Microelectromechanical Systems (MEMS)
Room 320 - Session MM-TuM

Development and Characterization of MEMS and NEMS Materials

Moderator: C.A. Zorman, Case Western Reserve University

8:20am **MM-TuM1 MEMS and NEMS from Chemical Vapor Deposited Nanodiamond Materials**, ***J.E. Butler,*** Naval Research Laboratory **INVITED**

Nanodiamond films grown by chemical vapor deposition exhibit a number of remarkable properties desirable for MEMS and NEMS. These include high Young's Modulus, thermal diffusivity, dielectric breakdown strength, mass density, secondary electron yields, fracture toughness, optical transparency, corrosion resistance, biological stability, and more. The nucleation, growth, and doping of these films on diverse substrate materials, including Si, poly Si, SiO2, and various metals, will be described along with various methods of processing into structures and devices.

9:00am **MM-TuM3 Engineering the Surface Properties of Ultrananocrystalline Diamond for High-Performance MEMS Devices**, ***A.V. Sumant,*** *D.S. Grierson,* University of Wisconsin, Madison*; J.E. Gerbi, J.A. Carlisle, O. Auciello,* Argonne National Laboratory*; R.W. Carpick,* University of Wisconsin, Madison

Characterization of MEMS and NEMS devices at small length scales is extremely important in order to understand the factors that dictate the performance of these devices. Ultrananocrystalline diamond (UNCD), in particular, has exceptional physical, electrical, chemical and tribological properties (nearly equivalent to those of single crystal diamond). UNCD is being considered as one of the most promising materials for the fabrication of high performance MEMS devices. However, little is known about the surface chemistry of this material, and how such surface chemistry will affect the UNCD performance, particularly in case of rolling or sliding contacts at both the micro and nano level. We have carried out detailed, systematic studies of UNCD thin films by various analytical techniques including Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and atomic force microscopy (AFM) to understand the chemical nature, phase, and microstructure of the UNCD surface. We have found that there is a significant difference in the structural and chemical properties between the as-grown UNCD top surface and the underside of the film as revealed after etching away the substrate. Characterizing the underside of the film is particularly important because in most cases (e.g. micro-engines and cantilever based switches), the underside of the film makes contact with the underlying surface below. We will discuss how these properties are influenced by various aspects of the microwave PECVD growth process, including the initial nucleation pre-treatment and the gas chemistry used during growth, and how one can engineer the surface by tuning these growth parameters. Finally, we will discuss how such changes may affect UNCD performance at MEMS length scales.

9:20am **MM-TuM4 AlN-based MEMS and NEMS Resonator Devices**, ***A.E. Wickenden,*** *L.J. Currano, M. Dubey,* U.S. Army Research Laboratory*; S. Hullavarad, R.D. Vispute,* University of Maryland, College Park

Electromechanical resonator devices using piezoelectric aluminum nitride (AlN) actuation are being developed for RF filters operating in the MHz-GHz frequency range. Composite structures are required for these micro- and nanoelectromechanical systems (MEMS, NEMS) devices, which include the piezoelectric film, metal electrode layers, and a flexural layer. AlN film quality is very dependant on the growth technique, and differences in crystallinity impact the subsequent piezoelectric response of the film. We have demonstrated the pulsed laser deposition (PLD) of highly oriented AlN thin films on Pt-terminated composite MEMS/NEMS structures. Characterization by X-ray diffraction demonstrates a FWHM of 0.2°, a ten-fold improvement over sputtered AlN films typically used in this application. Pattern transfer techniques have been developed for these composite device structures at both micro- and nano-scale. Fully released AlN MEMS beam resonator structures have been fabricated and tested. These devices demonstrate strong electromechanical response, with a 500 nm deflection observed in a fixed-fixed beam with a resonant frequency of 250 kHz. Device dimensions for resonant frequencies near 1 GHz are predicted to scale to near one micron in length and hundreds of nanometers in cross-sectional area. Free-standing 200nm wide x 150nm thick x 10µm long AlN beams with aligned metal electrodes have been demonstrated, using direct-write electron beam lithographic patterning techniques. We will present detailed harmonic and modeshape analysis of AlN MEMS resonators in the 0.15-15 MHz frequency spectrum, and highlight current results in the development of AlN NEMS resonators.

9:40am **MM-TuM5 Anchor Optimization for Quality Factor Improvement in Microresonators**, ***L.J. Currano,*** *A.E. Wickenden, M. Dubey,* U.S. Army Research Laboratory

Arrays of microresonators are of considerable interest for low-cost, high precision RF filters. The quality factor (Q) of a resonator is the figure of merit which determines the amount of signal lost from input to output as well as the slope of the cutoff of a bandpass filter. Some of the factors that degrade the quality factor in mechanical resonators are geometry and material properties, thermoelastic noise, and the transduction mechanism.@footnote 1@ Several discussions of the noise and dissipation mechanisms in microresonators have been published.@footnote 2@ One of the most important loss mechanisms is the transmission of mechanical strain energy to the substrate. The magnitude of strain energy transmitted to the substrate can be manipulated by changing the geometry of the interface between device and substrate. The quality factor for similar PZT-based clamped-clamped beam resonator devices has been found to double by changing the geometry of the anchor slightly. New models are necessary for reducing mechanical losses in the simplest resonator structure, a doubly clamped beam. Finite element analysis provides a vehicle for examining the losses due to transmission of strain energy from the resonator into the substrate and a window into some of the design methods that can be used to minimize mechanical losses. A finite element model which calculates the strain energy transmitted to the substrate in a clamped-clamped resonator beam has been devised, and the results show that the losses steadily decrease with anchor width. Results from the model along with results from electrical testing of PZT resonator devices will be presented. @FootnoteText@@footnote 1@A. N. Cleland and M. L. Roukes, "Noise processes in nanomechanical resonators," Journal of Applied Physics, vol. 92 pp 2758, 2002.@footnote 2@J. R. Vig, "Noise in microelectromechanical systems resonators," IEEE Trans. on Ultrasonics, Ferroelectrics, and Frequency Control, vol. 46 pp. 1558, 1999.

10:00am **MM-TuM6 Chemical Control of Mechanical Energy Dissipation in Micromechanical Silicon Resonators**, ***Y. Wang,*** *J.A. Henry, M.A. Hines,* Cornell University

Why are we unable to predict the dynamical performance of nanoscale devices from the well-known properties of macroscopic materials? For example, the quality (or Q) of micromechanical resonators plummets as the size of the device is reduced. We will show that the role of surface effects on energy loss cannot be ignored at this length scale. To investigate the role of surface dissipation, we have fabricated silicon torsional resonators with predominantly Si(111) faces. The resonators' surfaces are then chemically modified and characterized by infrared spectroscopy. Resonators terminated by an atomic layer of hydrogen have the lowest energy loss but the quality of the resonator decreases with time even in high vacuum (10@super -8@ Torr). Quantitative analysis of the time-dependent frequency shift suggests that the increased losses observed in vacuum are correlated with chemical adsorption. The quantitative effects of a variety of adsorbates will also be discussed. When the H-monolayer is replaced by one type of self-assembled monolayer (SAM), a small decrease in initial quality is observed; however, SAMs-terminated devices are much more stable with time. This stability is attributed, in part, to increased chemical resistance; however, aggressively oxidizing environments still lead to performance degradation. A second type of SAM leads to much higher energy losses. The chemical origins of this difference will be discussed.

