2006) - A short review.

9:20am **MN-MoM5 Science and Technology of Piezoelectric/Diamond Hybrid Heterostructures for High Performance MEMS/NEMS Devices**, ***O. Auciello***, *A.V. Sumant, J. Hiller, B. Kabius*, Argonne National Laboratory

A new generation of low power microelectromechanical and nanoelectromechanical system (MEMS/NEMS) devices will require new materials and the integration of dissimilar materials, and new micro and nanofabrication processing techniques to achieve high device performance. Most MEMS devices are currently based on silicon because of the available surface micromachining technology. However, the poor mechanical and tribological properties of Si are not suitable for many high-performance MEMS/NEMS devices, such as resonators and switches. A novel ultra-nano crystalline diamond (UNCD) material developed in thin film form at Argonne exhibits exceptional mechanical and tribological properties that make UNCD a suitable material for a new generation of high-performance MEMS/NEMS devices. Piezoelectric-based MEMS attracts much attention due to their high sensitivity and low electrical noise in sensing applications and high-force output in actuation applications. Piezoelectric Pb(ZrxTi1-x)O3 (PZT) thin films have been intensively investigated over the past decade due to its potential applications in a wide variety of devices, such as non-volatile ferroelectric memories and piezoelectrically actuated MEMS/NEMS devices, which can be actuated at comparatively lower voltages (5-10 V) to those actuated by electrostatic action that required higher voltages. Therefore, the integration of functional PZT thin films with the UNCD-based MEMS/NEMS structures opens up the tantalizing possibility of advanced MEMS/NEMS devices. However, the integration of PZT and UNCD is challenging, mainly due to the PZT/UNCD interface and the need to grow PZT at high temperature in oxygen in the presence of a carbon-based material such as diamond. We will review in this paper the fundamental and applied materials science performed in our laboratory to achieve integration of PZT as a piezoelectric actuation material and UNCD as a mechanically superior platform for MEMS/NEMS, and the development of fabrication processes to produce high-performance hybrid PZT/UNCD MEMS.NEMS devices. We will also present data from test of hybrid PZT/UNCD piezo-actuated resonator structures.

This work was supported by the US Department of Energy, BES-Materials Sciences, under Contract DE-AC02-06CH11357.

9:40am **MN-MoM6 NEMS Resonators of Carbon Nanotube Network and Metal-Carbon Nanotube Composites**, ***J.H. Bak***, *Y.D. Kim, B.Y. Lee, S.S. Hong, Y.D. Park*, Seoul National University, Korea

We present nanomechanical torsional resonator and doubly-clamped beam resonator structures fabricated from aluminum-carbon nanotube (CNT) and palladium-CNT composites. In addition, we realize nanoelectromechanical systems (NEMS) structures suspended by self-assembled carbon nanotube network on GaAs surface by adopting highly selective wet-etching and reactive ion etching techniques. Carbon nanotubes have been spotlighted for its great potential as a promising material as well as a future candidate material for nanoelectronics, with CNT's unique electrical and mechanical properties. NEMS structure combined with CNT can be applied to elucidate the nanotube's physical properties as well as further applications. Furthermore, metallic based NEMS resonator structures are of interest due to higher optical reflectivity, ductility, and conductivity compared to insulator- and semiconductor- based NEMS structures. The resonators are electrostatically driven and are detected at room temperatures under moderate vacuum conditions using optical modulation techniques. From identifying fundamental flexural and rotational modes as well as applying continuum mechanics equations, we observe a significant enhancement of the Young's modulus in metallic resonators structures with added CNTs. We will also discuss the characterization of mechanical properties of the structures by AFM force deflection spectroscopy and compare the two characterization techniques.

10:20am **MN-MoM8 Addition and Removal of Stress to Drastically Tune Frequency and Quality Factor of Nanomechanical Resonators**, ***S.S. Verbridge***, *D. Finkelstein Shapiro, H.G. Craighead, J.M. Parpia*, Cornell University

We have used optical drive and detection to study the mechanics of flexural nanostring resonators. Beam stress in devices made of both silicon and silicon nitride is tuned by macroscopically bending the resonator chip, resulting in a drastic tuning of the frequency of the correctly oriented doubly clamped beams. Frequency tuning by as much as several hundred percent is achieved with this technique. Over this wide range of frequency tuning, quality factor is also observed to be tuned by as much as several hundred percent. Highly stressed devices display the highest quality factors, and we therefore conclude that stress can be used as a parameter to increase device performance by increasing both resonant frequency as well as quality factor. Frequency can be drastically tuned and quality factor positively impacted by the addition of both tensile, as well as compressive stress. We discuss the sources of dissipation for these devices, and demonstrate a high tensile stress doubly-clamped beam resonator with sub-micron cross-sections, and a quality factor of 390,000 at 3.7 MHz, in vacuum, and at room temperature. The high frequency and quality factor exhibited by the high stress devices, as well as the significant tuning attained with the chip-bending technique, should prove useful for applications of nanomechanical resonant devices.

10:40am **MN-MoM9 Noise Temperature and Thermodynamic Temperature of Ultrasensitive Cantilevers Below 1 K**, ***A.C. Bleszynski***, *W.E. Shanks*, Yale University, *B. Ilic*, Cornell University, *J.G.E. Harris*, Yale University

Micromechanical systems can be fabricated with the sensitivity necessary for detecting ultra-small forces arising from quantum mechanical effects. We use cantilevers as torsional magnetometers to study the magnetic properties of systems mounted directly on a cantilever. Our goal is to study persistent currents in normal metal rings. The properties of these currents remain an outstanding controversy in mesoscopic physics. As with all sample-on-cantilever arrangements, there are two distinct temperatures that determine the performance of the experiment: the cantilever's Brownian motion temperature (T_n) and the temperature of the sample mounted on the cantilever (T_s). T_n is associated with a single macroscopic degree of freedom extended over the length of the cantilever. T_s on the other hand is associated with the very large number of microscopic degrees of freedom in the sample. For a high-Q cantilever, T_n, which sets the cantilever's ultimate force sensitivity, is in weak contact with the thermal bath at temperature T_b. T_s is in contact with the bath via phonon conduction through the cantilever. This contact can also be weak for a small, electrically insulating cantilever at low temperatures. It is thus a priori uclear whether in a practical experiment T_s and T_n will equilibrate with each other or even with T_b. It is also unclear how they will respond to a localized heat source, e.g. a laser used to monitor the cantilever's motion. We have used our sample-on-cantilever system to realize two primary thermometers to measure both T_n and T_s. We infer T_n by monitoring the cantilever's Brownian motion and T_s from the critical magnetic field of a superconducting sample mounted on the cantilever. We find that for modest laser powers incident on the sample, these two temperatures stay equilibrated to each other and to T_b down to 300mK. For higher laser powers T_s and T_n remain equal to each other but are hotter than T_b. The temperature difference is well-described by a simple model of phonon transport along the cantilever beam. We have also fabricated single crystal silicon cantilevers with integrated micron-scale metal rings. We have demonstrated attonewton force sensitivity with these devices and will present measurements of the rings' susceptibility in the normal and superconducting states.

11:00am **MN-MoM10 Process Development and Material Characterization of Polycrystalline BiTe and PbTe Thin Film Alloys on Si for MEMS Thermoelectric Generators**, ***I. Boniche***, University of Florida, *B.C. Morgan, P.J. Taylor*, U.S. Army Research Laboratory, *C.D. Meyer, D.P. Arnold*, University of Florida

Numerous opportunities exist in commercial and military applications for thermoelectric (TE) energy scavengers to act as integrated power sources. Bulk TE materials and modules are commercially available but are often tailored for heating/cooling applications, rather than power generation. Additionally, these bulk technologies limit the miniaturization of TE modules. This work seeks to develop and characterize vapor-deposited polycrystalline TE thin films on Si substrates for integration with MEMS devices, specifically investigating Bi_2Te_3 and PbTe alloys for both room and high-temperature applications. P-type polycrystalline Bi_2Te_3 and PbTe films from 0.4 μm to 9 μm thick have been successfully deposited on bare and etched Si, thermally oxidized Si, and Si/SiO_2 substrates with patterned metal traces. The films were vapor-deposited in UHV using congruent sublimation of the solid-source parent compounds. Fundamental microfabrication techniques for Bi_2Te_3 films, such as patterning and metallization, have recently been developed to augment previous work on PbTe alloys.[1] Dry etch rates of 0.4 μm/min and 0.7 μm/min were obtained for Bi_2Te_3 and PbTe, respectively. Wet etch rates of ~3 μm/min were achieved using bromine-based chemistries, but at the expense of mask undercut. Films have been characterized electrically using van der Pauw and transfer length method test structures. As-deposited resistivity was 23 mΩ-cm for Bi_2Te_3, and 126 mΩ-cm for PbTe films. Contact resistivities of $2x10^{-4}$ Ω-cm^2 were achieved for Cr/Pt/Au on Bi_2Te_3, and $4x10^{-4}$ Ω-cm^2 for Cr/Au on PbTe. The Seebeck coefficient was measured to be 94 μV/K for Bi_2Te_3 and ~100 μV/K for PbTe alloys. Analytical modeling of in-plane MEMS TE generators showed that film resistivity is a limiting factor for power generation. Various post-deposition annealing treatments were explored to reduce film resistivity, and thus enable higher power delivery. The results show that successive rapid thermal annealing in nitrogen at 400°C can reduce the resistivity of PbTe. The integration of these materials into prototype generator structures will also be discussed, particularly towards developing fabrication compatible TE, heat exchanger, and mechanical MEMS structures.

[1] I. Boniche, et al, PowerMEMS Conf., Nov. 2006.

11:20am **MN-MoM11 Fabrication of Metal-based High Aspect Ratio Microscale Structures by Compression Molding**, ***J. Jiang***, *F.H. Mei, W.J. Meng*, Louisiana State University

Metal-based high aspect ratio microscale structures (HARMS) are basic building blocks for metallic microdevices such as micro heat exchangers[1,2] and micro electromagnetic relays.[3,4] Metallic microdevices may function better when subjected to high stresses, high temperatures, and other harsh conditions. Metal-based HARMS can be fabricated by combining X-ray/UV lithography and electrodeposition, following the Lithographie/Galvanoformung (LiG) protocol.[5] Such primary HARMS made by LiG are expensive. In comparison, production of secondary HARMS by molding replication from HARMS inserts is fast and simple.[5,6] We have demonstrated successful molding replication of HARMS in Pb[7] Al[8], and Cu.[9] Molding replication of metal-based HARMS entails extensive plastic deformation within the molded metal. Understanding the mechanics of microscale compression molding is important for accurately assessing the capabilities and limitations of this technique. The present paper summarizes our results on instrumented compression molding of Pb, Al, and Cu as a function of the molding temperature. Measured molding responses are rationalized with companion elevated-temperature tensile testing of metals using a simple mechanics-based model of the micromolding process. The present results suggest that stresses on the insert during micromolding are determined primarily by the yield stress of the molded metal at the molding temperature and the frictional tractions on the insert sidewalls. Additional factors of complication during high temperature micromolding will be discussed.

