

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 Young's 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 Young's 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 an atomic force microscope (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 CH₄@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

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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, SiO₂, when exposed to air. The lubrication of MEMS materials requires both the replenishment of SiO₂ from the worn surfaces and the application of a lubricant film that will minimize wear. Organosilanes, R_nSi(OR)_{4-n}, such as triethoxyphenylsilane, (C₆H₅)₃Si(OCH₂CH₃)₃, are proposed as vapor phase lubricants that can perform both functions. The surface chemistry of triethoxyphenylsilane on Si(100) and SiO₂ surfaces has been studied, using temperature programmed reaction spectroscopy and Auger electron spectroscopy. This chemistry was compared to that of tetraethoxysilane, (CH₃CH₂O)₄Si, which is widely used as a precursor for SiO₂ film deposition. Triethoxyphenylsilane decomposed readily, depositing SiO₂ and graphitic carbon on Si(100) and SiO₂ 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 β-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 SiN_x/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 SiN_x layer by PECVD. The thickness of the Al layer was 200 nm and that of the SiN_x 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 350°C resulted in less deflected pixels, while a treatment at 375°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 longer 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. 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

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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).

MEMS and NEMS

Room 207 - Session MN-MoA

Materials and Processes for Bio-MEMS and Bio-NEMS

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

2:00pm MN-MoA1 Detection of Specifically Bound Biological Mass with Resonant Nanobeams and Nanochannels, S.S. Verbridge, J.M. Moran-Mirabal, Cornell University; D.M. Tanenbaum, Pomona College; H.G. Craighead, Cornell University

Nonlithographic techniques have been used for the fabrication of two types of nanostructures, used for detection of biological molecules. Polymeric electrospun fibers with dimensions on the order of 100 nm have been used in combination with photolithography to define free standing beams and channels, made of silicon nitride and glass, respectively. Beams are made by using electrospun fibers as etch masks, and channels by using fibers as a sacrificial core. Critical dimensions of both types of structure are hence determined by polymer nanofiber sizes. Nitride beams with resonant frequencies above 10 MHz, and quality factors above 10,000 have been used as binding sites for biological molecules. Nonspecific binding of proteins such as streptavidin to entire beams, as well as targeted binding using specific thiol linkages on gold binding sites (also defined with photolithography), have both been explored. These free standing nanobeams have been operated in resonance for the detection of the bound biological mass. Suspended glass channels have been used to observe fluorescence from labeled cellulase enzymes, at the single molecule level. Directions for using suspended nanochannels to do mechanical mass detection are also being explored, to make this sort of resonant nanostructure based mass detection technique more compatible with natural fluid systems of biological interest.

2:20pm MN-MoA2 Optically Driven Nanomechanical Resonant Structures for Detection of Single Molecules, B. Ilic, Y. Yang, K. Aubin, R. Reichenbach, J. Huang, Cornell University; S. Krylov, Tel Aviv University, Israel; H.G. Craighead, Cornell University

Resonant nanoelectromechanical systems (NEMS) are being actively investigated as sensitive mass detectors for applications such as chemical and biological sensing. NEMS devices, made by lithographic techniques, can be formed in highly uniform arrays in a form that can be readily integrated with motion transduction and microfluidic systems. The types of materials that can be structured in this way have low mechanical losses providing a high mechanical quality factor of the oscillators and therefore well defined resonant frequencies. The very specific resonant frequencies and small mass of the oscillator allows for detection of small amounts of additional bound mass. Experimental investigations illustrate that the ability to engineer nanoscale features on the surface of NEMS devices, combined with localized chemical functionalization, allows for specificity and calibration of these devices as detectors. In our work, we have detected the binding of functionalized 1578 base pair long double-stranded disulfide modified double stranded DNA molecules to nanomechanical oscillators by measuring the resonant frequency shift due the added mass of the bound molecules. The resonant frequency of individual oscillators in an array of resonator devices was measured by thermo-optically driving the individual devices and detecting their motion by optical interference. Localized binding sites created with gold nanodots create a calibrated response with sufficient sensitivity and accuracy to count small numbers of bound molecules. The number of bound molecules on each device was quantified as proportional to the measured frequency shift with a proportionality constant determined experimentally and verified by modeling of the mechanical response of the system. For the smallest and most sensitive cantilevers the mass sensitivity was 194Hz/attogram.