10:20am **MM-TuM7 Vapor-Phase Lubricants: Nanometer-scale Lubrication Mechanisms and Uptake on Silicon**, ***W. Neeyakorn,*** *M.R. Varma, J. Krim,* North Carolina State University

The concept of lubricating high temperature bearing surfaces with organic vapors which react with a surface to form a solid lubricating film has existed for at least forty years, with substantial efforts beginning in the 1980's and continuing to the present day. While vapor-phase lubricants have primarily been studied within the context of macroscopic system performance, they may well prove to be of critical importance to tribological performance in sub-micron mechanical systems as well. This is because the vapor phase may ultimately prove to be the most effective, if not only, means to deliver and/or replenish a lubricant that can withstand a variety of extreme environmental conditions that a MEMS device is likely to encounter. In order to investigate the viability of vapor-phase lubrication for MEMS applications, we have studied molecular scale tribological properties and gas uptake rates for four known organophosphate

lubricants in controlled environments on silicon and gold substrates. The first study involves Quartz Crystal Microweighing investigations of the uptake rates of lubricant vapors from the vapor phase in vacuum conditions. With the intent of modelling actual MEMS contacts, we have also constructed a simple nanomechanical system consisting of a Scanning Tunneling Microscope tip dragging on the surface of a Quartz Crystal Microbalance electrode. This system allows us to monitor lubricant performance in realistic sliding conditions of up to 2 m/s. Finally, work is in progress to study the effect of these vapor-phase lubricants on actual MEMS devices with contacting silicon surfaces.@footnote 1@ @FootnoteText@@footnote 1@ Work supported by NSF and AFOSR.

10:40am **MM-TuM8 Can We Predict Friction and Wear in MEMS?**, ***E.E. Flater,*** University of Wisconsin - Madison*; A.D. Corwin, M.P. DeBoer,* Sandia National Laboratories*; C.K. Bora, M.E. Plesha, R.W. Carpick,* University of Wisconsin - Madison

The design of reliable MEMS devices that involve sliding or rolling surfaces requires a predictive capability for friction and wear. We will describe our efforts to predict friction in MEMS by connecting single asperity friction measurements via atomic force microscopy (AFM) with multi-asperity friction measurements in a newly-developed MEMS friction test device through the use of analytical models of contact between rough surfaces. We will show that AFM resolves critical roughness features of MEMS surfaces from the nm-to-Âµm scale. From this information, we derive surface roughness parameters that are used as inputs to predict the interfacial mechanics using generalized models based on the Greenwood-Williamson approach and fractal surface models, respectively. We will discuss the validation of these approaches with reference to single-asperity AFM experiments and multi-asperity MEMS friction test device experiments.

11:00am **MM-TuM9 Nanomechanical Characterization of Digital Micromirror Devices**, ***G. Wei,*** *B. Bhushan,* The Ohio State University*; J. Jacobs,* Texas Instruments, Inc.

The Digital Micromirror Device (DMD) lies at the heart of Texas Instruments' Digital Light Processing@super TM@ (DLP@super TM@) technology, enabling the next generation of bright, lightweight projection displays. The DMD comprises a surface-micromachined array of up to 2.07 million mirrors fabricated on top of an SRAM array. The nanomechanical properties of the thin-film structures formed in the manufacturing process are important to the performance of the DMD. In this paper, the nanomechanical characterization of various materials used in the manufacture of the DMD has been performed using a nanoindentation technique. Properties including Young's modulus, hardness, scratch resistance, fatigue, and fracture toughness of the corresponding materials have been measured and analyzed. The impact of these properties on mirror performance is discussed.

11:20am **MM-TuM10 Measurement of Mechanical Properties of Silicon Nitride Thin Film at Cryogenic Temperatures**, ***W. Chuang,*** *T. Luger,* University of Maryland, College Park*; R. Fettig,* NASA Goddard Space Flight Center*; R. Ghodssi,* University of Maryland, College Park

Mechanical properties of MEMS materials at cryogenic temperatures are investigated to extend MEMS sensors and actuators into space and low temperature applications. Two-dimensional micro-shutter arrays, made in silicon nitride thin film, are being developed at NASA Goddard Space Flight Center (GSFC) for use in the James Webb Space Telescope (JWST). Reliability and exact mechanical properties of silicon nitride thin film at cryogenic temperatures are crucial in the development of the JWST. We have developed and installed a measurement setup inside a focused ion beam (FIB) system, which can provide scanning electron microscopy (SEM) and mask-less ion milling, to measure the mechanical properties of MEMS materials from room to cryogenic temperatures. A variety of low-stress silicon nitride T-shape cantilevers suspended on silicon micromachined v-grooves are fabricated as test devices. The resonant frequency method is used in experiments to minimize the required calibration in the measurement setup at different temperature ranges. A lead-zirconate-titanate (PZT) translator and a silicon diode are utilized as the actuator and temperature sensor in the measurement setup, respectively. Experiments are performed to measure resonant frequency, damping, coefficient of thermal expansion, Young's modulus, fracture strength and fatigue properties of the test devices from room to cryogenic temperatures. The measured resonant frequencies are varied from 15.81 kHz at 25.5 K to 14.61 kHz at 298 K (room temperature). The higher resonant frequency is consistent with the expected increased Young's modulus of silicon nitride thin film at cryogenic temperatures. The preliminary measurement results, detailed fabrication process and configuration of the measurement setup will be presented.

11:40am **MM-TuM11 Pointwise Strain Mapping a Multilayer MEMS Mirror Using Synchrotron Radiation**, ***Y.N. Picard,*** *S.M. Yalisove, E. Dufresne, C. Cionca, J. Guzman, R. Clarke,* University of Michigan, Ann Arbor*; D. Walko,* Argonne National Laboratory*; O.B. Spahn, D.P. Adams,* Sandia National Laboratories

Precise control over surface curvature of micromirror devices is critical for developing communications and power delivery applications. Furthermore, the control of this curvature must be maintained over time and in a range of operating conditions. Curvature control ultimately requires understanding of how stress in reflective coatings and thermal-stress compensation layers affect the ultimate performance of a variety of micromirror designs (different geometries, thickness, clamping arrangements, etc). Highly localized, non-destructive strain measurement techniques are required to assess variations in stress across and through micromirror coating layers on the actual device. We present results of strain mapping across a metal-coated polysilicon micromirror using a micron-sized x-ray beam at the Advanced Photon Source. Prior to x-ray analysis, a high reflectivity, low stress film of 10 nm Ti/150 nm Au was deposited by DC planar magnetron sputtering on a 2.25 micron thick, 500 micron diameter polysilicon mirror that had been etch-released prior to film deposition. A 10keV x-ray beam was focused down to a 5.3x12.8 micron spot size using two bendable Kirkpatrick-Baez mirrors and then used for point-by-point detection of Au and Si diffraction peaks. The peak positions were then measured and used to determine strain in the respective thin film after comparison to a standard powder sample. Because the freestanding micromirror was still clamped to the substrate, variations in strain were anticipated and indeed detected. Results of measured in-plane and out-of-plane strain for both the Au film and the polysilicon mirror will be presented, where an up to 23% variation in strain is detected from the center to the constrained edges of the micromirror. We also discuss strain resolution by this method and estimate that 3-5 MPa of stress can be resolved point-to-point within each material layer.