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[2] F. Arias, S. R. J. Oliver, B. Xu, R. E. Homlin, G. M. Whitesides, J. MEMS 2001, 10, 107

[3] I. J. Busch-Vishniac, Sensors and Actuators 1992, A33, 207-220

[4] J. D. Williams, W. Wang, Microsystem Technologies 2004, 10, 699

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[6] M. Madou, Fundamentals of Microfabrication, CRC Press, Boca Raton, Florida, 2000

[7] D. M. Cao, W. J. Meng, K. W. Kelly, Microsystem Technologies 2004, 10, 323

[8] D. M. Cao, W. J. Meng, Microsystem Technologies 2004, 10, 662

[9] D. M. Cao, J. Jiang, W. J. Meng, J. C. Jiang, W. Wang, Microsystem Technologies 2007, 13, 503.

11:40am **MN-MoM12 Process Characterization of Vapour Phase Sacrificial Etching**, ***A. O'Hara***, *G. Pringle, M. Leavy*, MEMSSTAR, UK

The manufacture of MEMS devices has primarily used processes and techniques developed for the semiconductor industry. The process characterization is well established for these methods and are then adapted to the MEMS structure. One process unique to MEMS manufacture is isotropic etching of a sacrificial layer. Historically these processes have been developed using wet etch methods, stagnant gas techniques or gas flow processes with limited process capability. Wet processing and stagnant gas processes employ a one process fits all approach. However, it is seen that different MEMS structures require significantly different process optimization and control. Using memsstar systems for etching, based on controlled continuous flow technology CCFT the process is optimised to the structure being etched. In this example for XeF_2 etching, a carrier gas is employed to transport a precise flow of XeF_2 to the process chamber. The flow of the carrier gas determines the flow of the XeF_2. When etching a structure with a large open access to the sacrificial material the etch is seen to be transport limited. The etch rate is dependent on the flow of XeF_2 into the chamber, the higher the flow the higher the etch rate. When the open access to the sacrificial material is very limited the etch is seen to be reaction limited. In this case the etch rate is dependent on the partial pressure of the XeF_2, the higher the partial pressure the higher the etch rate. Using controlled continuous flow of the process gases combined with fine chamber pressure control the sacrificial etch process can be tuned to the MEMS structure being manufactured. Experimentation with different structures is discussed to show that the etch process performance and process window varies depending upon the mechanical materials and dimensions.

MEMS and NEMS

Room: 615 - Session MN-MoA

Materials Processing, Characterization and Fab Aspects

Moderator: B. Ilic, Cornell University

2:00pm **MN-MoA1 Electromechanical Resonators from Graphene Sheets**, ***P.L. McEuen***, Cornell University **INVITED**

We fabricate nanoelectromechanical systems from single and multilayer graphene sheets by mechanically exfoliating graphite over trenches in SiO2. Vibrations with fundamental resonant frequencies in the MHz range are actuated either optically or electrically and detected optically by interferometry. The thinnest resonator consists of a single suspended layer of atoms and represents the ultimate limit of two dimensional nanoelectromechanical systems. The high Young's modulus (E = 1 TPa), extremely low mass (single layer of atoms), and large surface area make these resonators ideally suited for use as mass, force, and charge sensors. We will discuss recent work on nanochambers sealed with graphene membranes. The pressure of the gas inside the nanochamber determines the pressure and damping of the graphene resonator.

2:40pm **MN-MoA3 Determination of the Density, Viscosity and Activation Energy of Small Liquid Volumes using Microcantilevers**, ***G. Hähner****, N. McLoughlin, S.L. Lee*, University of St Andrews, Scotland, UK

The density and the viscosity are important parameters for the understanding and tailoring of many processes taking place in liquids. Their determination is generally performed with macroscopic liquid amounts and in separate measurements. In recent years some approaches have been proposed to determine these properties simultaneously. The majority of the methods applied still requires macroscopic liquid volumes and is based on macroscopic techniques. With the growing interest in microfluidic applications, however, alternative approaches for the determination of liquid properties on the microscale are desirable. We present a method for determining the viscosity and density of small liquid volumes (microliters) simultaneously from the resonance spectra of both magnetically driven as well as thermally excited microcantilevers.[1] Parameters characteristic of the resonance behavior of the system were extracted from resonance spectra recorded in a liquid of known density and viscosity. Subsequently, these parameters were used to determine the properties of further samples. In addition, temperature dependent spectra were exploited to extract the activation energy of viscous flow. The procedure we present is fast and reliable and requires no calibration of the cantilever force constant or specific knowledge of the cantilever geometry. Based around existing AFM technology the approach we propose can be easily adapted to suit a variety of microfluidic applications.

[1] N. McLoughlin, S. L. Lee, G. Hähner Appl. Phys. Lett. 89, 184106 (2006).

3:00pm **MN-MoA4 A Micromachined Ultrasound Transducer for Noncontact Nondestructive Evaluation**, *X. Wang, Y. Fan,* ***W.-C. Tian****, H. Kwon, S. Kennerly, G. Claydon, A. May*, General Electric Co.

We report a capacitive micromachined ultrasound transducer (CMUT) for air-coupled, noncontact, nondestructive evaluation (NDE) applications. Air-coupled ultrasound is an attractive inspection technique for materials or structures that are not suitable for contact or immersion ultrasound inspections, such as the honeycomb composites used in aircraft structures. In the past CMUTs have been extensively studied for water-coupled, immersion applications.[1] In principle, CMUTs have better acoustic impedance match with air compared with piezoelectric ultrasound transducers. This makes them ideally suited for air-coupled NDE. However, they are not widely used in air inspection due to large air attenuation of acoustic power. To overcome this problem, CMUTs' transduction efficiency must be improved. We have successfully developed a large gap air-coupled CMUT. The device has a 1MHz operation frequency and can function both as ultrasound transmitter and receiver. The CMUT structure employs a large gap and a specially patterned SiO2 layer to provide large acoustic output while avoiding dielectric charging. A wafer-bonding process was used in fabrication.[2-4] In this process, a Si substrate was first etched to create cavities and oxidized. The SiO2 on the cavity floor was patterned to form pillar structures, which prevented arcing and charging in actuation. An SOI wafer was then bonded to the substrate wafer followed by handle wafer removal and metallization steps to complete the device. In our tests, CMUTs with patterned SiO2 insulation layers showed consistent operation over time. In comparison, control devices with un-patterned blanket SiO2 layers suffered from unsynchronized cell vibrations and fast signal decay due to dielectric charging. Acoustic though-transmission tests with paired CMUTs showed a loop gain of -51dB. As a comparison, a pair of state-of-the-art commercial air-coupled ultrasound transducers showed a -65 dB loop gain, 14 dB lower than the CMUTs reported here. This clearly indicates that improvement of air-coupled CMUTs is achievable and can lead to wide use of CMUTs for air inspections.

[1] X. Jin, et al., J. Microelectromech. Syst., 8, 100 (1999).

[2] Y. Huang, et al., J. Microelectromech. Syst., 12,128 (2003).

[3] Y. Huang, et al., IEEE T. Ultrason. Ferr., 52, 578 (2005).

[4] W-C. Tian, et al., US Patent Publication #2006/0004289 (2006).

3:40pm **MN-MoA6 Design, Synthesis, and Fabrication of a Biomolecular Nanovalve**, ***H. Li****, L.E. Ocola, O. Auciello, M.A. Firestone*, Argonne National Laboratory

A device containing microfluidic and nanofluidic channels was designed and fabricated to study on the performance of a bio-nanovalve controlled by polarization of ferroelectric substrate. The microfluid channel consisting of 200 (W) x 200 (H) μm and 35 (W) x 200 (H) μm straight channels, micro-nozzles, and micro-diffusers, was designed to provide high driving pressure and low mass flow rate for fluid flow in the nanochannel. A recently developed lead-zirconium-titanate (PZT) substrate integrated with nano-electrodes was coated on the bottom of nanochannels to control the nanovalve made of biological molecules. By observing the fluid mixing behavior variation in nanofluid channels of 200 (W) x 200 (H) nm before and after the polarization of PZT substrate, the function of the bio-nanovalve would be demonstrated. The biovalve will prove useful for many applicatons including lab-on-a-chip and release-on-demand drug delivery systems. This device can also be used to study the basic science of fluid flow and heat transfer at the nanoscale with the purpose of improvement of flow and heat transfer efficiency in nanoscale devices.

4:00pm **MN-MoA7 XeF$_2$ Etching of Si and SiO$_2$ for MEMS Manufacturing**, ***J.-F. Veyan****, Y.J. Chabal,* Rutgers, The State University of New Jersey*, M.Y. Yan, E. Gusev, A.L. Londergan*, Qualcomm

XeF$_2$ is used for etching a number of materials during MEMS fabrication, such as silicon,[1] and metals. Its strong reactivity with silicon and metals leads to rather violent reactions that make it difficult to characterize with typical surface science techniques under typical manufacturing conditions (XeF$_2$ pressure in a few Torr range). The fundamental reactions involved under these conditions are therefore harder to understand than typical elementary surface reactions in ultra-high vacuum conditions. It is therefore important to investigate etching mechanisms under such conditions. This work focuses on the characterization of gas phase, surface species, and substrate surfaces during XeF$_2$ etching using in-situ infrared absorption spectroscopy (IRAS) for both silicon and silicon oxide surfaces under typical etching conditions. To that end, a compact reactor has been constructed out of non-reactive materials (e.g. stainless steel, aluminum, Teflon and Kalrez o-rings), with the capability to perform IR spectroscopy. Despite these precautions, IRAS is critical to detect the presence and role of fluorinated contaminants (from reaction with molecules adsorbed on the walls) and the presence of products.[2] Thus, while XeF$_2$ induces a strongly exothermal reaction with Si, producing large amounts of SiF$_4$ gas (with a characteristic IR signature), and the incorporation of SiF, SiF$_2$ and SiF$_3$ in the subsurface region (~30Å deep) as previously observed in UHV studies, the presence of H$_2$O and HF gas, and CF$_x$ and othe r adsorbed impurities can also be observed, pointing to side reactions on the walls despite thorough baking. XeF$_2$ etching of SiO$_2$ is much weaker and thereby harder to study. IRAS studies confirm that amorphous SiO$_2$ is etched at the rate of ~2-3Å/cycle at room temperature, where a cycle consists of 2Torr in 427cm^3 (i.e. ~ 3.10^{19} XeF$_2$ molecules). Although SiF$_4$ gas is also detected, it is not possible to exclude the potential etching of Si due to scratches. In contrast, no measurable SiF$_4$ gas is observed when crystal quartz is placed in contact with XeF$_2$, indicating that quartz does not etch. Preliminary data indicate that SiO$_2$ etching is highly dependent on the substrate temperature. Based on IRAS data, this talk will discuss various etching mechanisms and optimization of etching conditions for both Si and SiO$_2$.

