MEMS and NEMS
Room 207 - Session MN-MoM

Processing & Characterization of Materials for MEMS & NEMS

Moderator: S. Burkett, University of Arkansas

8:20am **MN-MoM1 Mechanical Properties of Polysilicon Thin Films using Micromachined Membranes and a Design of Experiments Methodology**, *A.J. Fleischman, A. Dubnisheva, R.S. Butler,* The Cleveland Clinic Foundation*; R. Rosenblum,* ***C.A. Zorman,*** Case Western Reserve University*; S. Roy,* The Cleveland Clinic Foundation

The Youngs modulus, residual stress, and burst pressure of 4 micron-thick polysilicon films were determined from the load-deflection characteristics of suspended membranes. Specimens were prepared by the deposition of undoped amorphous Si films onto Si3N4 coated, Si wafers by LPCVD. The wafers (called Poly2) were annealed at 1100C for 1 hour to crystallize the films and lower the residual stress. For roughly 50% of the samples (called Poly1), a second identical annealing step was performed to evaluate changes in mechanical properties when such films are used in multilayer devices. Membranes that were 1.2 x 1.2 mm2 in area were fabricated by KOH-based anisotropic etching, using the underlying nitride film as an etch stop. Each test chip was subjected to pressure cycling tests using an interferometric load-deflection setup developed specifically for membrane evaluation. Data were collected using two test chip mounting schemes: (1) epoxy mounting, and (2) mechanical slot screw clamping. We found that mechanical clamping was sufficient to achieve the desired results with the added benefit that the specimens could be demounted without damage. A design-of-experiments testing methodology and one way ANOVA analysis at the 95% confidence interval was employed to factor out environmental and testing setup variables associated with the measurement technique. We found the Poly1 samples had an average Youngs modulus of 163 GPa and a residual stress of 121 MPa, while those for Poly 2 were 141 GPa and 23 MPa. The membranes were also evaluated for burst pressure in a setup capable of pressurizing the membranes well above the lowest average burst pressure of 78 psi. SEM analysis was used to examine the membranes after bursting. We found that the membranes did not delaminate, but rather failed at sites near the membrane edges. The presentation will detail the testing and data analysis procedures, as well as the use of these data in designing MEMS structures subject to failure.

8:40am **MN-MoM2 Characterization of Nanoscale Wear Processes in Polysilicon-Based MEMS Devices using AFM and PEEM-NEXAFS Spectromicroscopy**, ***A.V. Sumant****, D.S. Grierson, G. Wabiszewski, R.W. Carpick,* University of Wisconsin at Madison*; A. Corwin, M. De Boer,* Sandia National Laboratories

We present studies aimed at elucidating mechanisms of nanoscale wear in polysilicon-based microelectromechanical systems (MEMS) devices. Current silicon-based MEMS devices that involve frictional sliding fail due to wear. Coating MEMS parts with self-assembled monolayers (SAMs), which act as lubricating and passivating layers, can improve the performance of these devices to some extent. However, devices coated with SAMs have finite lifetimes and can fail after unsuitably short periods of time. We seek to determine the precise causes of failure to ultimately improve the performance of MEMS devices. We use a atomic force microscopy (AFM) and PEEM-NEXAFS (Photoelectron Emission Microscopy combined with Near-Edge X-ray Absorption Fine Structure) spectromicroscopy to obtain quantitative information on structural damage and chemical changes inside the wear track of a MEMS device specifically designed to conduct friction and wear tests under controlled conditions. The ability of the PEEM-NEXAFS technique to spatially resolve and chemically characterize regions of interest is unparalleled and therefore ideally suited for this work. The results show for the first time that it is possible to detect chemical changes occurring within the micro-scale wear track. Furthermore, we are able to correlate the spectroscopically-observed features from the PEEM-NEXAFS measurements with AFM measurements of the modified surface topography in the wear track. One critical challenge is to minimize radiation damage of the SAMs due to synchrotron X-rays exposure during characterization. We show that by using radiation-blocking shutters and adjusting photon exposure and flux, damage can be reduced and reliable data can be obtained.