Microelectromechanical Systems (MEMS)
Room 320 - Session MM-TuA

Fabrication and Characterization of MEMS Devices

Moderator: C.B. Freidhoff, Northrop Grumman

2:00pm **MM-TuA1 Development of a Deep Phase Fresnel Lens in Silicon**, ***B. Morgan,*** *C.M. Waits,* University of Maryland, College Park*; J. Krizmanic,* NASA - Goddard Spaceflight Center*; R. Ghodssi,* University of Maryland, College Park

Astronomical observations at Gamma and hard X-ray energies are presently hindered by instruments with low sensitivity and poor angular resolution. Fresnel Zone Plates and their derivatives, could achieve higher sensitivity and greater angular resolution.@footnote 1@ For ground testing of a Phase Fresnel Lens (PFL), lateral dimensions of each lens feature must be on the order of 10 µm, while vertical dimensions must be >20 µm, both a natural fit for MEMS processing. Silicon, the standard material used in MEMS, has low absorption of Gamma and X-ray radiation, making it a good material choice for the fabrication of a PFL for ground testing. Gray-scale technology was selected as the fabrication method for developing such a lens because of two main advantages: (1) the multiple heights required for increased efficiency may be fabricated without alignment, and (2) Deep Reactive Ion Etching (DRIE) with precise selectivity control enables the fabrication of deep (>20 µm) silicon lenses. Multiple PFL's, with diameter >1.6mm and varying heights, have been successfully fabricated in silicon. The optimization of gray-scale lithography processing for large-scale structures was realized through the use of a custom calibration mask. Advanced gray-scale optical mask design allows the fabrication of small gray level geometries over a large area, enabling precise profile control to maximize lens efficiency. Depending on target photon energy, etch depths required to produce the appropriate phase shift in silicon have been between 20 and 100 µm. Highly accurate vertical dimension control is also necessary to ensure the proper interference pattern at the lens focus. Therefore, PFL height was controlled by finely tuning etch selectivity during DRIE, which adjusts the scaling factor between photoresist and silicon, and provides the appropriate PFL profile in silicon. @FootnoteText@ @footnote 1@G.K. Skinner, Astronomy & Astrophysics, v.375, no.2, 2001, p.691-700.

2:20pm **MM-TuA2 Fabrication and Characterization of a Capacitive Micromachined Shunt Switch**, ***S.L. Firebaugh,*** United States Naval Academy*; H.K. Charles, Jr., R.L. Edwards, A.C. Keeney, S.F. Wilderson,* Johns Hopkins University

Microelectromechanical switches offer many advantages over solid-state devices, including greater linearity, increased isolation and lower insertion loss.@footnote 1-5@ One disadvantage of such switches is that they require high actuation voltages (20-100 V), leading to problems with dielectric charging and system integration. Furthermore, when reducing the actuation voltage one must consider the dependence of power handling capability on actuation voltage.@footnote 6@ This paper describes the design, fabrication and testing of a shunt switch@footnote 3,4@ based on a bridge suspended over a coplanar waveguide. When a sufficient DC voltage is applied the bridge pulls down towards the signal line, creating a high-frequency short circuit and causing the signal to reflect back towards the source, blocking transmission. The switch is used as a test vehicle to explore the effects of bridge shape on performance, as well as to investigate the limits of micromachined switches. A common problem for MEMS switch developers is obtaining equipment to test device characteristics such as power handling limits on a wafer probe station. In this work, standard microwave connectors and equipment are facilitated by a custom test fixture. The pull-in effect is studied in detail, as well as methods for switching the device while avoiding dielectric charging effects and maintaining reasonable power handling levels. @FootnoteText@ @footnote 1@ C.T.C. Nguyen et al., Proc. IEEE, vol. 86, no. 8, pp. 1756-1768, August 1998.@footnote 2@ J.J. Yao, J. Micromech. Microeng., vol. 10, pp. R9-R38, 2000.@footnote 3@ Z.J. Yao et al., IEEE J. Microelectromech. Syst., vol. 8, no. 2, pp. 129-134, 1999. @footnote 4@ J.B. Muldavin and G.M.Rebeiz, IEEE Trans. Microwave Theory and Tech., vol. 48, no. 6, pp. 1045-1052, June 2000.@footnote 5@ D. Hyman et al., Electron. Lett., vol. 35, no. 3, pp. 224-226, February 1999.@footnote 6@ B. Pillans et al., 2002 IEEE MTT-S Int. Microwave Symp. Dig., pp. 329-332.

2:40pm **MM-TuA3 Design and Process Integration of an Electric Induction Micromotor**, ***C. Livermore,*** *J.L. Steyn,* Massachusetts Institute of Technology*; J.U. Yoon, A. Forte,* MIT Lincoln Laboratory*; R. Khanna,* Massachusetts Institute of Technology*; T. Lyszczarz,* MIT Lincoln Laboratory*; S.D. Umans, J.H. Lang,* Massachusetts Institute of Technology

INVITED

We present the development of a millimeter-scale electric induction machine designed to output Watt-level power for portable power applications. The micromotor comprises a stack of five micromachined silicon wafers. A 4 mm spinning silicon rotor disk is encapsulated within the stack; facing the spinning rotor is a six-phase electric stator. Mechanical-electrical power conversion is accomplished by the interaction of the stator potential with induced charges on the rotor. To operate at high power levels, the micromotor must operate under extreme conditions: high speed rotation (near one million rpm), high voltages (300 V across 3 µm to 4 µm gaps), and high electric frequencies (about 1.5 MHz). These requirements in turn place stringent requirements on the device design and fabrication flow: low electric losses, excellent resistance to electric breakdown, and essentially leak-free wafer bonds among the five silicon wafers. This presentation describes the approaches that are used to meet these requirements simultaneously in a complete, functional device. A thick oxide liftoff process is used to embed islands of oxide in the silicon substrate under the electric elements to reduce stray capacitance and electric losses. The island structure also minimizes overall bow from the stressed films, making wafer bonding possible. The stator's two-level interconnected electric elements are made of platinum and fabricated by a liftoff process. The platinum electrodes and interconnects reduce line resistance, minimize ohmic losses, and provide a smooth, break-down resistant line shape.@footnote 1@ @FootnoteText@ @footnote 1@ The Lincoln Laboratory portion of this work was sponsored by the Defense Advanced Research Projects Agency. Opinions, interpretations, conclusions, and recommendations are those of the authors and are not necessarily endorsed by the Department of Defense.

3:20pm **MM-TuA5 Microfabrication of a Pressure Sensor Array Using 3D Integration Technology**, *M. Khbeis,* Laboratory for Physical Sciences*; **X. Tan**,* University of Maryland*; G. Metze,* Laboratory for Physical Sciences*; R. Ghodssi,* University of Maryland

A novel microfabrication approach that enables successful integration of a piezoresistive pressure sensor array on an airfoil for detection of micro-scale turbulent vortices is presented. These sensor arrays will be used to study the dynamics of turbulent air flow with the ultimate goal of reducing drag on aircraft. Minimization of surface undulations on the sensor is required to avoid generating additional air turbulence. Therefore it is essential to implement an enabling fabrication approach that eliminates the dependence on topside electrical connections. We have developed a 3D integrative process to provide backside interconnections, while maintaining the specified 200µm sensor element pitch. This 3D fabrication effort incorporates several advanced process technologies including: low temperature SiO@sub 2@-to-Si wafer bonding (<250°C), bulk wafer thinning (to 20µm), high-aspect ratio (HAR 20:1) Si and SiO@sub 2@ etching using m=0 resonant induction (M0RI), and HAR (10-15:1) metallization using high pressure Aluminum reflow. This integrative process will facilitate vertical stacking of multiple device components on different layers, allowing the integration of MEMS and microelectronic devices for the realization of Small Smart Systems (SSS). Process developments and preliminary experimental results are presented.