[1] Harold.F Winter and I.C. Plumb J. Vac. Sci. Tech. B 9(1) 197 (1990)

[2] J. I. Steinfeld, Chem. Rev., 89, 1291, (1989)

4:20pm **MN-MoA8 Advances in Magnetometry through Miniaturization**, ***A.S. Edelstein****, J. Burnette, G.A. Fischer,* U.S. Army Research Laboratory*, S.F. Cheng,* U.S. Naval Research Laboratory*, E.R. Nowak*, University of Delaware **INVITED**

Recent innovations will lead to magnetic sensors that are smaller, more sensitive and/or cost less than current magnetometers. Examples of this are the chip scale atomic magnetometer, magnetic tunnel junctions with MgO barriers, and a device for minimizing the effect of 1/f noise, the MEMS flux concentrator. In the chip scale atomic magnetometer researchers have been able to fabricate the light source, optics, heater, optical cell, and photodiode detector in a stack that passes through a silicon wafer. There are limits on decreasing the size of the cell, because collisions with the cell walls limit the spin lifetime. A search is underway for materials to be used as cell liners that have a smaller effect on the spin lifetime. Theoretical and subsequent experimental work led to the observation of magnetoresistance values of 400% at room temperature in magnetic tunnel junctions with MgO barriers. The large magnetoresistance occurs because electrons in the majority band can tunnel more easily through the MgO barrier than electrons in the minority band. The MEMS flux concentrator has the potential to increase the sensitivity of magnetic sensors at low frequencies by orders of magnitude. The MEMS flux concentrator does this by shifting the operating frequency to higher frequencies where 1/f noise is unimportant. The shift occurs because the motion of flux concentrators on MEMS flaps modulates the field at kHz frequencies at the position of the sensor. The concept and development of the MEMS flux concentrator will be presented.

5:00pm **MN-MoA10 Fabrication of Stationary Micro-Optical Shutter Based on Semiconductor-To-Metallic Phase Transition of W-doped VO2 Active Layer Driven by an External Voltage**

***M. Soltani**, M. Chaker,* INRS-Energie, Matériaux et Télécommunications, Canada*, E. Haddad, R. Kruzelecky,* MPB Communications Inc.*, J. Margot,* Université de Montréal, Canada*, P. Laou, S. Paradis*, Defence R and D Canada-Valcartier

At a transition temperature of $T_t = 68$ °C, thermochromic vanadium dioxide (VO_2) smart coatings undergo a reversible semiconductor-to-metallic phase transition (SMT). This phase transition is accompanied by an important modification of the electrical resistivity and optical properties in the infrared region. The T_t can be controlled by doping the coating with donorlike or acceptor like centers. In addition, the SMT of VO_2 can be controlled by external parameters such as temperature, pressure, photo-carrier injection into a VO_2 heterostructure, and an electric field. VO_2 smart coatings are thus excellent materials for various switching applications. Recently, we have successfully fabricated micro-optical switch device based on semiconducting and transmitting (on) state to the metallic and reflecting (off) state of W(1.4 at. %)-doped VO_2 operating at $\lambda = 1.55$ µm and driven by an external voltage.[1] This device exhibited an extinction ratio (on/off) as high as 28 dB. In addition, the electro-transmittance switching modulation of the device was demonstrated at 1.55 µm by controlling the SMT with superposition of a dc and ac switching voltages. In this paper, we present our recent results on the mico-fabrication and characterization of stationary optical shutter device based on transmittance switching (on/off) of W-doped VO_2 active layer. This shutter consists on 16 smart micro-slit arrays, which can be controlled individually by an external voltage (either a dc or ac switching voltage). This control allows to perform any desirable on-off combination of the micro-optical slits. The starting W-doped VO_2/Al_2O_3 was synthesized by reactive pulsed laser deposition. The micro-slit arrays were patterned by photolithography and plasma etching, whereas Au/NiCr electrical contacts were integrated on the top of the micro-slit by means of the lift-off process. The response of the device was investigated at 1.55 µm by controlling individually the transmittance switching of the active slits by an external voltage. The results show clearly that this device can be used as stationary Hadamard shutter to increase the sensitivity of infrared spectrometer.

[1] M. Soltani, M. Chaker, E. Haddad, R. V. Kruzelecky, and J. Margot, J. Vac. Sci. Technol. A 25(4), Jul/Aug (2007).

MEMS and NEMS

Room: 615 - Session MN-TuM

Integration and Packaging in MEMS/NEMS

Moderator: E. Gousev, Qualcomm MEMS Technologies

8:00am **MN-TuM1 Wafer Level Integration of Ultrananocrystalline Diamond (UNCD) Film with CMOS Devices for Monolithically Integrated Diamond MEMS/NEMS-CMOS Systems**, ***A.V. Sumant***, *O. Auciello,* Argonne National Lab*, H.C. Yuan, Z. Ma,* Univ. of Wisconsin-Madison*, B. Kabius,* Argonne National Lab*, V. Adiga, R.W. Carpick,* Univ. of Pennsylvania*, D.C. Mancini*, Argonne National Lab

Most devices for MEMS are currently based on silicon because of the available surface micromachining technology. The average mechanical and tribological properties of Si, however, are not suitable for many high-performance devices for current MEMS and future NEMS, such as resonators and switches which involve mechanical motion and intermittent contact. Other materials, such as SiC and AlN, have shown some promises due to better mechanical, chemical, and tribological properties compared to silicon. SiC thin films are grown at temperatures above 600°C and therefore incompatible with the CMOS thermal budget. Ultrananocrystalline diamond (UNCD), a novel material developed in thin film form at Argonne, exhibits exceptional mechanical, electrical, chemical, and tribological properties that make it excellent for high-performance MEMS/NEMS. Most importantly, UNCD is the only diamond film that can be grown at 400 °C, and retain properties comparable to that of single crystal diamond. In order to exploit excellent properties of UNCD to develop next generation of devices for MEMS and NEMS, however, such devices have to be integrated with CMOS at the wafer level, which will require a materials integration strategy and detailed understanding of degradation mechanism of CMOS upon integration. This paper discusses integration of UNCD with CMOS devices at wafer level (200 mm), 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 °C in a plasma-deposition system using microwave-plasma-enhanced chemical vapor deposition with Ar-rich/CH4 gas mixture. The CMOS devices 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 cross-section TEM and RBS studies of the UNCD/CMOS interface and discuss the possible mechanisms responsible for the degradation of CMOS performance.

This work was supported by DARPA under contract MIPR06-W238 and US Department of Energy, BES-Materials Sciences, under contract DE-AC02-06CH11357.

8:20am **MN-TuM2 Optical Excitation of Higher Flexural and Torsional Modes of Nanoelectromechanical Oscillators**, ***B. Ilic,*** Cornell University*, S. Krylov,* Tel Aviv University, Israel*, M. Kondratovich, H.G. Craighead*, Cornell University

Excitation of biologically functional micro and nanomechanical structures using optical fields is a recently emerging arena of research that couples the fields of optics, fluidics, electronics and mechanics with potential of generating novel chemical and biological sensors. We present experimental and theoretical elucidation of optical excitation of higher order flexural and torsional modes in resonant nanoelectromechanical systems (NEMS). The modulated optical fields were coupled directly into the NEMS device layer causing amplified mechanical vibrations. Dynamic detection of vibrational characteristics of nanomechanical resonators, fabricated from low-stress silicon nitride and mono-crystalline silicon thin film layers, was accomplished using optical interferrometry. As a model system, 200nm and 250nm thick single crystal silicon cantilevers with dissimilar lengths and widths ranging from 6 to 12µm and 500nm to 1µm, respectively, were fabricated using surface micromachining techniques. We have analyzed the actuation mechanism using finite element modeling, and we found that the dominant actuation mechanism in close proximity of the clamped end was primarily thermal. In contrast, mechanical traveling waves are attributed as possible excitation mechanisms in the far-field regime. Higher order modes of slender cantilevers, calculated using linear Euler-Bernoulli beam model, differed significantly from the measured values. Three dimensional finite element analysis incorporating shear, rotational inertia, deplanation and non-ideal boundary conditions due to the structural undercut, are shown to adequately describe the dynamics of the nanomechanical structures. The quality factor of a particular in-plane harmonic was consistently higher than the transverse mode. The increased dissipation of the out of plane mode was attributed to material and acoustic loss mechanisms.

8:40am **MN-TuM3 Ultrasensitive, Magnet-tipped Cantilevers for Magnetic Resonance Force Microscopy**, ***S.A. Hickman***, *S.R. Garner,* Cornell University*, L.E. Harrell,* United States Military Academy*, B.I. Penkov, S. Kuehn, 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 a single molecule - an extremely exciting prospect. To date we have demonstrated a sensitivity of ~10^5 proton spins. Achieving the attonewton force sensitivity necessary to image single proton spins requires custom-fabricating cantilevers with extreme aspect ratios and mitigating sample-induced dissipation. 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. We have developed an electron-beam-lithography process for batch fabricating nanoscale tip magnets overhanging the leading edge of ultrasensitive silicon cantilevers. As proof of concept, we will present a 50-nm wide overhanging cobalt magnet fabricated by a process involving electron beam lithography and anisotropic KOH etching, as well as cantilevers with 50-600 nm wide, non-overhanging magnets. Our current goal is to integrate the separate processes of cantilever fabrication and magnet fabrication. With our designed cantilever, we expect a sensitivity of better than 10^3 protons. Even through the process integration challenges are daunting, our successes so far indicate that batch fabricating cantilevers capable of detecting single proton magnetic resonance may indeed be feasible.

9:00am **MN-TuM4 Fabrication of Reliable Through-Silicon via (TSV) Interconnects for 3D Stacking**, ***I.U. Abhulimen***, *A. Kamto, Y. Liu, S. Burkett, L. Schaper*, University of Arkansas

The formation of through-silicon vias (TSVs) provides vertical interconnects that can be used in 3D stacking technology. A sloped via sidewall is essential for conformal coverage in subsequent deposition steps that provide insulation (SiO_2), barrier (TaN) and metal seed (Cu) layers. In this paper, varying via sidewall angles (82° - 90°) are investigated which allow variable degrees of conformal lining of the insulation, barrier and seed layers. The critical thickness of these lining layers that enable conformal coverage of the via sidewall is also investigated. Via insulation is deposited by plasma enhanced chemical vapor deposition (PECVD), while barrier and Cu metal seed layers are deposited by sputtering. A modified Bosch process using a deep reactive ion etch (DRIE) time multiplexing tool is used to create the different via profiles on 125 mm diameter silicon wafers. The cross-sectional view of via lining materials (SiO_2, TaN, and Cu) are examined with both an optical microscope and an environmental scanning electron microscope (ESEM). The via profile is examined using the ESEM. Furthermore, for a fixed via sidewall angle, variable aspect ratios are examined to determine the via profile that can be conformally lined and filled by Cu electroplating without any voids. The aspect ratios of the vias under study are 3, 4, 5, 6, and 8. Electrical performance and via integrity of the TSV process is also reported. Test structures are created during TSV processing that allow for a thorough study of interconnect reliability. This includes tests for via chain continuity, single via resistance, and via isolation.