9:00am **MN-MoM3 Science and Technology of Ultrananocrystalline Diamond Thin Films and Their Integration for Fabrication of Advanced MEMS/NEMS**, ***O. Auciello,*** Argonne National Lab*; J. Birrell,* Presently with Advanced Diamond Technologies*; J.A. Carlisle,* Argonne National Lab*; K.-H. Kim, B. Peng, H.D. Espinosa,* Northwestern Univ.*; A.V. Sumant, D.S. Grierson, N. Guoqing, R.W. Carpick,* Univ. of Wisconsin-Madison **INVITED**

The science and technology of multifunctional thin films and interfaces and new phenomena sustained in film-based nanostructures are opening the way for a new generation of multifunctional microelectromechanical and nanoelectromechanical system (MEMS/NEMS) devices. In this talk, we will review the fundamental and applied science related to the synthesis and characterization of ultrananocrystalline diamond (UNCD) thin films and their integration with other materials for the fabrication of advanced MEMS/NEMS devices. UNCD films are grown using a novel Ar-rich CH@sub 4@/Ar plasma chemistry that results in the synthesis of films with 3-5 nm grains and atomically abrupt grain boundaries, and it is this unique nanostructure that is responsible for the unusual combination of mechanical, tribological, chemical and electronic (when doped) properties of UNCD relevant to MEMS/NEMS. We will discuss results from joint research between Argonne and Northwestern University that provided unique insights into the mechanical properties of UNCD relevant to MEMS and NEMS as well as the fabrication of MEMS structures and characterization of their mechanical properties, We will also discuss results from joint research between Argonne and University of Wisconsin-Madison that provided unique insights into the effect of seeding layers such as W on the growth of UNCD films and on their morphology and microstructure and the resulting effects on nanoscale tribological and electrochemical properties, We will discuss characterization of the surface chemistry and bonding probed by total electron- and fluorescence-yield near edge x-ray absorption fine structure (NEXAFS) spectroscopy to distinguish between near-surface and deeper (“bulk”) changes in the film and correlations with tribological and electrochemical properties that depend sensitively on the surface chemistry and bonding. The fundamental and applied science of UNCD films will be discussed in view of fabrication of structures for advanced MEMS/NEMS devices. *Work supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences, under Contract W-31-109-ENG-38.

9:40am **MN-MoM5 Quantitative Work-of-Adhesion Values for use as an In-Fab Monitor of Stiction**, *E.J. Thoreson,* Worcester Polytechnic Institute*; J. Martin,* Analog Devices, Inc.*;* ***N.A. Burnham,*** Worcester Polytechnic Institute

The Atomic Force Microscope (AFM), a common tool in the fab, can measure the work of adhesion between AFM tips and MEMS surfaces, which could become an eventual predictor of device stiction and failure. The goal of our study was to ensure reliable and quantitative values for the work of adhesion, i.e., the adhesive ("pull-off") force normalized for tip radius. Seventeen tips of four different types were used, with radii from 200 nm to 60μm, covering the range of typical MEMS contacts. The samples were unpatterned amorphous silicon dioxide MEMS die with two types of surface conditions (untreated and treated with a few angstroms of vapor deposited diphenylsiloxane). The cantilever's length, angle of repose, the radius and height of the tip, and the surface roughness all contribute to the measured pull-off force and work of adhesion. A simple correction for the surface roughness resulted in the expected linear dependence of pull-off force on radius, but the magnitudes for the pull-off force and work of adhesion were higher than expected. Normal heat-treated AFM tips have minimal surface roughness and result in magnitudes that are more reliable. The typical relative standard deviation and current relative uncertainty for these data are 20% and 15%, respectively. In this presentation, we derive how the cantilever and tip parameters contribute to the measured work of adhesion, show how the corrected results compare with theory, and list our recommendations for using the AFM as a quantitative in-fab stiction monitor. Work-of-adhesion data can now be meaningfully compared to actual device performance.

10:00am **MN-MoM6 An SPM-Based System for Contact Reliability Characterization**, ***L. Chen****, N. McGruer, G. Adams,* Northeastern University*; R. Coutu, K. Leedy,* Air Force Research Laboratory

An SPM-based test station has been established for studying reliability physics of contacts in a micromechanical switch. A wide contact force range is accessible, from nN to mN, as determined by the mechanical properties of the microfabricated contact test cantilevers. During testing the contact force, the contact adhesion, and the contact resistance are measured. Material transfer is observed with an SEM. The cycling rate can

reach 200 kHz by driving a piezo actuator at resonance. The contact properties of gold, ruthenium, rhodium and platinum and gold alloy have been studied and compared. Gold-on-gold contacts typically show decreased resistance, increased adhesion, and large amount of material transfer as the number of cycles increases. Ruthenium-on-gold and ruthenium-on-ruthenium contacts have better mechanical performance, but a higher contact resistance which increases with cycling. Two different shaped contacts, flat-top and hemispherical shaped, are used to study mechanical contact degradation. This work was supported by DARPA under its HERMIT program through research grant F33615-03-1-7002 to Northeastern University.