3:40pm **MM-TuA6 Simulation of Field Emission-based Pressure Sensors**, ***A.M. Nair,*** *N. Badi, A. Bensaoula,* University of Houston

This paper reports on simulating the moving part of a field emission-based pressure sensor. The device is comprised of a membrane made of silicon or other stiffer materials acting as the anode of a device comprised of a fixed flat cold cathode emitter. This is achieved by modeling the deflection and mechanical stress of a diaphragm of varying geometry under selected input pressures. Realistic field emission characteristics from our boron nitride and carbon nitride cold cathodes@footnote 1@ were used to model the current density distribution in the deflected diaphragm. The total current output was achieved by integrating the current density over the entire diaphragm area as a function of membrane bending due to external pressure. Results show that simple and reliable field emission devices can be designed to yield extremely sensitive pressure sensors but their characteristics are critically dependent on the specific geometry. Simulation data will be presented for devices with geometries similar to those being fabricated in our laboratory. @FootnoteText@@footnote

1@N. Badi, A. Tempez, D. Starikov, A. Bensaoula, V.P.Ageev, A. Karabutov, M.V. Ugarov, V. Frolov, E. Loubnin, K. Waters and A. Shultz, "Field Emission from as-grown and Surface Modified BN and CN Thin Films" J. Vac. Soc.Technol. A 17, 1191 (1999). Acknowledgment: This material is based upon work supported by the National Science Foundation under Grant No. 0010100

4:00pm **MM-TuA7 Nanotribological Characterization of Digital Micromirror Devices using Atomic Force Microscopy**, ***H. Liu***, *B. Bhushan,* Ohio State University

Texas Instruments' Digital Micromirror Device (DMD) comprises an array of fast digital micromirrors, monolithically integrated onto and controlled by an underlying silicon memory chip.@footnote 1@ It is one of the few success stories in the emerging field of microelectromechanical systems (MEMS). In this study, atomic force microscopy (AFM) has been used to characterize the elements of the mirror structure of the DMD. AFM images of the mirror, hinge and yoke, and metal arrays are characterized. An AFM methodology was developed to identify and remove mirrors of interest. The surface roughness and adhesion properties of contacting surfaces were extensively studied. The influence of relative humidity and temperature on the behavior of the DMD was also investigated. Potential mechanisms for stiction accrual@footnote 2@ are discussed in light of the findings. @FootnoteText@ @footnote 1@L.J. Hornbeck, MRS Bulletin, 26, 325(2001) @footnote 2@B. Bhushan, (ed.), Tribology Issues and Opportunities in MEMS, (Kluwer Academic, Dordrecht, Netherlands, 1998).

4:20pm **MM-TuA8 PZT Dry Etching using ICP Etcher for MEMS Devices**, ***M. Dubey***, *R.G. Polcawich, E. Zakar, J. Pulskamp, A.E. Wickenden, L.J. Currano,* U.S. Army Research Laboratory

Piezoelectric Lead Zirconate Titanate (PZT) thin films deposited on platinized silicon substrates were reactively ion etched using an inductively coupled plasma (ICP) etching system. Etch rates for PZT, Pt, and silicon dioxide were determined from room temperature to 90 °C using Ar, He, Cl@sub 2@, CF@sub 4@, C@sub 2@H@sub 4@, C@sub 4@F@sub 8@, and SF@sub 6@ gases at RF powers from 500 to 1400 W. Measured etch rate for PZT varied from 500 to 2600 Å/min, for Pt from 940 to 1750 Å/min, and for oxide from 50 to 300 Å/min. Experimental results will be presented on selectivity and etched profile of PZT, Pt, and silicon dioxide films at different operating pressures and bias voltages. MEMS (microelectromechanical systems) test structures fabricated using ICP etchers will also be described.

4:40pm **MM-TuA9 Integration of Silicon Anisotropic Wet Etching and BCB Processes**, ***N. Ghalichechian***, *A. Modafe, R. Ghodssi,* University of Maryland, College Park

We have developed a fabrication process for integration of Benzocyclobutene (BCB) dielectric film and anisotropically etched deep silicon v-grooves in potassium hydroxide (KOH) solution for development of MEMS actuators such as an electrostatic micromotor supported on microball bearings. BCB is a low dielectric constant polymer (k=2.65) with several advantages over silicon dioxide for dielectric insulation such as simple deposition process, low-temperature cure, and low residual stress that are desired in a reliable, efficient electrostatic micromotor. KOH solution is used for fabrication of silicon v-grooves that form a bearing structure to house the micro-balls. We have used gold (Au) film as the etch mask and chromium (Cr) film as the adhesion layer that bonds the Au to the underlying BCB. It was initially observed that the metal films lost adhesion and no longer protected the BCB film during long KOH etching (7 hours in 20 %w solution) process. A fabrication process is developed to improve the adhesion between the BCB and the metal films during the silicon anisotropic bulk micromachining using KOH. This is achieved through several unit process experiments to investigate (a) the effect of cure temperature (210-250 °C), (b) surface treatment of BCB, (c) adhesion improvement at the interface between thin film metals and BCB by applying an adhesion promoter, AP3000, prior to metallization, and (d) the required Cr and Au thickness that results in a robust adhesion to BCB and reliable etch mask for KOH. The optimized experimental results in this work have enabled successful and repeatable fabrication of deep v-grooves (280 µm wide, 220 µm deep) in silicon with BCB (1 µm thick) and Cr/Au (20/500 nm thick) multilayer etch mask. The detailed process parameters and experimental results for this integrative process technology will be presented.

5:00pm **MM-TuA10 Optical Characterization of Poly-Dimethyl Siloxane (PDMS) during Inductively Coupled Plasma Processing for Implementation in a PDMS-based Photonic Crystal***, *E.A. Joseph, L.J. Overzet, M.J. Goeckner,* ***D.S. Park***, *M. Tinker, J.B. Lee,* University of Texas - Dallas

Poly-dimethyl siloxane (PDMS) is a silicone elastomer quickly gaining popularity in MEMS and EUV lithography markets. Material etch rates and plasma susceptibility however are not yet fully understood and may significantly effect optical MEMS device performance. In this work, PDMS etch susceptibility is studied in oxygen and CF4 inductively coupled plasma mixtures with in-situ spectroscopic ellipsometry. Initial etch rates as high as 40 µm/hr using a 3:1 mixture of CF4:O2, nearly double that of optimized conventional RIE@footnote 1@ have been obtained, while pure oxygen plasma exposure has been found to increase the refractive index by 7%. A more detailed study of etch rate and dielectric constant dependence on gas flow will be presented along with the implications of using PDMS in a photonic crystal MEMS device. @FootnoteText@ *This work is supported by grants from NSF / DOE, CTS-0079783 & CTS-0078669 and NSF ECS-0296018.@footnote 1@J. Garra, T. Long, J. Currie, Schneider, R. White, and M. Paranjape, J. Vac. Sci. Technol. A 20(3), May/June 2002.

Microelectromechanical Systems (MEMS)
Room Hall A-C - Session MM-TuP

Poster Session

MM-TuP1 Electrostatic Actuation in BioMEMS, ***T.L. Sounart,*** *T.A. Michalske,* Sandia National Laboratories

Electrostatic MEMS actuators exhibit fast response times and are easily integrated into microsystems because they can be fabricated with standard silicon IC micromachining processes. Although electrostatic actuators have been used extensively in "dry" MEMS, they have received little attention in microfluidic bioMEMS, despite the added advantage of 80 times the energy density in water relative to that in air. This is probably because electrostatic actuation in most liquid media presents new challenges such as electrolytic gas generation, anodic oxidation, and electrode polarization. Electrolysis is avoided completely at O(1) V electrode potentials, and although such potentials are also too low for actuation in air, they are sufficient to actuate many devices in water. Unfortunately, at equilibrium ionic solutes in conducting fluids screen the electrode potential (electrode polarization) and disable the actuator. We are currently investigating electrostatically-driven biological sensors and other bioMEMS devices by employing ac drive signals to prevent charge screening, which enables electrostatic actuation in many liquids, at potentials low enough to avoid electrochemistry. Here we measure the frequency response of an interdigitated silicon comb drive actuator in liquids spanning a decade of dielectric permittivities and four decades of conductivity, and present a simple theory that predicts the characteristic actuation frequency. The analysis demonstrates the importance of the native oxide on silicon actuator response, and suggests that the actuation frequency can be shifted by controlling the thickness of the oxide. For native silicon devices, actuation is initiated at frequencies less than 10 MHz, in electrolytes of ionic strength up to 100 mmol/L, and thus electrostatic actuation is feasible in many bioMEMS and other microfluidic applications.