9:20am **MN-TuM5 Micro and Nano Electro-Mechanical Systems: Technology for Engineering Metamorphosis**, ***A. Lal***, DARPA **INVITED**

The core of MEMS technology is the capability to form chip-integrated micro beams, cantilevers, and plates. DARPA has mined MEMS technology since the early 1990s in an effort to provide increased performance and functionality to integrated circuits. MEMS allows one to use reduced mass for increased resonance frequencies in micro-resonators; craft long and thin high aspect ratio structures for high thermal resistances; and generate huge surface-to-volume ratios for increased interaction with the environment. MEMS technology changes systems so rapidly that there is less of an evolution, and more of a metamorphosis; gradual growth is replaced by complete transformation. This is especially true in the last decade of MEMS, where integration over multiple MEMS devices is finally making the Systems part of MEMS closer to reality. This talk will describe scaling issues in realizing MEMS systems with a particular emphasis on micropackaging, new materials, and design principles on reliability of MEMS and NEMS subcomponents. The successful realization of the chip-scale atomic clock, which is a multiphysics microsystem, will be described in the context of MEMS thermal isolation.

10:40am **MN-TuM9 Self-Packaged Micro Fluorescence Detection Systems**, ***S.S. Kim***, *E. Saeedi,* University of Washington, *D.R. Meldrum,* Arizona State University, *B.A. Parviz*, University of Washington

We present a self-packaging micro-fluorescence biosensor chip which assembles fully functional separately fabricated micro-components onto a common substrate. The essential components of the fluorescence based sensor are: 1) an excitation light source, and 2) a means to detect fluorescence emission. AlGaAs LEDs and silicon pn-junction photosensors are self-assembled onto a glass template to meet these requirements. The micro-components range in size from 100 to 300 microns and were specifically designed to optimize and aid in the self-assembly process. The powder-like collection of micro-components are suspended in a liquid and flowed onto a glass substrate. They self-assemble into their receptor site locations through gravity, shape recognition, and capillary forces, resulting in an array of individually addressable fluorescence detection units. Self-assembly provides a number key advantages over traditional packaging and fabrication approaches. First and foremost, it allows us to integrate micron-scale heterogeneous materials together onto a common substrate. This gives us a unique ability to have all the essential components of a fluorescence detection system on chip, without the need for an external scanner device or benchtop system. Second, the self-assembly process is parallel in nature and benefits from economies of scale. Large arrays of devices can be packaged at the same time. We have demonstrated the packaging of a 10K element array onto a plastic substrate. Thousands of individually addressable fluorescence detection units are possible allowing for data acquisition of a large number of samples simultaneously without being limited by the field of view of the optics. Third, self-assembly gives us the ability to use low cost substrates such as glass and plastics. Only small amounts of semiconductor materials are used where they are needed, reducing the total amount used for each device. This has the potential to drive down the overall cost per device low enough to make them disposable, opening new doors to biosensing applicaitons which require a fluorescence detection platform which is both portable and disposable. Potential applications include point-of-care diagnostics, bioterrorism, food/industrial testing, HIV/STD testing in developing countries, and so on. The benefits of mature fluorescence based assays could be realized on a portable and disposable chip-level platform using this approach to device packaging.

11:00am **MN-TuM10 Bi-Directional Transport of Ultra-Low Volume Droplets Using Capacitive Sensing**, ***P. Dykstra***, *X.Z. Fan, M. Mischiati, L.A. Mosher, N.P. Siwak, R. Ghodssi*, University of Maryland

MEMS cantilever sensors are utilized to detect trace quantities of specific agents in liquid. One of the most common methods to deliver the fluid is by flooding the cantilever's surroundings, but this often leads to stiction-related failures due to surface tension. We present a bi-directional microfluidic device for analyte delivery to MEMS sensors. Our device can deliver a picoliter-sized droplet to the cantilever sensor and then retract the droplet without wetting the entire sensor, thus eliminating this problem. The most commonly used drop-on-demand technology through a nozzle, exploited for inkjet printing and for the selective deposition of polymers, is based on the release of an entire droplet over the target. Our design proposes to eject only enough to reach the target area, without breaking the droplet from the bulk of the liquid. This allows the droplet to be retracted by reversing the flow, thus achieving bi-directional transport of ultra low volumes of liquid. Droplet control is facilitated by position-sensing from a capacitive sensor. As the droplet lowers, a change in capacitance is measured by a sensor electrode located below the nozzle. Our microfluidic system, consisting of packaging, micronozzle, and microsensor wafers, was fabricated using conventional MEMS techniques. The through-etched micronozzle was diced and aligned to the microsensor wafer using peg-in-hole (SU-8 pegs in etched silicon holes) assembly. This precisely aligns the nozzle over a gold electrode on the sensor wafer. Capacitance is measured between the nozzle die itself and the gold electrode using an Agilent CV meter. The liquid is administered using a syringe pump at a constant flow rate. Our initial results show that the capacitance slowly rises as the droplet is formed. A significant change in capacitance occurs when the droplet makes contact with the microsensor. A complete analysis of droplet formation measurements, by way of capacitive sensing, will be presented. Transport will be characterized to enable the integration of our device with any MEMS sensor.

11:20am **MN-TuM11 Microfluidic Transport Control Using Remotely Powered Semiconductor Diodes**, ***D.N. Petsev***, University of New Mexico, *S.T. Chang, O.D. Velev,* North Carolina State University, *V.N. Paunov*, University of Hull, UK

The precise control of fluid transport in microchannels is of paramount importance for the successful design and operation of fluidic devices. We have recently demonstrated[1] that using miniature diodes embedded in the walls of fluidic microchannels in combination with AC field is a very simple and convenient tool to manipulate the flows in microchannels. Our focus is on two particular problems briefly described below Mixing. Due to the low Reynolds number of microflows, mixing of components is a real challenge. Due to the laminar character of the flow different solution tend to flow side by side and the only way for solutes to cross the streamlines is by diffusion. Using properly (anti-parallel) oriented diodes, placed alongside the channel walls, allows generating a vortex fluid motion by simply turning on a properly connected Alternate Current (AC) field source. Such vortex dramatically improves the mixing in the microfluidic device. Another advantage of this approach is that such diode mixer can easily be turned on and off through the AC field power source. Separation. Using parallel oriented diodes and a combination of AC and Direct Current (DC) fields in a loop-shaped channel allows complete decoupling of the fluid electroosmosis from the analyte electrophoresis. Balancing the electrophoretic and convective forces on the different analytes allows for a very easy and efficient separation. The parallel oriented pair of diodes, powered by the applied AC field, acts as a miniature pump and drives the fluid in a circulatory motion in the loop shaped channel. Any charged analytes, however, will not migrate in the AC field. Applying DC field to the fluidic device will not drive the fluid motion because this particular design (closed symmetric loop) cancels the electroosmotic driving force. Hence, combining the two fields (AC and DC) allows decoupling of the fluid flow from the particle electrophoretic migration.

[1] S. T. Chang, V. N. Paunov, D. N. Petsev and O. D. Velev, "Self-Propelling Microdevices and Microfluidic Pumps Based on Remoutely Powered Miniature Semiconductor Diodes, Nature Materials, 6 (2007) p. 235.

11:40am **MN-TuM12 Towards Feedback Control with Integrated Position Sensing in Micromachines**, ***M.I. Beyaz***, *N. Ghalichechian, A. Modafe, R. Ghodssi*, University of Maryland

Micromachines require closed loop systems to facilitate synchronization and yield maximum performance. However, little effort has been spent on implementing feedback control in these devices. We present for the first time the design, fabrication and testing of an integrated feedback control system for a synchronous electrostatic micromotor. This system aims to synchronize the mechanical motion and the electrical excitation to improve stability and performance. The micromotor is composed of a stator and a movable slider supported on microball bearings. Interdigitated photodiodes are located on the stator to detect the position of the slider moving relative to the stator electrodes. Through holes, created by deep reactive ion etching, are aligned with the poles on the slider. This allows light, provided from the top, to reach the photodiodes on the stator. The design is such that the optical sensing of slider position is achieved by simultaneous alignment of pole-electrode and hole-photodiode pairs, causing an increase in photodiode current. The change in current is sensed and the appropriate voltages are applied to stator electrodes by a feedback circuit. The designed photodiodes have been implemented on n-type 20 Ω-cm silicon wafers. The fabrication consists of etching the native oxide on the wafer, aluminum sputtering and wet etching. Prior to the integration of the photodiodes with the micromotor, a test setup was built to verify the feasibility of the feedback system. In this setup the stator, on which only the photodiodes are fabricated, is fixed to an oscillating platform driven sinusoidally at 5 Hz at an amplitude of 1.6 mm. The slider is kept stationary and a light source is provided from the top. Resulting photodiode current depends on how much light it receives through the holes. The motion of the stator is monitored by the photodiode response that is in the form of a triangular wave. Each peak on the waveform corresponds to a complete alignment between the photodiode and a hole on the slider. Using this peak detection, the instantaneous platform speed is calculated showing good agreement with the applied sinusoidal motion with an R^2 value of 0.925. This work verifies the feasibility of the feedback system for the given micromotor to achieve higher speeds and to stand varying load conditions. Detailed fabrication steps and experimental results of the micromotor with the control system will be presented.

12:00pm **MN-TuM13 An Adaptive Feedback Control Circuit for Resonator Sensors**, ***X.Z. Fan***, *N.P. Siwak, R. Ghodssi*, University of Maryland

Integration of smart electronics with MEMS sensors will enable systems to be versatile, compact, and portable. MEMS resonator sensors are powerful tools for the detection of target analytes due to the high sensitivity of resonant frequency to absorbed mass. We present an adaptive feedback control circuit to detect and trace the resonant frequency of MEMS resonator sensors. The purpose of our feedback circuit is its integration with a fully developed III-V optical resonator system for chemical and biological sensors, facilitating testing and data acquisition. Feedback circuits with similar functions, such as self-excitation systems, have been reported before in literature. These systems, however, require phase and amplitude compensation stages that require separate designs for each resonator measured. Our feedback circuit utilizes a hill climbing algorithm which is valid for any resonator sensor that exhibits any range of resonant frequency, thus broadening the applicability of the circuit. The hill climbing algorithm sweeps the driving frequency searching for maximum cantilever response. The algorithm is implemented using a four-stage CMOS circuit consisting of an amplitude detector, a differentiator, a digital logic circuit, and a voltage controlled oscillator (VCO). The feedback circuit receives the displacement output of the resonator and supplies the actuation signal to the resonator from the VCO output. Utilizing the hill climbing algorithm, the resonator is driven at its resonant frequency. By monitoring the VCO input voltage, the resonant frequency with respect to time can be measured. We have confirmed the adaptability of the design of the circuit with initial testing results. The results have demonstrated that the maximum amplitude of an input signal can be detected with input frequencies ranging from 100 KHz to 500 KHz. This range is only limited by the frequency response of the CMOS components. A delay of 3 ms was observed between the input and output signal of the circuit, which is acceptable due to a significantly larger sensor time constant. We will present the test results of the combined circuit with indium phosphide MEMS cantilever sensors. The flexibility of the circuit and its improved capabilities over conventional measurement circuits will be demonstrated.