2:40pm MN-MoA3 Biofabrication: Enlisting Biological Materials for Fabrication, G.F. Payne, L.Q. Wu, H. Yi, W.E. Bentley, J.N. Culver, University of Maryland Biotechnology Institute; G.W. Rubloff, R. Ghodssi, University of Maryland

INVITED

Biological materials offer unique properties that facilitate fabrication. Well-known are the self-assembly properties of biological materials that enable the bottom-up self-fabrication of nano-scale structures (e.g. nanowires and nanotubes). Yet, biological materials offer additional properties. They can be acted upon by enzymes enabling highly selective biocatalysts to be enlisted for enzymatic-assembly. And, biological materials often possess stimuli-responsive properties that enable a range of external stimuli to be enlisted for directed-assembly. We are studying the stimuli-responsive

amino-polysaccharide chitosan as a versatile interface material. Chitosan's pH-responsive electrostatic properties allow its directed assembly (i.e. electrodeposition) in response to localized electrical signals that can be imposed from electrodes. Chitosan's directed-assembly can be controlled by controlling deposition conditions, and high lateral resolutions have been observed when the electrical signals are imposed from micropatterned electrodes. Once neutralized, the chitosan deposit is stable (chitosan is insoluble under neutral and basic conditions) although it can be resolubilized by washing with mild acid. In addition to its stimuli-responsive properties, chitosan also offers chemical properties that permit the facile conjugation of proteins and nucleic acids to previously-deposited chitosan. These chitosan-bound proteins and nucleic acids can confer important functional properties (e.g. recognition, catalysis and binding). We are particularly interested in using the hybridization capabilities of chitosan-bound nucleic acids to serve as "nucleation sites" for the self-assembly of higher-ordered structures. Together, the results demonstrate that chitosan's unique properties enable the integration of biological materials for biofabrication at the micro- and nano-scale.

3:20pm MN-MoA5 Toward a Chitosan-Based Micromechanical Biosensor, S.T. Koev, M.A. Powers, H. Yi, R. Ghodssi, University of Maryland, College Park

In this work, the electrically deposited polysaccharide chitosan is used to biofunctionalize a microcantilever biosensor which detects the presence of target molecules on the chitosan as a shift in the resonant frequency of the cantilever. We have previously demonstrated the use of chitosan for spatially selective assembly of various biomolecules and now extend its functionality to a micromechanical sensor. Chitosan offers significant advantages over other materials commonly used for immobilization of biomolecules. The electrodeposition of chitosan would allow facile patterning of different probe biomolecules in sensor arrays. Additionally, chitosan's surface roughness, which can be controlled by the deposition conditions, leads to a large effective surface area for target molecule coupling. The microcantilever consists of layers of chitosan (100 nm), Cr/Au (110 nm), and Si@sub3@N@sub4@ (500 nm) fabricated on a Si substrate. The Au layer is used both for chitosan deposition and electrostatic actuation. The cantilever's resonant frequency is measured by actuating it at different frequencies and recording the amplitude with an optical profilometer in dynamic mode. Amine terminated ssDNA probe molecules are coupled to the chitosan amine groups using glutaraldehyde as a crosslinker and are hybridized with their complements. Resonant frequency measurements are performed after each of the following steps: chitosan deposition, addition of probe DNA, and addition of complementary target DNA. The data are analyzed to extract the surface mass density of DNA immobilized on the chitosan. The detailed fabrication, characterization, and measurement results will be presented.