MM-TuP2 Capillary Electrophoresis On-chip: Glass and Polymeric Materials for Cell Fabrication, ***Y. Mourzina,*** *A. Offenhaeusser,* Research Centre Juelich, Germany

The advances of bioanalytical chemistry and biotechnology stimulated interest in on-chip integrated microfluidic systems and analytical techniques for accurate, precise and high-effective analysis of proteins. Capillary electrophoresis on chip is rapidly developing field by learning on fabrication technologies in the field of MEMS. In the present work, two approaches to realize 3-D microstructures in glass wafers or polymeric materials for on-chip capillary electrophoretic separation of proteins are presented. The microchannel configurations in Pyrex glass wafers have been realized by means of photolithography, thin film deposition and etching steps. The dimensions of the channels are 6 to 16 µm depth and 40 to 120 µm width. Different processing sequences are compared. The structures have been visualized by means of REM to observe the profile and the edge roughness. Special attention has been paid to the optimization of the deposition of the metal thin films as sacrificial layer for glass etching, and to the influence of the composition of the etch solution on glass etch velocity, undercut phenomenon and the quality of the structures. Soft lithography is presented as an alternative approach to realize microfluidic channels in polymeric material. The master has been fabricated of the high aspect ratio SU 8 photoresist on Si wafers. Depending on the type of photoresist and the parameters of processing, the masters with different height of the structures (10 to 25 µm) have been obtained. The master has been used for microreplication in polymer PDMS. The dimensions of the separation channels of the polymeric devices are 10 to 25 µm depth and 20 to 80 µm width. Fluorescent microscopy was used to visualize the microchannels. To validate the performance of on-chip electrophoresis, the results of the separation of phosphoproteins in the prepared devices will be compared with the resolution of the conventional gel electrophoresis.

MM-TuP3 Stiction Measurements Made with an Atomic Force Microscope on Test Structures Mounted with Various Die-Attach Materials, ***E.J. Thoreson,*** Worcester Polytechnic Institute*; J. Martin,* Analog Devices*; N.A. Burnham,* Worcester Polytechnic Institute

An atomic force microscope (AFM) was used to determine the stiction between silicon oxide tips and silicon oxide substrates coated with a few angstroms of phenylsiloxane. The substrates were mounted in their usual packaging with three different types of die-attach materials, which were silicone, polyimide silicone, and silver glass. There was also a control group in which the substrates were not attached. The packages were opened and an AFM determined the adhesive force between the AFM tip and the substrate in force spectroscopy mode. A preliminary data set showed that the adhesive force normalized to the tip radius was respectively twice and four times as big for the polyimide silicone and silver glass as for the control group and silicone, the latter two being close in value. The percent variations in the measurements were 70% to 80% percent for the control group and silicone, 150% for polyimide silicone, and 25% for silver glass. Further work will verify these initial results and also study the dependence of adhesive force upon the tip radius.

MM-TuP4 Vacuum Encapsulation of Micron-Sized Vacuum Field Emission Triodes, ***S.J. Randolph,*** University of Tennessee, Knoxville*; M.A. Guillorn,* University of Tennessee, Knoxville and Oak Ridge National Lab*; M.D. Hale, P.D. Rack,* University of Tennessee, Knoxville

In recent years, carbon nanotubes have shown promise for use as stable field emitting elements in gated cathode devices. Vacuum conditions are ideal for the operation of field emission triodes, however, issues of practicality require that they be able to function outside the confines of a vacuum chamber. For this reason, a microfabrication technique has been developed for encapsulating a field emission triode in a micron-sized, vacuum-sealed environment. Patterned photoresist is thermally treated in order to form a temporary structural mold covering the device. The effects of photoresist thickness and geometry are being studied in order to minimize the duration and temperature requirements of this treatment process. The photoresist mold is then metallized and a reactive ion etch (RIE) process is used to create vias for photoresist removal. Also under investigation are the relationships between the film stresses and structural stability of the devices. Upon removal of the photoresist, a final metallization by an evaporation process is used to seal the structure under vacuum conditions. In this presentation the process flow for the vacuum micro-encapsulation package will be described and the materials requirements will be enumerated.

MM-TuP5 Investigation on Metal-coated Nano-aperture Array, ***D.W. Kim,*** *J.T. Ok, S.S. Choi,* Sun Moon University, Korea*; J.W. Kim, J.H. Boo,* Sungkyunkwan University, Korea*; C.K. Chun,* Sun Moon University, Korea*; J.S. Yang,* Myongji University, Korea

There have been considerable interests in the nano-aperture due to its potential application for promising near-field optical recording. Near-field optical recording can increase the data storage density drastically as it circumvents the diffraction limit. For the development of the practical optical storage device, a parallel processing technique based on nano-aperture array has been being investigated. In this work, the controllable method for the fabrication of metal-coated nano-aperture array and its optical characteristics in the far-field regime will be presented. At first, the arrays of inverted pyramidal structures were generated by anisotropic etching using 5 µm size pattern. Next, the stress-dependent oxide growth on the concave Si surface of the hollow pyramids was performed at 1000 °C. Backside Si etching by alkaline solution was followed and released the hollow oxide pyramids array with intentionally designed convex lens-like facets. Nano-apertures have been opened at the apices of pyramids by 50:1 diluted HF solution and the relation between the aperture diameter and the etch time showed good linearity with the aperture opening rate of ~25 nm/min. Finally, Al thin layer was deposited on the outer surface of oxide pyramid by PVD for the purpose of further reducing the aperture diameter, which will play the role of a wave-guide as well. The diameter of the completed aperture was observed to be inversely proportional to the thickness of Al layer. The details of the fabrication procedure and the far-field optical characteristics will be reported.

Microelectromechanical Systems (MEMS)
Room 320 - Session MM-WeM

New Frontiers in Microsystems: NEMS and BioMEMS

Moderator: R. Ghodssi, University of Maryland

8:20am **MM-WeM1 The Science and Technology of Nanomechanical Resonant Structures**, ***L. Sekaric,*** Cornell University **INVITED**

The development of nanometer-scale mechanical structures into sensitive and integrated devices has enabled us to envision applications such as cell phones the size of a wrist watch, fast and sensitive pathogen detectors, and ultra-sensitive field and force gauges. An exact understanding of the physical phenomena affecting NEMS behavior should aid in the successful application of these systems. Several important achievements in this field will be described - from fabrication of ultra-small and ultra-high frequency resonant structures in a variety of materials to understanding their behavior and improving their performance. Some of the topics to be discussed include the time scales of nanoscale dynamics, the operation of NEMS in vacuum and in air, the sources of energy dissipation, and the effects of thin film mechanics in NEMS.@footnote 1@ @FootnoteText@ @footnote 1@In collaboration with: M. Zalalutdinov, K. Aubin, A. T. Zehder, J. M. Parpia, and H. G. Craighead, Cornell Center for Materials Research, and J. E. Butler, Naval Research Labs.

9:00am **MM-WeM3 Investigation of Nanostructuring by Use of Focused Ion Beam Fine Milling**, ***Y. Fu,*** Nanyang Technological University, Singapore

Micro-pillars with nano-sizes for application of molecular controlling were fabricated by use of focused ion beam (FIB) fine milling on substrate of silicon. The nano-pillars can realize cell/molecular adhesion, and movement control by its high-density contact dots and tiled cone angle of the pillars, which can be obtained by means of FIB directly fine milling with a stage in a certain tilted angle. The milling process was investigated under different beam current and the stage-tilting angle, which determines an aspect ratio and the tilted angle of the pillars. Chemical assistant etching (GAE) with chemical gas of XeF2 was used for the purpose of deviating the pillars with higher aspect ratio. With these features, the pillars can realize cell/molecular movement in only one direction and cannot be backward. It will be helpful for DNA and protein analysis, such as molecule separation and purification, molecular detection, and DNA hybridization, etc.