MEMS and NEMS

Room: 4C - Session MN-TuP

MEMS and NEMS Poster Session

MN-TuP1 Surface Potential and Resistance Measurements for Detecting Wear of Chemically-Bonded and Unbonded Molecularly-Thick Perfluoropolyether Lubricant Films Using Atomic Force Microscopy, ***M. Palacio***, *B. Bhushan*, The Ohio State University

The wear of perfluoropolyether (PFPE) lubricants applied on Si (100) and a Au film on Si (100) substrate at ultralow loads was investigated by using atomic force microscopy (AFM)-based surface potential and resistance measurements. Surface potential data is used in detecting lubricant removal and the initiation of wear on the silicon substrate. The surface potential change is attributed to the change in the work function of the silicon after wear, and electrostatic charge build-up of debris in the lubricant. It was found that coatings that are partially bonded, i.e., containing a mobile lubricant fraction were better able to protect the silicon substrate from wear compared to the fully bonded coating. This enhanced protection is attributed to a lubricant replenishment mechanism. However, an untreated lubricant coating exhibited considerable wear as it contains a smaller amount of lubricant bonded to the substrate relative to the partially bonded and fully bonded coatings. A sample subjected to shear is shown to have improved wear resistance, and this enhancement is attributed to chain reorientation and alignment of the lubricant molecules. The detection of wear of PFPE lubricants on Au by an AFM-based resistance measurement method is demonstrated for the first time. This technique provides complementary information to surface potential data and is a promising method for studying the wear of conducting films.

MN-TuP2 Wetting Behavior During Evaporation and Condensation of Water Microdroplets on Superhydrophbic Patterned Surfaces, ***Y.C. Jung***, *B. Bhushan*, The Ohio State University

Superhydrophobic surfaces have considerable technological potential for various applications due to their extreme water repellent properties. The superhydrophobic surfaces may be generated by the use of hydrophobic coating, roughness and air pockets between solid and liquid. The geometric effects and dynamic effects, such as surface waves can destroy the composite solid-air-liquid interface. The relationship between the water droplet size and geometric parameters governs the creation of composite interface and affects transition from solid-liquid interface to composite interface. Therefore, it is necessary to study the effect of droplets of various sizes. We have studied the effect of droplet size on contact angle by using evaporation studies with droplet radii ranging from about 700 to 300 µm. Experimental and theoretical studies of wetting properties of silicon surfaces patterned with pillars of two different diameters and heights with varying pitch values are presented. We propose a criterion where transition from Cassie and Baxter regime to Wenzel regime occurs when the droop of the droplet sinking between two asperities is greater than depth of the cavity. Based on the experimental data and the proposed transition criteria, the trends are explained. For the first time, environmental scanning electron microscopy (ESEM) is used to form smaller droplets of about 20 µm radius and measure the contact angle on the patterned surfaces. The investigation has shown that ESEM provides a new approach to wetting studies on the microscale.

MN-TuP3 The Effect of Sliding and Peeling Motion on Gecko Adhesion, *T.W. Kim*, ***B. Bhushan***, The Ohio State University

The attachment pads of geckos exhibit the most versatile and effective adhesive known in nature. Their fibrillar structure is the primary source of high adhesion and their hierarchical structure produces the adhesion enhancement by giving the gecko the adaptability to create a large real area of contact with surfaces. Although geckos are capable of producing large adhesive forces, they retain the ability to remove their feet from an attachment surface at will. Detachment is achieved by a peeling motion of the geckoâ?Ts feet from a surface. Recent investigations have shown that a load applied normal to the surface was insufficient for an effective attachment of seta. The maximum adhesion force was observed by sliding the seta laterally along the surface under a preload. Therefore, in this study, for the three-level hierarchical model recently developed by the authors, the sliding motion of a gecko seta is considered to understand how the sliding contributes the adhesion and friction forces. In addition, the peeling motion of a gecko seta has also modeled. The peeling force is obtained as a function of peeling angle for the gecko setae contacting with the different random rough surface. This works are useful for understanding biological systems and for guiding of fabrication of the biomimetic attachment system.

MN-TuP4 Nanotribological Studies of Platinum Coated Probes Sliding against Coated Silicon Wafers For Probe-based Recording Technology, ***K.J. Kwak***, *B. Bhushan*, The Ohio State University

Some of the new alternative information storage technologies being researched are probe-based recording techniques. In one of techniques, a phase-change medium is used and phase change is accomplished by applying either high or low magnitude of current which heats the interface at different temperatures. Tip wear at high temperature is a serious concern. For wear protection of the phase-change chalcogenide media, diamondlike carbon (DLC) film and various lubricant overcoats were deposited on the recording layer/silicon substrate. Nanotribological properties of platinum (Pt) coated probes with high electrical conductivity have been investigated in sliding against the coated media using an atomic force microscope (AFM). A silicon grating sample and SPIP software of Image Metrology were used to characterize the change in the tip shape and evaluate the tip radius. The wear tests were performed at the sliding velocity ranging from 100 µm/s to 100 mm/s. Pt-tips on the lubricant coated DLC film surfaces showed less sensitivity to the velocity and the load as compared to the unlubricated DLC film surfaces. The lubricant coatings could be used to reduce Pt-tip wear and friction between Pt-tip and DLC film surfaces. The results suggest that wear mechanism at low speed appears to be adhesive. At high speeds, the wear primarily depends on surface chemistry of the coated layers such as tribochemical oxidation.

MN-TuP5 A Microfluidic Device for the Spring Constant Calibration of Micro-Cantilevers and for Measuring Fluid Flow Velocities, ***G.V. Lubarsky***, *G. Haehner*, University of St. Andrews, UK

We present a new microfluidic device and method based on cantilever sensing technology. Utilizing an artificially created force gradient the method can be applied for the non-destructive calibration of the normal spring constant of micro-cantilevers. In contrast to the most recent efforts to advance cantilever calibration, our method can be performed in situ, is easy to use, reliable, accurate, and non-destructive, i.e., does not involve any contact between the cantilever and another surface. The method has great potential for the calibration of modified probes, bio-sensor cantilevers etc. In addition, provided the dynamic properties of the cantilever sensor are designed and clearly defined, the device can be easily incorporated in microfluidic systems to measure the velocity of fluid flows.

MN-TuP6 Improvement of Surface Roughness of Cerium Oxide Thin Film by Chemical Mechanical Polishing for Oxygen Gas Sensor, *P.-J. Ko, Y.-K. Jun, P.-G. Jung,* Chosun University, Korea, ***N.-H. Kim***, Sungkyunkwan University, Korea, *W.-S. Lee*, Chosun University, Korea

Cerium oxide (CeO2) is one of the most widely used materials for the oxygen gas sensors. Surface roughness of CeO2 thin films must be improved because the electrical and sensing properties of CeO2 thin films are determined by these characteristics. Chemical mechanical polishing (CMP) processing was selected for improving the surface roughness of CeO2 thin films. Surface roughness and within-wafer non-uniformity (WIWNU%) of spin coated CeO2 thin films were examined with a change of CMP process parameters. The optimized process condition, reflected by both the surface roughness and the hillock-free surface with the good uniformity, was obtained. The effects of the improved surface roughness on the sensing property of CeO2 thin films were also confirmed. The improved sensing property of CeO2 thin films for oxygen sensors were obtained after CMP process by the improved surface morphology. Therefore, we conclude that sensing property of CeO2 thin film is strongly dependent on the surface roughness of CeO2 thin films. Acknowledgement: This work was supported by Korea Research Foundation Grant (KRF-2006-005-J00902).

MN-TuP7 Fabrication of Body Insulated Conductive Cantilever with Metalic Nano Tip, ***S.H. Park***, Myongji University, Korea, *S.H. Kim*, Korea Electronics Technology Institute, *C.J. Kang, Y.J. Choi, J.W. Kim, Y.S. Kim*, Myongji University, Korea

Scanning Probe Microscopy (SPM) can be used to analyze specimens at sub-micrometer scale either by profiling their surface morphology or by measuring their electrical property. SPM has become one of the essential research tools in the field of biology because of its lateral resolution superior to optical microscopy. For the application to biological samples, the SPM probe should be occasionally immersed into an aqueous environment in the form of buffer solution. If we want to get electrical information of the sample in the aqueous environment with commercially available surface conductive cantilevers, electrical leakage from the cantilever body to the solution takes place, which results in poor image resolution. In this study, we fabricated body insulated conductive cantilever in order to avoid leakage current through cantilever surface. Electron Beam Induced Deposition (EBID) technique was used to fabricate well localized conductive nano probe. By optimizing the EBID tip growth condition, we could acquire probe whose base diameter and effective length are below 300nm and a few µm. Tungsten hexacarbonyl [W(CO)6] is a popular precursor material used in placing conductive deposits with the EBID method. We analyzed the constituents of fabricated nano probe by Energy Dispersive X-ray Spectroscopy (EDXS) and microscopic Fourier Transform Infrared spectroscopy (µ-FTIR) which detects element species and chemical bond. We also examined resolution of the fabricated nano probe compared with the conventional metal probe by recording topographic images and electrostatic force images (EFM) of gold electrodes simultaneously. When measuring an electric signal in buffer solution, we could reduce a leakage electric current. This result suggests the possibility of direct fabrication of high aspect ratio and conducting nano SPM probe on the body insulated conductive cantilever.

MN-TuP8 A Robust Parametrically Excited MEMGyroscope, ***L.A. Oropeza-Ramos***, *C.B. Burgner, C. Olroyd, K.L. Turner*, University of California, Santa Barbara

We present a novel scheme for a robust micro gyroscope which is actuated parametrically and is less sensitive to parameter variations. We experimentally demonstrate that using a parametric resonance based actuator, the drive mode signal has rich dynamic behavior with a large response in a large bandwidth. In this way the system is able to induce oscillations in the sense mode due to Coriolis force, despite that there is a clear disparity on the drive and sense natural frequencies. Thus we propose a scheme that reduces the sensitivity loss due to mismatching in the drive and the sense natural frequencies, which is a common problem in micro gyroscopes based on harmonic oscillators, and also increases significantly the range of frequencies where the gyroscope can operate due to its inherent dynamical properties. Rate table characterization is given. Extensive effort has been applied to gyroscopic structures based on two or more degree of freedom (DOF) harmonic oscillator.[1] For the 2 DOF type, the drive and the sense resonant modes are tuned to be equal (or nearly equal) in most cases, in such a way that the output is amplified by the quality factor Q, resulting in high sensitivities. Due to the current fabrication processes, structural asymmetries are inevitably present; therefore matching of frequencies commonly requires external trimming or implementation of control schemes. In this paper we present the realization of a novel Micro Electro Mechanical Gyroscope actuated by a set of noninterdigitated comb fingers which generate a force with time and displacement dependent stiffness coefficients. Thus, parametric resonance excitation amplifies the drive mode response over a wide set of frequencies. In this way, differences in drive and sense natural frequencies do not compromise the sensitivity in 1kHz range. The 2 DOF micro gyroscope is fabricated using the standard SOI process flow and rate table characterization is presented under 50 mTorr pressure. The sensor response is detected with a capacitive readout hybrid wire bonded to the gyroscope on a chip. Our device has demonstrated a scale factor nonlinearity of 0.8% within ±150 °/sec. Thus, in this demonstration the micro gyroscope is robust to parameter variations.

[1] Yazdi, N., et.al., IEEE Proceedings, Vol.86, No.8, 1998.