10:20am **MN-MoM7 Surface Chemistry of Organosilanes as a Vapor Phase Lubricant for Si-based MEMS Applications***, **D. Kim**, A.J. Gellman,* Carnegie Mellon University

Microelectromechanical systems (MEMS) offer the opportunity to improve a number of technologies and to develop devices that are significantly smaller and more lightweight than those in current use. Unfortunately, the long term operation of MEMS devices requires some form of lubrication for components in sliding contact. While traditional liquid lubricants cannot be used, vapor phase lubrication may be an effective solution. MEMS devices are made from single-crystal silicon and polycrystalline silicon, the surfaces of which are covered with a thin film of silicon dioxide, SiO@sub 2@, when exposed to air. The lubrication of MEMS materials requires both the replenishment of SiO@sub 2@ from the worn surfaces and the application of a lubricant film that will minimize wear. Organosilanes, R@sub n@Si(OR)@sub 4-n@, such as triethoxyphenylsilane, (C@sub 6@H@sub 5@)Si(OCH@sub 2@CH@sub 3@)@sub 3@, are proposed as vapor phase lubricants that can perform both functions. The surface chemistry of triethoxyphenylsilane on Si(100) and SiO@sub 2@ surfaces has been studied, using temperature programmed reaction spectroscopy and Auger electron spectroscopy. This chemistry was compared to that of tetraethoxysilane, (CH@sub 3@CH@sub 2@O)@sub 4@Si, which is widely used as a precursor for SiO@sub 2@ film deposition. Triethoxyphenylsilane decomposed readily, depositing SiO@sub 2@ and graphitic carbon on Si(100) and SiO@sub 2@ surfaces. Triethoxyphenylsilane formed more graphitic surface films than tetraethoxysilane on Si(100) surface at T=300-600 K. Triethoxyphenylsilane decomposes by C-O cleavage to ethyl groups which desorb as ethylene via @beta@ -hydride elimination. The remainder produces phenyl groups which desorb as benzene or decompose further to deposit graphitic carbon onto the surface. These results suggest that lubrication of MEMS materials could be accomplished, using vapor phase organosilanes such as triethoxyphenylsilane.

10:40am **MN-MoM8 Post-Processing Curvature Modification of Cantilever Microbolometer Focal Plane Arrays***, **S. Huang**, X. Zhang,* Boston University

Infrared vision is a key technology in a variety of military and civilian applications. Recent advances in MEMS have led to the development of uncooled microcantilever bolometers, which function based on the bending of bimaterial cantilevers upon the absorption of IR energy. Such microbolometer FPAs, however, always curve up or down because of the imbalanced residual stresses in the dissimilar materials, significantly weakening their performance and functionality. We report a post-processing engineering approach to address this issue: the method we used includes a combination of ion beam machining and rapid thermal annealing treatments. In our experiments, bimaterial SiNx/Al for microbolometers were fabricated using the surface micromachining technique with the polyimide as sacrificial material. The Al layer was deposited by electron beam deposition and the SiNx layer by PECVD. The thickness of the Al layer was 200 nm and that of the SiNx layer was 250 nm. To modify the curvature of the as-fabricated FPAs, first, ion beam machining was used. We found 20-min machining resulted in a significant improvement in the FPAs curvatures. Second, RTA was adopted to further modify the residual stresses and hence the curvatures of the FPAs. The FPAs initially bent down to the substrate, totally losing their function. A 5-min RTA treatment at 350Â°C resulted in less deflected pixels, while a treatment at 375Â°C led to pixels with an acceptable curvature. High-temperature, however, could deteriorate the residual stress state, causing the FPAs bent even upwards. In summary, we demonstrated that a combination of ion beam machining and RTA techniques can be used effectively to eliminate the residual stress-induced curvatures in cantilever microbolometer FPAs. Such an engineering approach also shines a light on a certain possibility to control "unwanted" initial curvatures in many other kinds of free-standing MEMS structures, such as micromirror arrays.