9:20am **MM-WeM4 Electron Interactions in Nanoscale Focused Electron Beam Processing**, ***P.D. Rack,*** *J. Kim, J.D. Fowlkes, S.J. Randolph, D.C. Joy,* University of Tennessee

Focused electron beam induced processing has recently been demonstrated to be a viable technique for selective nanoscale processing. The technique is similar in principle to focused ion beam processing, however, the electron-stimulated reactions have been shown to have a smaller effective beam width (~ 50nm) and do not suffer the collateral damage associated with gallium implantation. In this presentation, we will show our recent progress in electron beam stimulated deposition and etching. Particular attention will be given to the effects that secondary and backscattered electrons have on the deposited or etched structure. Electron-solid and electron-gas Monte Carlo simulations will be correlated to the observed deposition and etching profiles profiles. Beam energy and current density effects will also be shown and explained. Finally, application of the process to several nanoscale devices will be demonstrated.

9:40am **MM-WeM5 BioMEMS-Based Platforms for Drug Delivery: Implantable, Ingestible, and Beyond**, ***T.A. Desai,*** Boston University **INVITED**

Microfabrication techniques, which permit the creation of multifunctional platforms that possess a combination of structural, mechanical, and electronic features, may surmount several challenges associated with the conventional delivery of therapy. In this talk, in vivo delivery concepts are presented which capitalize on the strengths of micro and nanofabrication. Current work on micromachined nanoporous implantable biocapsules for the immunoisolation of pancreatic islet cells - as a possible treatment for diabetes -- will be described. In addition, asymmetrical, reservoir-containing microfabricated particles and arrays with specific biorecognition ligands will be discussed for improving the oral delivery of peptides and drugs. Such microengineered interfaces may be optimized for biomolecular selectivity and surface bioactivity. With the capability to design components spanning from the millimeter down to the nanometer range, few other engineering technologies can so closely parallel the multidimensional size scale of the living cells and tissues, with control and reproducibility, in the same fabrication process. Micro/Nanotechnology can add flexibility to current practices while becoming an enabling technology leading not just to new therapies and laboratory techniques, but to new models for delivering healthcare to the patient.

10:20am **MM-WeM7 Flexible, Polyimide-Based Microfluidic Devices for BioMEMS**, ***S. Metz,*** *A. Bertsch, Ph. Renaud,* Swiss Federal Institute of Technology Lausanne (EPFL), Switzerland

We present flexible, polyimide-based microfluidic devices for a wide range of applications in the field of BioMEMS. Fluidic microchannels are manufactured by a modified lamination technique or a sacrificial layer method. For the lamination technique a layer of uncured polyimide is irreversibly bonded to open channel structures of semi-cured polyimide, which yields very high bond strengths. The sacrificial layer technique implies the use of a heat-depolymerizable polycarbonate sacrificial material. The material is embedded in two layers of polyimide and diffuses through the channel cover layer during the last fabrication step leaving empty microfluidic channel networks behind. The microchannels can be combined with metallization layers for the integration of microelectrodes inside the microchannels, which is a major requirement in the field of miniaturized bio-chemical analysis. The electrodes inside the channels can be used for fluid actuation or detection of substances. The embedded layers of metal can also be used as microelectrodes for the recording or stimulation of bio-electric activity. This results in devices, which are capable of selectively delivering fluids to cells and at the same time enable electrophysiological monitoring. Additionally, the channel walls can be made porous by ion track technology, which yields sub-micron, high aspect-ratio pores perpendicular to the fluidic structure with a pre-defined pore density. The pores can be generated in the top and/or bottom channel walls of the microfluidic device and the pore size is adjustable down to tens of nanometers. These devices can be used for the separation of particles by cross-flow filtration.

Homeland Security Topical Conference
Room 309 - Session HS+MM-WeA

Detection of Explosives and Other Chemicals for Homeland Security

Moderators: R. Cavanagh, National Institute of Standards and Technology, R.T. Lareau, Transportation Security Administration

2:00pm **HS+MM-WeA1 MEMS Chemical Sensors for Homeland Security**, ***D.C. Meier***, National Institute of Standards and Technology; *C.J. Taylor,* Pomona College; *R.E. Cavicchi, Z. Boger, S. Semancik,* National Institute of Standards and Technology

Chemical sensors capable of accurate detection of trace quantities of chemical warfare (CW) agents would provide a potent tool for perimeter security, treaty verification, and battlefield threat detection. Ideally, such sensors would be highly sensitive, selective, compact, and require low power. Since many CW agents are lethal at µmol/mol (ppm) concentrations, reliable trace detection is the critical prerequisite to the employment of life-saving countermeasures. In order to meet these goals, microsensors consisting of CVD TiO@sub 2@ and SnO@sub 2@ sensing films on MEMS array platforms have been fabricated. Their response measurements to the CW agents GA (tabun), GB (sarin), and HD (sulfur mustard) in dry air at concentrations between 5 and 200 nmol/mol (ppb), as well as to CW agent simulants CES (chloroethyl ethyl sulfide) and DFP (diisopropyl fluorophosphate) between 250 and 3000 ppb are reported. The devices yield both excellent signal-to-noise and good reproducibility for similar devices. Detection in backgrounds of common battlefield interferants is also discussed. The temperature of each sensor element is independently controlled by embedded microhotplate structures that drive both the CVD process (375 °C) and sensor operation at elevated temperatures (325 to 475 °C). The concentration-dependent analyte response magnitude is sensitive to sensing film growth conditions. Long-term stability studies verify stable sensor responses to GB and HD for 14 hours of agent exposure. Use of fast (200 ms) temperature programmed sensing (TPS) over a broad range (20 to 480 °C) enhances analyte selectivity, since the resulting signal trace patterns include only kinetic data that are unique for each agent tested. Recursive training of an artificial neural network (ANN) assembles the set of most relevant inputs from the TPS data; subsequent validation of the trained ANN verifies positive agent and simulant identification and quantification.

2:20pm **HS+MM-WeA2 Development of a Piezoelectric Microphone for Trace Gas Detection**, ***R.G. Polcawich***, *P. Pellegrino,* U.S. Army Research Laboratory

Escalating environmental awareness has led to more restrictive regulations on air quality in both the workplace and the environment in general. As a result, there is an increasing desire to have portable trace gas analyzers especially for chemical and biological agent detection. To meet these goals, a gas detection system must be miniaturized allowing large scale production of affordable small sensing systems. Initial examination of the scaling principles associated with photoacoustic spectroscopy (PAS) in respect to microelectromechanical system (MEMS) dimensions indicate the photoacoustic signals would remain at similar or greater sensitivities commonly found in macro-scale devices.@footnote 1@ Several other issues including: increased stability, noise avoidance, small source-to-detector distances and monolithic sensor construction support the idea that a MEMS photoacoustic chemical sensor can be realized.@footnote 2@ Our current research efforts have focused on fabrication of a piezoelectric microphone for trace gas sensing using a MEMS PAS. Using lead zirconate titanate (PZT) thin films as the piezoelectric sensor, 500 to 2000 micrometer diameter acoustic sensors have been fabricated and have an unamplified sensitivity of 0.1 to 1.0 µmV/Pa, depending on geometry. Using a limit of detection determined from the noise floor of the PZT microphone, it is anticipated that a PZT based acoustic sensor should detect SF@sub 6@ at slightly less than 1 ppb. Our presentation will cover the fabrication, packaging, and testing of a piezoelectric microphone for use in a MEMS based PAS detector. @FootnoteText@ @footnote 1@ S.L. Firebaugh, K.F. Jensen, and M.A. Schmidt, Miniaturization and integration of photoacoustic detection, J. Appl. Phys., vol 92, pp.1555-1563 (2002).@footnote 2@ P. Pellegrino and R. Polcawich, Evaluation of a MEMS Photoacoustic Sensor, submitted to 2002 Joint Service Scientific Conference Chemical Biological Defense Research, Hunt Valley.