MN-TuP9 Modeling of Asymmetric Microelectrode Array and Capillary Forces for Fluidic Self Assembly in MEMS, ***A. Dang***, Netaji Subhas Institute of Technology, India, *M. Dhayal*, National Physics Laboratory, India

Modeling and quantitative design play a key role in MEMS to explore the difference parameter space that can influence the performance of micro fluidic devices. In this study the effects of actuation profile incorporated with geometrical dimensions has been investigated on self-assembly of different types of fluids in micro fluidic devices. Different types of asymmetric electrode array were designed and associated electric field profile had been modeled. The optimization of operating conditions of asymmetric field profile with controlled geometrical dimensions on self-assembly of different type of polar solutions has been investigated for different biological applications.

MN-TuP10 Effect of Au Promoter Layers on NOx Sensitivity of Indium Oxide Solid State Sensor, ***S. Kannan***, *M. Sorenson, L.W. Rieth, F. Solzbacher*, University of Utah

Stricter global emission regulations have generated an immediate need to develop high temperature compatible (>500°C) gas sensors for monitoring exhaust. Indium Oxide (In_2O_3) thin films with Au promoter layers have exhibited excellent sensitivity (S ~ 20) for detection of NO_x at temperatures greater than 500°C. Gas sensitivity results will be interpreted as a function of In_2O_3 film structure and morphology. Thin film In_2O_3 layers (~150 nm) were RF sputter deposited in an ambient of pure argon (10 mtorr, 600 W, 2.4 nm/min) on Pt inter-digitated electrodes (IDE). Au promoter layers (~3nm) were deposited on top of the In_2O_3 film in a separate sputtering system. The thin films were annealed in nitrogen, oxygen, forming gas (2% H_2 in Ar) or mixtures of nitrogen and oxygen up to 15 hours at temperatures from 700°C to 1000°C. X-ray diffraction (XRD) results reveal as-deposited and films annealed in oxygen have cubic crystallite structure with several reflections present. In_2O_3 films annealed in nitrogen show the presence of (321) (411) grains in addition to the as-deposited peaks. Atomic force microscopy (AFM) reveals as-deposited films having an average particle size of 25 nm (RMS = 1.5 nm) which increase to a particle size of up to 65 nm with annealing (RMS = 2.6 nm). These images also suggest all annealed samples with Au promoter change in morphology and increase roughness to 6.5 nm. X-ray photoelectron spectroscopy (XPS) revealed the annealed films to be In rich (45% In, 55% O). Films annealed in forming gas islanded exposing Si XPS peaks from the substrate.Four point probe measurements show the resistivity increase from as-deposited values of 0.0045 Ω-cm to larger than probe can measure(~ 7 Ω-cm) for forming gas annealed films and the results suggest the annealing ambient is important. In_2O_3 films were tested for gas sensitivity (S) towards NO_x (0-25ppm), NH_3 (25 ppm), CO_2 (1000 ppm), H_2 (5000 ppm) in synthetic air (80% N_2, 20% O_2) and nitrogen as carrier gas. In_2O_3 films with Au as a promoter annealed in N_2 at 900°C for 5 hours exhibited excellent sensitivity (S~5) operating at 500°C and S~1 at operating temperature 650°C for detection of NO_x. Sensitivity to NH_3 as well as CO_2 was low relative to NO_x at 500°C and 650°C (S~0.1).

MN-TuP11 XeF_2 Etching of Metallic Films, ***O. Celik***, *N. Shankar, A.V. Ermakov, L. Goncharova, Q. Jiang, L. Wielunski, E. Garfunkel,* Rutgers University, *X.M. Yan, A.L. Londergan, E. Gousev*, Qualcomm MEMS Technologies

The controlled etching of micro/nano structures is very important for a variety of technological applications, including MEMS fabrication. XeF_2 is an isotropic and selective vapor phase etchant used to etch Si and metals in MEMS and other devices. For better process control and device functioning, it is important to understand the etching mechanism at the molecular level. In this study we have explored the surface and gas phase chemistry of XeF_2 etching of metallic films. Down stream mass spectrometry is used to identify the gas phase by-products in the etching process. RBS and MEIS are used to measure the thickness of the films and the depth profile of near-surface species after etching. The etch rate is calculated from film thickness changes. The etched surface composition and chemical state are further investigated by XPS. Based upon the gas phase by-products during etching, surface species and their depth profiles, and the etching rate, a reaction mechanism of XeF_2 etching is proposed.

MN-TuP12 Parametric Amplification in Electromagnetically Actuated Resonant Chemical Sensors, ***K. Lukes***, *K.L. Turner,* University of California, Santa Barbara, *J. Rhoads, S. Shaw*, Michigan State University

This work presents a novel implementation of mechanical domain, parametric amplification in electromagnetic microcantilevers. Parametric amplification is the amplification of a signal due to pumping energy into the system parametrically. This class of resonators shows great potential for implementation as chemical sensors, since they exploit the induced electromotive force (emf) for sensing[1] giving potential for complete onchip integration. It is difficult to recover the sense signal because it is several orders of magnitude smaller than the drive signal. Parametric amplification offers low noise gain for signal recovery. We describe the model and experimental validation of parametric amplification. A measure of merit for the amplifier is its gain. Gain is defined as the ratio of sensor's amplified to harmonic response. Analysis of the gain function shows the response is amplified asymptotically as the pump approaches the limiting magnitude defined by the onset of parametric resonance, and depends on the square root of sin(2φ), where φ is the phase shift between the input signals. Parametric amplification is achieved by the microcantilevers. Each cantilever has metal wires deposited on the surface, making it a closed current loop. The device sits on an angled permanent magnet. An AC current, consisting of the sum of the harmonic and parametric signals, passes through the device producing the Lorentz force. The force can be broken into a normal component, the harmonic forcing, and axial component, the parametric pump. The dynamics of the resonators are measured using a single point laser vibrometer in vacuum.[2] These devices have shown the capability for emf sensing;[1] vibrometry is used for proof of concept. Experimental data confirms the expected relationship of gain to φ and pump magnitude. We have successfully shown parametric amplification in electromagnetically actuated microcantilevers. Theoretical results anticipate large gains and experimental data confirms that these gains are feasible. The ability to mechanically amplify the signal of the transducer shows potential for a complete on-chip chemical sensor.

[1] Requa, M.V. and K.L. Turner. APL, 2006. 88(26)

[2] Turner, K., Hartwell, P., MacDonald, N. in 10th International Conference of Solid-State Sensors and Actuators (Transducers 1999). 1999.

MN-TuP13 Capacitive Displacement Sensing for Comb Drive Actuators Operating in Aqueous Media, ***P. Ponce***, *V. Mukundan, B. Murmann, B.L. Pruitt*, Stanford University

We present a system that is capable of measuring displacements in a comb drive actuator operating in aqueous electrolytes. Underwater electrostatic actuators are promising tools for manipulation of biological samples in media.[1,2] Optical techniques have been reported for measurement of these actuator displacements.[3] Apart from accuracy and portability, electrical measurements are beneficial in setting up feedback systems for controlled actuation. Relative changes between the capacitances of the comb drive are measured by connecting them to parallel oscillator circuits. The operational frequencies of each oscillator depend directly on its corresponding capacitance value. In order to avoid electrolysis and electrostatic shielding effects in ionic media, the oscillators resonate at high frequencies (around 2-10 MHz) and the voltages across the comb drive electrodes are limited to approximately 100 mV, peak-to-peak. A major advantage of the described system is its ability to operate in ionic media without common adverse effects, such as electrolytic breakdown and electrode corrosion. The resultant oscillating signals are multiplied with each other and filtered to obtain a sinusoidal signal whose frequency is determined by the capacitance offset between the parallel oscillators. The sine wave is then applied to a frequency-to-voltage converter that yields a DC voltage signal. The system exhibits a change of approximately 16 kHz for each pF offset in capacitance. Based on the performance of the circuitry used, these results translate into a capacitive offset measurement accuracy on the order of 10 fF. The signal that contains information of the measured comb drive displacement is a DC voltage. This purely electrical signal allows the current device to be considered as an abstract "black box" for the purposes of creating a controllable feedback system. One plausible use for this system topology is the development of a method for applying specific forces onto cells adhered to the comb drive actuator.

[1] T. L. Sounart, T. A. Michalske, and K. R. Zavadil, "Frequency-Dependent Electrostatic Actuation in Microfluidic MEMS," Journal of Microelectromechanical Systems, vol. 14, pp. 125-133, 2005.

[2] V. Mukundan and B. L. Pruitt, "Experimental Characterization of Frequency Dependent Electrostatic Actuator for Aqueous Media," presented at Solid State Sensors and Actuators, Hilton Head Island, 2006.

[3] D. J. Burns and H. F. Helbig, "A System for Automatic Electrical and Optical Characterization of Microelectromechanical Devices," Journal of Microelectromechanical Systems, vol. 8, pp. 473-482, 1999.

MN-TuP14 Assembly and Testing of Metal-based Microchannel Heat Exchange Devices, ***F.H. Mei***, *J. Jiang, W.J. Meng, P.R. Parida, S.V. Ekkad*, Louisiana State University

Since Tuckerman and Pease suggested the use of microfluidic devices for high heat flux removal in 1981, intense studies of heat transfer within microchannel devices at the mm to µm length scales have been carried out over the last two decades.[1,2] A majority of studies on microscale fluid flow and heat transfer have been conducted in Si-based microchannels because of the prevalence of Si microfabrication technology[3] and the lack of suitable microfabrication techniques for metal-based microchannel devices. Metal-based microchannel heat exchangers (MHEs) have important potential advantages over Si-based devices due to their higher thermal conductivities and better mechanical characteristics. Realization of metal-based microdevices requires the fabrication of metallic high-aspect-ratio microscale structures (HARMS). We have demonstrated successful HARMS replication in Pb,[4] Zn,[5] Al,[6] and Cu[7] from HARMS mold inserts by compression molding. To form any functional metal-based microdevice from such replicated metallic HARMS, proper assembly and packaging are required. Recently, we have successfully bonded Al-based and Cu-based HARMS by using eutectic bonding with Al-Ge composite thin films as intermediate layers, utilizing the Al-Ge eutectic with an eutectic temperature of 424°C.[8] We also evaluated the bond quality through measurements of the tensile bond strength in Al-based specimens.[9] In this paper, we report successful assembly of Cu-based microchannel devices and investigation of their heat transfer characteristics. Further studies on the bond quality of Cu-based specimens will be carried out, and results of assembly of metal-based MHE prototypes and testing of their overall heat transfer performance will be reported.