11:00am **MN-MoM9 Using Geometric Moiré to Measure the Deformation in Polymeric Nanostructures***, **Y. Zhao**, X. Zhang,* Boston University

This paper demonstrates a novel approach to measure the deformations in polymeric nanostructures. To our knowledge, it is the first effort to use geometric moiré technique into polymeric nanostructures. This approach has significance in the development of various biological microsystems comprising polymeric components, especially where they serve as mechanical sensors. The application of polymer material has recently extended to mechanical sensors, which measure forces on the order of nN or even smaller. Since many polymer used for this application is transparent and not compatible for electronic read-out, current approaches for deformation measurement are mainly based on direct optical observation. However, this approach is no long appropriate for nanostructures because the nanoscale deformation can hardly be resolved optically. In this work, geometric moiré recognition was utilized by interference between the polymer nanostructures and the scanning raster of the imaging system. A PDMS substrate with nanostructures was fabricated through a nanoimprinting process. The deformation is induced by thermal expansion of the polymeric substrate upon heating. The image of the nanostructures was taken by a CCD camera and transferred to a computer for data analysis. The deformation in nanostructures can thus be predicted. The results show that although the individual nanostructures can not be clearly viewed, the geometric moiré fringes by the interference between the polymeric nanostructures and the scan raster of the scanning imaging system can be obtained by adjusting the magnification. The moiré fringes amplify the tiny dimensional changes in the nanostructures (about 6nm between neighboring structures) as the form of pitch change or rotation of the fringes. Therefore, the deformation due to the thermal expansion can be predicted with a given temperature change, which is on the order of nanometer and can not be resolved by direct optical observation.

11:20am **MN-MoM10 High Sensitivity and Broad Dynamic Range MEMS Humidity Sensor***, A. Zribi, **W.-C. Tian**, A. Knobloch,* GE Global Research Center

A new design concept of a high sensitivity and broad dynamic range MEMS-based humidity sensor is introduced in this work. A simple MEMS structure combined with ultra thin films of polystyrene sulfonic acid (PSSA, H+) is operated in two different transduction modes to enable moisture detection within the entire range -60 to 25°C Dew Point (DP), i.e. 0.04 to 100% RH (assuming an ambient temperature of 25°C). The transducer comprises two identical freestanding silicon nitride membranes and micro-conductors patterned into various geometries on top of these membranes. Only one of the two membranes is coated with a 50 to 200 nm-thick PSSA film. When used in resonant mode, the MEMS transducer is sensitive to mechanical outputs from the PSSA film (gravimetric and stress/strain) and the overall sensor output is a shift in the resonant frequency of the nitride membrane. These high performance resonant sensors provide high resolution (2 ppm), low non-linearity (0.14%), low hysteresis (0.07%) and high sensitivity (70 Hz/°C DP) and is designed for moisture detection between 0.04 and 50% RH. However, when operated in calorimetric mode, the differential heating power induced by the heat of adsorption/desorption of moisture from the PSSA film is used to measure moisture between 50 and 100% RH. The PSSA adsorbent transfer function for thermal and mechanical transductions are analyzed in this work. We will also discuss the design, modeling, and optimization of the transducer in both operational modes and the advantages of this design approach over the state of the art in terms of performance and reliability.

11:40am **MN-MoM11 Vacuum Packaging MEMS Devices***, R. Patel, M.U. Pralle, E.A. Johnson, **A.C. Greenwald**,* Ion Optics, Inc.

MEMS devices have unique packaging considerations to allow for mechanical motion. Vacuum packaging is required to reduce atmospheric drag for high frequency motion, to reduce thermal convection, etc. To achieve stable low pressures material selection is of paramount importance. Low temperature options are not suitable for very long-term reliability so that sealing materials are limited to impervious ceramics or metallic composites. Ion Optics has developed a MOEMS infrared gas sensor using photonic crystal technology.@footnote 1@ Operation requires a high temperature thermally isolated filament and vacuum packaging. Experiments in vacuum packaging this device have been performed for both single die in leadless chip carriers (LCC) and with wafer level packaging (WLP). Both low-melting point solders and ceramic frits have been tested for sealing. Different getter materials to reduce initial pressure and improve long-term reliability were also tested. The electrical performance of the heater is a sensitive function of package

pressure.@footnote 2@ Devices were calibrated prior to sealing and then used as gauges to measure internal package pressure during the sealing and operation. Pressures down to 10mtorr were achieved. Process optimization studies included time-temperature profiles for bake-out using an RGA to study gas evolution from package and chip materials. Additional post-sealing studies broke packages in a sealed chamber and measured evolved gas composition and volume. Despite pre-bakeout at over 200C, water vapor was the highest volume gas retained/evolved, followed by oxygen, carbon dioxide and nitrogen. The minimum pressures attained in WLP were lower than that for LCC. @FootnoteText@ @footnote 1@ Nicholas Moelders, et. al., Mat. Res. Soc. Symp. Proc. Vol. 729, paper U5.2(2003).@footnote 2@ Nicholas Moelders et. al., Mat. Res. Soc. Symp. Proc. Vol. 782 paper A5.32(2004).

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