2:40pm **HS+MM-WeA3 Fabrication, Packaging and Testing of Micronozzles for Gas Sensing Applications**, ***S. Li,*** University of Maryland, College Park; *C.B. Freidhoff, R.M. Young,* Northrop Grumman Electronic Systems Inc.; *R. Ghodssi,* University of Maryland, College Park

There has been recent, rapid development of MEMS-based gaseous microfluidic devices (GMDs) working under standard atmospheric conditions. One of the applications considered for the GMD is for a frontend of a miniature chemical sensor to improve its sensitivity. We report a fabrication technology for developing linear converging-diverging micronozzles using low temperature wafer-level adhesive bonding with SU-8. The process is quick, repeatable and relatively insensitive to presence of particles on the wafers to be bonded. A selection of wafer bonding tests with SU-8 as the intermediate bonding material has been performed to investigate the influence of different parameters on the bonding of structured wafers. A crack-opening method is used to evaluate the surface energy of bonded wafers to be in the range of 0.42-0.56 J/m@super 2@. Based on the results of wafer bonding test with SU-8, sealed micronozzles with throat widths as small as 3.6 µm are fabricated successfully. For the purpose of comparison, micronozzles of same geometries and dimensions are also fabricated using deep reactive ion etching (DRIE) and silicon-glass anodic bonding techniques. The micronozzles are packaged to interface with a gas flow test setup using capillary needles, O-rings and flexible tubing. Gas flow rates and pressure distributions in the micronozzles are measured and compared with those predicted from the one-dimensional isentropic flow model, which demonstrate that these developed techniques may extend the flexibility of fabricating and packaging microfluidic devices for gas sensing applications. The detailed fabrication process and testing results will be presented.

3:00pm **HS+MM-WeA4 Characterization of Portal-Based Trace Explosive Detection Systems**, ***G. Gillen***, *R.A. Fletcher, S.V. Roberson, C. Zeissler, E.S. Etz, J. Verkouteren, M. Verkouteren, E.S. Windsor, G.A. Klouda,* National Institute of Standards and Technology; *R.T. Lareau,* Transportation Security Administration

In collaboration with the Transportation Security Agency's (TSA) Trace Explosive Detection Group, the NIST Chemical Science and Technology Laboratory (CSTL) has been working to build a chemical metrology program to help support the widespread operational deployment and effective utilization of trace explosives detection devices throughout the United States. A second objective is to develop at NIST the specialized measurement expertise that will be needed to support the next generation of explosive detection equipment. Of particular interest for this work is the characterization of walk-through trace explosive detection portal (TEDPâ€™s) systems currently under evaluation by the TSA. The low volatility of most high explosives makes direct analysis of vapors impractical. Therefore, many detection systems are based on either airborne or surface swipe collection of micrometer-sized explosive particles with subsequent thermal vaporization of the particles into an ion mobility spectrometer for identification. The effective collection and thermal desorption of the explosive particles is the critical front-end process for the successful and reproducible detection of explosives. In order to understand this process in detail we are employing a number of microanalytical techniques including: SIMS and TOF-SIMS, SEM, Raman, Optical and Fluorescence Microscopy, Infrared Thermometry and Optical Particle Counting. These techniques are being used to study, at the single particle level, the collection and removal of individual explosive particles from surfaces and the chemical and morphological changes that occur during sampling. This presentation will review the NIST-TSA project and will present our recent findings with an emphasis on chemical characterization of individual explosive particles.

3:20pm **HS+MM-WeA5 Polymer Electronics for Ultra-Sensitive Chemical and Biological Sensors**, ***T.M. Swager***, Massachusetts Institute of Technology **INVITED**

This presentation will describe the design of electronic polymers that have the ability to undergo self-amplified responses.@footnote 1@ Optimal energy and charge transport properties are key to the amplifying ability of these materials. Design principles have been developed that can be used to improve the mobility and lifetime of excitons will be presented. To elicit a selective sensor response different molecular recognition principles have been integrated into the polymers. Effective implementation of recognition elements requires effective transduction events that are compatible with the amplifying ability of the polymers. Designs based upon energy transfer, quenching, and excimer formation will be presented for the detection of DNA, Proteins, Chemical Warfare Agents, Explosives, and Ions. I will also

discuss our continuing efforts in the design of sensory materials that utilize molecular recognition events to product changes in conductivity. In many cases we use transition metal ions are redox active elements in these materials and I will outline the design principles for producing conducting polymers that utilize the metal ions as part of the conducting pathway. These materials have allowed us to produce new sensors for nitric oxide, a signaling agent in biological systems. @FootnoteText@ @footnote 1@ (a) Swager, T. M. "The Molecular Wire Approach to Sensory Signal Amplification" Accts. Chem. Res. 1998, 31, 201-7. (b) McQuade, D. T.; Pullen, A. E.; Swager, T. M. "Conjugated Polymer Sensory Materials" Chem. Rev. 2000, 100, 2537-2574. (c) Self-AmplifyingSemiconductingPolymers for Chemical Sensors Swager, T. M.; Wosnick, J. H. MRS Bulletin, 2002, June, 446. (d) J.H. Wosnick and T.M. Swager, "Molecular Photonic and Electronic Circuitry for Ultra-Sensitive Chemical Sensors" Curr. Opin. Chem. Biol. 4 (2000) p. 711.

4:00pm **HS+MM-WeA7 DIOS-MS and LC-DIOS-MS for Chemical Analysis**, *Z. Shen, C. Fish,* University of California, San Diego*; G. Siuzdak, M.G. Finn,* The Scripps Research Institute*; **J.E. Crowell**,* University of California, San Diego

Desorption/Ionization On Silicon Mass Spectrometry (DIOS-MS) is a new mass spectrometry strategy based on pulsed laser desorption/ionization from a porous silicon surface. DIOS-MS is similar to matrix-assisted laser-desorption ionization mass spectrometry (MALDI-MS) in that it utilizes the same instrument; however, in DIOS-MS, porous silicon is used to trap analytes deposited on the surface and laser radiation is used to vaporize and ionize these molecules, without the presence of any matrix material. We have shown that DIOS-MS can be used for a wide range of biomolecules, organic molecules, and metal-containing compounds at the femtomole and attomole level with little or no fragmentation. DIOS-MS offers many unique advantages including good sensitivity, low background ion interference, and high salt tolerance. We will discuss the coupling of liquid chromatography separation with DIOS-MS for protein identification and peptide sequencing, and the use of chemical and surface modification for tailored analysis.

4:20pm **HS+MM-WeA8 A Novel Chemical Detector Using Cermet Sensors and Pattern Recognition Methods**, ***S.L. Rose-Pehrsson***, Naval Research Laboratory*; J. Ziegler,* General Atomics*; M.H. Hammond,* Naval Research Laboratory*; D. Gary, K. Caudy,* General Atomics

Smart microsensor arrays are being developed by combining cermet electrochemical sensors, intelligent firmware and software to drive the sensors and analyze the data. The chemical microsensors offer a small size, light weight, low power and low cost alternative to conventional electrochemical sensors. The chemical microsensor architecture may be modified for detection selectivity of a variety of chemical species including chemical agents and combustible or corrosive gases. The microsensor arrays have potential application for monitoring hazardous chemicals in the part-per-million to part-per-billion range in a variety of internal and external environments. The arrays sense analytes using pattern recognition techniques to determine the presence of vapors of interest. General Atomics and the Naval Research Laboratory are developing this technology for the detection of chemical warfare agents and toxic industrial compounds (TICs). A test demonstrator has been developed with a three-sensor array, readout electronics, and system control software. The three-sensor array was exposed to 15 test vapors. The 15-analyte sources, including 2 blood agents, 10 TICs and 3 simulants were generated at 5 different concentrations in humid air. The cermet sensor array provided unique responses for the various analytes tested. Similar analyte types produced similar results. The sensitivity is sufficient to detect all the analytes at their respective exposure limits. Two different pattern recognition methods were developed to identify the analytes.