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[2] I. Mudawar, IEEE Trans. Components and Packaging Tech., 2001, 24, 122

[3] R. Chein, J. Chuang, Int. J. Thermal Sci., 2007, 46, 57

[4] D. M. Cao, W. J. Meng, K. W. Kelly, Microsyst. Technol., 2004, 10, 323

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[6] D. M. Cao, W. J. Meng, Microsyst. Technol., 2004, 10, 662

[7] D. M. Cao, J. Jiang, W. J. Meng, J. C. Jiang, W. Wang, Microsyst. Technol., 2007, 13, 503

[8] Fanghua Mei, J. Jiang, W. J. Meng, Microsyst. Technol., 2007, 13, 723

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Tribology

Room: 617 - Session TR1+MN-WeA

Surfaces and Interfaces in MEMS

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

1:40pm **TR1+MN-WeA1 Glassy-like Behavior of GaAs Nanomechanical Oscillators at Millikelvin Temperatures**, ***S.B. Shim****, S.W. Cho,* Seoul National University, Korea*, N. Kim, J. Kim,* Korea Research Institute of Standards and Science*, Y.D. Park*, Seoul National University, Korea

We report on the mechanical properties of single crystalline GaAs doubly-clamped beam resonator structures characterized by magnetomotive techniques in millikelvin temperatures. Clean nanomechanical GaAs resonators are realized from a lattice-matched GaAs/InGaP/GaAs heterostructures without plasma etching processing with typical quality (Q) factor of ~ 17,400 at 45 mK with resonance frequency of 15.816 MHz. We find dissipation (Q^{-1}) to have weak temperature dependence ($\sim T^{0.32}$) as compared to Si nanomechanical resonators of similar size ($\sim T^{0.36}$).[1] Furthermore, we find shift in the resonance frequency as function of temperature to be nontrivial with a crossover behavior (i.e. at low temperatures shift in the resonance frequency is positive with increasing temperature and at high temperature (T > ~1 K), negative). Such observations are similar to those observed in sound attenuation experiments in disordered glass systems.[2] We will also discuss other possible dissipation mechanisms as well as the effect of differing surface conditions and treatments.

[1] G. Zolfagharkhani et al., PRB 72, (2005).

[2] W.A. Phillips, Rep. Prog. Phys. 50, 1657 (1987).

2:00pm **TR1+MN-WeA2 MEMS Tribology in Extreme Environments[1]**, ***J. Krim***, North Carolina State Univ.*, M. Aggleton,* Univ. of California at Irvine, *C.J. Brown,* North Carolina State Univ.*, J.C. Burton,* Univ. of California at Irvine*, D.A. Hook,* North Carolina State Univ.*, J. Wenner,* Univ. of California at Irvine, *M.T. Dugger,* Sandia National Labs, *A. Morris,* WiSpry, Inc.*, J.E. Rutledge, P. Taborek*, Univ. of California at Irvine **INVITED**

Microelectromechanical systems, MEMS, have become a remarkably successful technology since the beginnings of MEMS development 30 to 40 years ago. However the overwhelming majority of MEMS are used near room temperature and atmospheric pressure. Consequently there is little empirical data to guide the design of MEMS for use in environments such as space where low pressures and cryogenic temperatures must be tolerated. In addition, it is well known that friction and wear severely constrain MEMS design. MEMS that have sliding contact between surfaces have shorter lifetimes and lower reliability than MEMS that do not. We have measured the characteristics of two microelectromechanical systems, namely a silicon sidewall tribometer and an RF MEMS direct contact switch, at cryogenic temperatures and in ambient gas environments below atmospheric pressure, and report on the tribological issues and possible solutions for operation of MEMS in such extreme environments.

[1] This work has been supported by EXTREME FRICTION AFOSR MURI #FA9550-04-1- 0381, and partially by Sandia National Laboratories. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE- AC04-94AL85000.

2:40pm **TR1+MN-WeA4 Performance of RF MEMS Switch Contacts at Cryogenic Temperatures**, ***C.J. Brown****, J. Krim,* North Carolina State University, *A.S. Morris III*, wiSpry, Inc.

A series of experiments were performed to characterize RF MEMS switch performance under variable pressure, atmospheric conditions and temperature. A vacuum system was constructed allowing for switch operation in cryogenic temperatures and pressures in the miliTorr range. Vacuum environments were chosen to limit stiction failures due to moisture; however the switches encountered bouncing problems at closure for low pressures. Helium and nitrogen were chosen as substitute atmospheres to lower stiction failure rates while circumventing switch bouncing issues. Contact resistance measurements were taken across a temperature range of 77 to 293 Kelvin using both gasses. Results showed no differences in contact resistance due to atmospheric conditions except at cryogenic temperatures. Contact resistance values were observed to be lower at cryogenic temperatures but are orders of magnitude higher than values predicted for constriction resistance in gold asperity contacts. Results obtained across the cryogenic temperature range support the conclusions of previously published work at high temperatures, which asserted changes in contact resistance were due mostly to the presence of thin films on the contacts.[1] Additionally, the data indicates these films are less mobile at cryogenic temperatures. Application of the asperity-heating model indicates contact voltages can applied which selectively disassociate films from the contact surface while not softening the gold asperity contacts. This research is funded by AFOSR MURI Grant No. FA9550-04-1-0381.

[1] B. Jensen, L. Chow, K. Huang, K. Saitou, J. Volakis and K. Kurabayashi, "Effect of nanoscale heating on electrical transport in RF MEMS switch contacts," J. Microelectromechanical Systems, vol. 14, no. 5, pp. 935-946, 2005.

3:00pm **TR1+MN-WeA5 Macro-, Micro-, and Nano-scale Lubrication using Alcohol Vapor: Implications to MEMS.**, *D.B. Asay,* Pennsylvania State University, *M.T. Dugger,* Sandia National Laboratories, ***S.H. Kim***, Pennsylvania State University

Friction, adhesion, and wear are dramatically affected by the environment in which surfaces come into contact. In the case of an alcohol vapor environment, the silicon surface reacts to form an alkoxide. Shearing these surfaces also produces higher weight oligomers. These molecules are continuously replenished in the contact region, drastically reducing wear and friction provided that the alcohol vapor pressure is near or above the vapor pressure required for monolayer coverage. At these conditions, the lubricating layer protects the silicon surfaces with little to no wear observed. Tribological properties are reported at the nanoscopic (AFM), mesoscopic (MEMS), and macroscopic (tribometer). In all cases, the vapor successfully lubricates and minimizes wear. In the case of MEMS sidewall friction, the lifetime of the device is radically increased.

4:00pm **TR1+MN-WeA8 MEMS Reliability in Harsh Environments**, ***R. Maboudian****, C. Carraro*, University of California at Berkeley **INVITED**

Many applications require sensors and actuators that can survive harsh environments, including high temperature and high relative humidity. This presentation will examine the behavior of polycrystalline silicon based micro-electromechanical systems in a variety of harsh environments. Then, the effectiveness of self-assembled monolayers and silicon carbide for enhanced MEMS reliability under these conditions will be discussed.

4:40pm **TR1+MN-WeA10 Water Vapor Effects on the Lubrication of Silicon MEMS by Alcohol Vapor**, ***M.T. Dugger***, Sandia National Laboratories, *D.B. Asay,* Pennsylvania State University*, J.A. Ohlhausen,* Sandia National Laboratories*, S.H. Kim*, Pennsylvania State University

Adhesion, friction and wear have been the greatest limitations to development of robust MicroElectromechanical Systems (MEMS) that rely on contact between surfaces. Chemisorbed monolayers such as alkyl and amino-silanes have been successful in creating initially-free structures, but have not demonstrated adequate long duration operation in sliding contacts, and recent studies suggest that they degrade with long term static exposure to water vapor in storage. A new lubrication approach has been demonstrated on silicon surfaces, which consists of alcohol molecules in the vapor phase that form a friction and wear reducing film dynamically, preferentially at contact locations. ToF-SIMS analysis of wear tracks from pin-on-disk experiments suggest formation of high molecular weight oligomers where the stress is highest. Experiments with MEMS tribometers result in a factor of at least 10^5 increase in operation life without failure, and no wear or debris formation. Practical device operation requires lubrication in the presence of some concentration of water vapor inside sealed packages. Friction experiments in environments containing 400 ppm alcohol and 1000 ppm H_2O show that lubrication by alcohol is inhibited at these relative concentrations.

[*] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

5:00pm **TR1+MN-WeA11 Monolayer Degradation and Sidewall Tribometer Studies of Vapor Phase Lubricants for MEMS**, ***D.A. Hook***, Sandia National Laboratiories, North Carolina State University, *S.J. Timpe,* Sandia National Laboratiories, University of California Berkeley, *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 as well as adhesion as devices are stored for long periods of time.[1] It has also been observed that the presence of these monolayers lowers the coefficient of friction in tribological contact. However these same contacts cause rapid degradation of these monolayers.[2] The loss of the monolayers contributes to an increase in the adhesive contact force and leads directly to device failure whether it be unpredictable operation of the device or complete cessation of movement. This study reports on degradation of (tridecafluoro-1,1,2,2-tetrahydrooctyl)tris(dimethylamino)-silane (FOTAS) monolayers on normal as well as sliding contacts in MEMS interfaces. The degradation of the monolayer in the normal loading case was probed by measuring the change in adhesive force of the contact over the course of 300,000 normal loading cycles. In the sliding experiment a decrease in oscillation amplitude was used to probe the status of the monolayer. The onset of monolayer degradation was observed in the normal contacting experiment after approximately 80,000 normal contacting cycles, while in the case of sliding degradation was observed almost instantaneously. Work funded by the AFOSR Extreme Friction MURI and Sandia National Labs MESA Project. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

[1] Srinivasan, U., Houston, M.R., Howe, R.T., Maboudian, R., "Alkyltrichlorosilane-Based Self-assembled Monolayer Films for Stiction Reduction in Silicon Micromachines", Journal of Microelectromechanical Systems 1998, 7, 252-260.

[2] DePalma, V., Tillman, N., "Friction and Wear of Self-Assembled Trichlorosilane Monolayer Films on Silicon", Langmuir 1989, 5, 868-872.

Tribology

Room: 617 - Session TR2+BI+NS+MN-ThM

Biolubrication, Sensing and Adhesion

Moderator: R.W. Carpick, University of Pennsylvania

8:00am **TR2+BI+NS+MN-ThM1 Resonant Nanomechanical Sensors for Protein Detection**, ***P.S. Waggoner***, *H.G. Craighead*, Cornell University

Micro- and nanoelectromechanical systems (MEMS and NEMS) are of interest in sensing applications due to their high sensitivity, label-free operation, and potential for mutilplexed detection on a single chip. Resonant MEMS and NEMS devices have demonstrated detection of masses on the order of femtograms or less, transducing changes in mass into changes in resonant frequency. Appropriate functionalization of the sensor surface allows specific, label free detection for analytes of choice. In this work we have detected prostate specific antigen (PSA), a biomarker used in the early detection of prostate cancer, as a model system using immunospecific functional layers present on the resonator surfaces. We have also studied the surface chemistry in order to minimize non-specific binding during sensor functionalization and use. In addition, sandwich assay techniques have been investigated for use in secondary mass tagging in order to enhance sensor response for dilute analytes while still preserving specificity.

8:20am **TR2+BI+NS+MN-ThM2 Correlation between XPS Data and Liquid Phase Self-Assembly of Alkanethiols**, ***H.M. Meyer III***, *T.G. Thundat, R. Desikan*, Oak Ridge National Laboratory, *R.G. White*, Thermo Fisher Scientific, UK

The relative ease in which self-assembled monolayers (SAM) can be applied have made them part of the standard tool set used for functionalizing and patterning surfaces at the nanoscale. Recently, alkanethiol-based SAMs have been used for immobilizing selective chemical receptors on the gold-coated side of a microcantilever. In this configuration, adsorption on the funtionalized side of the microcantilever generates nanomechanical motion (i.e. bending) which can be accurately sensed and used for detecting a variety of chemical and biological molecules. Achieving reliable selectivity and sensitivity depends primarily on the reproducible formation of the functional layer on one side of the microcantilever. We have recently investigated the effect of chain length on the packing density of the alkanethiols and, in turn, how this affects the sensitivity of the sensor. We present XPS characterization of microfabricated cantilevers functionalized with alkanethiol-based SAMs. The results are correlated with similar cantilevers that have been monitored during adsorption/immobilization of the same alkanethiols in the liquid phase. Previous liquid phase results indicated an unusual change in packing density of the thiol molecules as the chain length was increased and were difficult to correlate with preliminary XPS data, indicating major difference between how these films form in liquid phase vs. post-formation analysis in-vacuuo. These new results attempt to illuminate those differences. Research sponsored in part by grant NSF Award ID 0330410 in collaboration with Drs. V.P. Dravid, G. Shekhawat, and A. Majumdar and in part by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of FreedomCAR and Vehicle Technologies, as part of the High Temperature Materials Laboratory User Program, Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U.S. Department of Energy under contract number DE-AC05-00OR22725.

8:40am **TR2+BI+NS+MN-ThM3 Surface-Chemical Aspects of Implant Biotribology and Biomimetic Lubrication**, ***N.D. Spencer***, ETH Zurich, Switzerland **INVITED**

Following implantation of a hip prosthesis, the synovial membrane reforms and generates a liquid, pseudo synovial fluid (PSF), which is comparable in composition to synovial fluid itself. This complex solution of proteins, glycoproteins, polysaccharides and lipids is then responsible for the lubrication of the implanted joint. Surprisingly, the interactions of PSF components with the surfaces of joint materials, such as UHMWPE, CoCrMo, or alumina, remain remarkably unexplored. Not only is their relative propensity for adsorption unknown, but, once adsorbed, their efficacy for lubrication remains to be determined. We have investigated these issues by means of tribometry (on both macro and nano scales), combined with fluorescence microscopy, and determined, for example, that while albumin, the major component of PSF and the model protein used for implant testing, is strongly adsorbed on the surface of implants, it can be displaced during sliding by Î±-glycoprotein, a species present at much lower concentrations, but a much more effective lubricant. The fluorescence approach has also enabled us to detect transfer of polyethylene under conditions where it was not previously thought to occur. Natural lubricated surfaces tend to be soft and covered with species such as polysaccharides that have the ability to retain large amounts of water. We have attempted to imitate and understand this mechanism of lubrication by means of tethering various highly hydrated polymer chains in a brush-like structure onto both hard and soft surfaces and measuring their tribological properties. Interestingly, on soft surfaces, the effect of chain tethering seems to be to effectively eliminate boundary lubrication entirely. Fluid-film-like behavior is thought to persist to very low sliding speeds, by virtue of water retention in a thin layer between the brush-covered surfaces.

9:20am **TR2+BI+NS+MN-ThM5 In-situ Measurement of Boundary-Lubrication on Articular Cartilage Surfaces**, *J.M. Coles,* Duke University, *G.D. Jay,* Brown University, *F. Guilak,* ***S. Zauscher***, Duke University

The diarthrodial (synovial) joints of the body enable locomotion and activity while withstanding millions of cycles of loading at several times body weight. Recent macroscopic tribological experiments and biochemical analyses suggest that heavily glycosylated proteoglycans encoded by the gene proteoglycan 4 (Prg4), which are expressed by synoviocytes in synovial fluid as lubricin and by superficial zone chondrocytes of articular cartilage as surface zone protein (SZP), provide boundary lubrication in cartilage in the absence of interstitial fluid pressurization. Improved understanding of the role of Prg4 on the cartilage surface could thus provide important insight into the development of new therapies for joint diseases such as OA. The development of powerful new methods for the genetic manipulation of mice has led to the creation of modified murine strains in which specific gene inactivation (PRG4-/-) results in age-related joint degeneration that recapitulates the symptoms of OA. Here we show that atomic force microscopy with a colloidal probe is uniquely suited to study boundary lubrication of murine cartilage in-situ and in absence of other lubrication mechanisms. Here we report on friction measurements on the superficial surface layer of articular cartilage from the femoral head of Prg4 knockout and wildtype mice under boundary lubrication conditions. Furthermore, we report on the measured RMS roughness and Young’s modulus to quantify morphological and mechanical changes of the cartilage superficial zone induced by the absence of Prg4. Our measurements suggest that the absence of Prg4 leads to increased friction, as well as degradation of the mechanical and topographical properties of cartilage. We propose that, while lubricin plays a role as a boundary lubricant, its role in chondroprotection is equally, if not more, vital.

9:40am **TR2+BI+NS+MN-ThM6 Humidity Dependent Ordering of Water and its Effect on Adhesion and Friction between Silica Surfaces**, ***B.I. Kim***, *J. Bonander*, Boise State University

Adhesion and friction related to water are major problems limiting both the fabrication and long-term use of micro-machines. Adhesion and friction between two silica surfaces were measured as a function of separation distance using interfacial force microscope (IFM) for different relative humidity (RH) between 3% - 78%. The IFM provides force-distance curves without having the "snap-to-contact" problems associated with atomic force microscopy using voltage-controlled force feedback. The measured friction force-distance curves show that the friction force is oscillatory as the separation changes below thickness of water droplet. The oscillatory period is close to the mean diameter of a water molecule. The number of oscillation increased as the relative humidity increases up to RH 60% while it decreased with humidity above RH 60%. The origin of the oscillatory feature in the "interfacial" water may come from the "solid-liquid transition" between solid (ordering) and liquid (disordering). Strong correlation between the number of oscillation and the strength of the adhesion and friction indicates that the humidity dependent adhesion and friction may be attributed to the ordered structure of water molecules between two silica surfaces.

10:00am **TR2+BI+NS+MN-ThM7 Nanomechanical Properties of Arachidic Acid Langmuir Blodgett Films**, ***G. Oncins***, University of Barcelona, Spain, *J. Torrent-Burgues,* Universitat Politecnica de Catalunya, Spain, *F. Sanz*, Universitat de Barcelona and Center of Nanobioengineering of Catalonia (IBEC), Spain

Scanning Probe Microscopies development has given biophysics the possibility to deal with the interactions arisen in biological membranes from a nanomentric point of view, revealing that van der Waals, hydrogen bonding and electrostatic interactions play a crucial role in the membrane cohesion. Unfortunately, although interesting experimental conclusions have been reported in the past, these systems are complex and difficult to study.[1] In order to isolate the effect of the different cohesive interactions, Langmuir-Blodgett (LB) fatty acid monolayers provide excellent model systems because of the controlled area per molecule, linear hydrocarbon chain geometry, amphiphilic nature, high mechanical stability and the possibility to test solid and liquid phases at room temperature. The nanomechanical properties of arachidic acid LB films extracted at surface pressures of 1, 15 and 35 mN/m and deposited on mica are investigated by Atomic Force Microscopy, Force Spectroscopy and Friction Force Microscopy. It is experimentally demonstrated that the molecular ordering depends on the extraction pressure, while discrete molecular tilting angles of 50°, 34° and 22° are detected and identified as conformations that maximize van der Waals interactions between alkyl chains. The vertical force (F_v) needed to puncture the monolayer strongly depends on the molecular tilting angle, ranging from 13.07±3.24 nN at 1 mN/m to 22.94±5.49 nN at 35 mN/m. The friction force (F_f) measurements performed from low Fv until monolayer disruption reveal three friction regimes corresponding with a low F_f elastic monolayer deformation at low F_v, followed by a sharp increase in F_f due to a sudden plastic deformation of the monolayer. The last regime corresponds with the monolayer rupture and the contact between tip and substrate. Interestingly, as the extraction pressure increases, the friction coefficient of the monolayer reduces while the F_v needed to trigger the monolayer plastic deformation increases, facts that are discussed in terms of sample compactness and monolayer rupture mechanism.[2]

[1] Garcia-Manyes, S.; Oncins, G.; Sanz, F. Biophys. J. 2005, 89, 1812.

[2] Oncins, G.; Garcia-Manyes, S.; Sanz, F. Langmuir 2005, 21, 7373.

10:20am **TR2+BI+NS+MN-ThM8 Optimal Roughness for Minimal Adhesion**, *D.L. Liu,* Worcester Polytechnic Institute, *J. Martin,* Analog Devices Inc., ***N.A. Burnham***, Worcester Polytechnic Institute

Differing views on the effect of surface roughness on adhesion have appeared in the literature recently. 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] An experimental study showed that roughness can determine the adhesion in nanometer contacts and indicated that continuum mechanics still works down to nanometer length scales.[2] In this work, we use a single-asperity model to describe a smooth tip in contact with a rough surface and predict that there is an optimal size of asperity that will yield a minimum of adhesion. Experimentally, adhesive forces on silicon wafers with varying roughness from 0.2 nm to 39 nm were measured using AFM (atomic force microscope) cantilevers with varying tip radii ranging from 75 nm to 9.08 um. It is found that for all tip radii, the adhesion falls significantly for roughness greater than 1-2 nm and drops at higher roughness for larger tips. Minimum adhesion was observed as predicted in the 1-10 nm range and the optimal roughness for minimal adhesion increases as the tip radius increases, which is also consistent with our 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.

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

[2] E.J.Thoreson, J. Martin, N.A. Burnham, J. Colloid Interface Sci. 298, 94-101 (2006).

10:40am **TR2+BI+NS+MN-ThM9 A Tribological Study of Bound plus Mobile Lubricants for MEMS Application from the Nano- to the Macro-scale Regime**, ***B.P. Miller***, *M. Brukman,* North Carolina State University, *C.C. Baker,* Naval Research Laboratory, *R.J. Nemanich,* North Carolina State University, *K.J. Wahl,* Naval Research Laboratory, *J. Krim*, North Carolina State University

One possible solution to the in-use stiction problem in Micro-Electro-Mechanical systems (MEMS) is the introduction of a bound plus mobile lubricant combination.[1] For this system, one monolayer of the bound lubricant (perfluorodecyltrichlorosilane, PFTS) bonds to the surface of the device. The mobile lubricant (tricresyl phosphate, TCP) can replenish the layer after rubbing contacts wear it away, thus keeping the device free of contamination. Friction behavior of this lubricant combination was examined over three different regimes using a macroscopic tribometer, an AFM, and a quartz crystal microbalance (QCM). A comparative study of bound only versus bound plus mobile lubricants showed a decrease of the coefficient of friction with the addition of the mobile lubricant to the bound layer. Dynamic properties of the mobile film were characterized with the QCM. This work is funded by AFOSR Extreme Friction MURI Grant #FA9550-04-1-0381.

[1] W. Neeyakorn, M. Varma, C. Jaye, J. E. Burnette, S.M. Lee, R. J. Nemanich, C. Grant, J. Krim, Dynamics of Vapor-Phase Organophosphates on Silicon and OTS, Tribology Letters, in press.

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