4:40pm **HS+MM-WeA9 Chemical Warfare Agent Detection Using Random Networks of Single-Wall Carbon Nanotubes**, ***E.S. Snow***, *J.P. Novak, E.J. Houser,* Naval Research Laboratory

Single-wall carbon nanotubes (SWCNT) are unique structures in that they are composed entirely of surface atoms while exhibiting transport properties superior to single crystalline Si. These features make them an ideal candidate for a new class of molecular sensors. We report the use of random networks of SWCNTs as a sensor for chemical warfare agents. Random networks of SWCNTs are used to provide high-yield sensor fabrication using conventional microlithographic techniques. Molecular adsorption of chemical agents onto the nanotube networks results in a charge transfer that manifests itself as a change in the network conductance. In such devices sub-part-per-billion sensitivity to agent simulants is easily achieved. We discuss these results and our approach to functionalizing the networks to provide additional sensitivity, selectivity against potential interferents, and a high degree of chemical specificity. Arrays of such functionalized devices should provide highly sensitive and specific electronic detection of a wide range of chemical warfare agents and other toxic chemicals.

5:00pm **HS+MM-WeA10 Carbon Nanotubes for Molecular Sensors and Electronic Circuit Elements**, ***M.J. Bronikowski***, *D.S. Choi, M.E. Hoenk, B.D. Hunt, R.S. Kowalczyk, E.W. Wong, A.M. Fisher,* Jet Propulsion Laboratory/California Institute of Technology*; B. Rogers, J.D. Adams,* University of Nevada, Reno*; J. Xu,* Brown University*; J.F. Davis, L.W. Epp, D.J. Hoppe,* Jet Propulsion Laboratory/California Institute of Technology

This talk will focus on recent efforts at JPL's Microdevices Laboratory in developing several different types of nano-scale electronic devices based on carbon nanotubes (CNT). CNT exhibit a coupling between electronic structure and mechanical deformations: mechanical stress or deformation can result in charge injection into the nanotube, or likewise, charging of a nanotube can result in mechanical deformations. This electromechanical coupling can form the basis for nanotube-based oscillators, signal processors, and RF rectifiers. Nanotube electronic properties, specifically their resistance and current-voltage characteristic, can also change when various molecules bind to their surfaces. This property can form the basis for CNT-based chemical and molecular sensors. For both types of device, CNT are grown directly on silicon substrates in pre-patterned device structures: nanotubes grow by CVD from patterned arrays of particles of catalytic metals, with the pattern of the catalyst determining the pattern of CNT. Of key importance to producing devices by this means is controlled placement of catalyst on the substrate: several methods for generating catalyst patterns on surfaces and devices will be demonstrated and discussed.

Thin Films
Room 326 - Session TF+MM-WeA
Sensors, Smart Films and Functional Materials

Moderator: C.H. Stoessel, Consultant

2:00pm **TF+MM-WeA1 CMOS-Based Microsensors**, ***O. Brand***, Georgia Institute of Technology **INVITED**

CMOS-based microsensors combine, on a single chip, the necessary transducer elements and integrated circuits. This way, the microsensors benefit from well-established fabrication technologies and the possibility of on-chip circuitry. Besides sensor biasing and signal conditioning, added on-chip functionality, such as calibration, self-testing, and digital interfaces, can be implemented. A number of microsensors, including magnetic field and temperature sensors, are completely fabricated within the regular CMOS process sequence. A far larger number of microsystems can be realized by combining CMOS or BiCMOS technology with compatible micromachining and thin film deposition steps. These additional fabrication steps are performed either before, in-between, or after the regular CMOS process sequence. Commercially available examples include pressure sensors, accelerometers, gyroscopes, humidity sensors, mass flow sensors, and imaging devices. In the first part, the paper summarizes major technological approaches to CMOS-based sensors. In the second part, a packaged CMOS-based chemical microsystem, developed at ETH Zurich, Switzerland for the detection of volatile organic compounds in air is highlighted. On a single chip, the microsystem combines a sensor array featuring three different sensing principles with circuitry for sensor biasing, signal read-out, analog-to-digital conversion, and digital interfacing. The chemical microsystem is fabricated using an industrial CMOS technology in combination with post-processing bulk-micromachining to release the micromechanical sensor structures. After packaging the microsystem using flip-chip technology, the three sensor structures are coated with chemically sensitive polymer films. Absorption of volatile organic compounds in the polymer films results in a change of the (physical) film properties, such as the mass, dielectric constant, or temperature, which is then recorded by the underlying sensor structure.

2:40pm **TF+MM-WeA3 Behavior of Thin Ionic Liquid Films Studied with Atomic Force Microscopy**, ***J.J. Nainaparampil***, *B.S. Phillips,* AFRL/MLBT*; K.C. Eapen,* University of Dayton Research Institute*; J.S. Zabinski,* AFRL/MLBT

Ionic liquids (IL's) represent a new class of solvents having the character of molten salts. They have no detectable vapor pressure, are moisture, air and temperature stable and therefore are excellent solvents. Most of these IL's

consists of cations such as different alkyl imidazolium or alkyl pyridinium ions and anions such as BF@sub 4@, PF@sub 6@, N (CF@sub 3@SO@sub 2@)@sub 2@, CF@sub 3@SO@sub 3@. In this work, crystals of alkyl imidazolium+ PF@sub 6@ are dissolved in water or acetonitrile to form 0.2% to 0.5% solutions and deposited on Si surface to form thin films. Atomic force microscope working in non-contact mode capable of providing biased tip lithography is used to characterize these films. It is observed that scanned films give rise to certain geometrical structures that are repeated in recrystallized surfaces. A biased AFM tip is used to mobilize these structures to form other complex structures. It is noted that the same solution, when used as a lubricant in sliding contacts gives rise to drastic friction reduction compared to other lubricants. These IL's were used to lubricate a micro electro mechanical system (MEMS) electro static output motor. Results from MEMS endurance tests and an interface model that is based on the AFM study will be presented. Electro-migration of crystallites and adhesion of transfer films affect the friction and durability significantly.

3:00pm **TF+MM-WeA4 Bilayer Transition-edge Sensors for X-ray Calorimeter and Infrared Bolometer Arrays*, J.N. Ullom**, *J.A. Beall,* National Institute of Standards and Technology*; J. Beyer,* PTB, Guest Researcher NIST*; S. Deiker, W.B. Doriese, G.C. Hilton, K.D. Irwin, C.D. Reintsema, L.R. Vale,* National Institute of Standards and Technology **INVITED**

Microcalorimeters and bolometers made from thin superconducting films cooled to temperatures near 100 mK have made dramatic progress in recent years. These devices provide an order of magnitude improvement in energy resolution over existing semiconductor x-ray sensors and are likely to be used in upcoming astronomical instruments spanning the spectrum from x-ray to millimeter wavelengths. The sensitivity of these devices is derived from the low heat capacities and thermal conductivities possible near 100 mK and from the strong dependence of resistance on temperature in the superconducting-to-normal transition. Our devices are made from bilayers of a normal metal and a superconductor. Use of a bilayer allows the transition temperature and resistivity of the sensors to be precisely controlled. In this talk, we describe recent progress towards kilopixel sensor arrays using multiplexed SQUID readout. We are building arrays of x-ray microcalorimeters for two applications: energy-dispersive x-ray spectroscopy on scanning electron microscopes and for the upcoming NASA satellite Constellation-X. We are building arrays of submillimeter bolometers for the SCUBA-2 camera on the James-Clerk Maxwell Telescope on Mauna Kea. At this time, the measured noise in both microcalorimeters and bolometers approaches but does not equal the value predicted from simple thermodynamics. We will present measurements of this excess noise and describe recent mitigation efforts.

Author Index

Bold page numbers indicate presenter