4:00pm MN-MoA7 Polymeric Intermediate Layer Bonding in Micro/Nano Devices at Low Temperature for Bio-MEMS/NEMS Applications, M. Dhayal, Dongshin University, South Korea

In this study using low pressure plasma polymerized thin intermediate layer bonding process the silicon-to-silicon and glass-to-glass substrate bonding in micro/nano devices was successfully carried out. This process has advantage for bonding of glass and silicon types of substrate materials at low bonding temperature up to 130°C. The bond strength was more than 2 MPa for an about 100 nm intermediate plasma polymerised acrylic acid, p-xylene, styrene, 1-vinyl-2-pyrrolinone and allylamine intermediate layers on glass and silicon substrates. The intermediate plasma polymerised thin layer bonding process was also tested for continuously more than 24 hours with changing the room temperature from 25 to 35 °C and bonding does not show any problem. This bonding process has advantage in the micro/nano devices applications in biology where the control of surface properties is required and also this process allows the device to be reusable. In this study the fabrication of bio-MEMS was carried out using plasma polymerisation process with optical lithography, wet and dry etching techniques on silicon/glass substrate. An asymmetric electrode array used for micro pump in micro fluidic device with small electrode (4 Åµm wide) separated from the large electrode (20 Åµm wide) by 20 Åµm and 6 Åµm gaps in both sides respectively.

Monday Afternoon Poster Sessions, October 31, 2005

MEMS and NEMS

Room Exhibit Hall C&D - Session MN-MoP

General Aspects of MEMS and NEMS Poster Session

MN-MoP1 AFM Studies of Conditioner Thickness Distribution and Binding Interactions on Hair Surface, B. Bhushan, N. Chen, The Ohio State University

How common hair care products, such as conditioner, deposit onto and change hair properties are of interest in beauty care science, since these properties are closely tied to product performance. The binding interaction between conditioner and hair surface is one of the important factors in determining the conditioner thickness distribution, and consequently the proper functions of conditioner. In this study, AFM is used to obtain the local conditioner thickness distribution, adhesive forces and effective Young's modulus mapping of various hair surfaces. The conditioner thickness is extracted by measuring the forces on the AFM tip as it approaches, contacts, and pushes through the conditioner layer. The effective Young's moduli of various hair surfaces are calculated from the force distance curves using Hertz analysis. The binding interactions of different silicones on the hair surface, as well as their effect on the effective Young's modulus of the hair are also discussed.

MN-MoP2 Plasma Enhanced Chemical Vapor Deposition of Low Stress Silicon Nitride Using Diethylsilane as Precursor, L.M. Fischer, S. McColman, B. Szeto, K. Westra, S. Evoy, University of Alberta, Canada

The stable surface and high stiffness-to-density ratio of silicon nitride offer interesting advantages over regular silicon for the production of high-frequency and high-quality nanoelectromechanical (NEMS) resonators. Such machining however requires a mechanical material possessing very low residual stress. Silicon-rich low-stress silicon nitride is typically produced using silane/nitrogen or silane/ammonia as precursors. Silane is however highly flammable and thus poses significant safety hazard. In addition, typical low-stress films produced by these methods may still contain residual stress levels exceeding 100 MPa. We here report the PECVD of silicon nitride using the relatively safer diethylsilane (DES) as precursor. We also report the control and reduction of residual stress in the films through post-deposition anneal. Compressive residual stress in the as-deposited material ranged from 555 MPa to 1GPa as the NH₃:DES ratio varied from 1:1 to 16:1, while nitrogen content increased and carbon content decreased over the same range. This correlation is related to the increased formation of N-H radicals within the films. Compressive residual stress also increased from 558 MPa to 849 MPa as the deposition temperature was varied from 240 C to 315 C. Such temperature dependence is in turn attributed to an increased densification of the deposited films. A post-deposition anneal in inert nitrogen at temperatures of 500 to 600 C however relieves the stress and enables its control from the compressive to the tensile range. Tensile stresses as low as 50 MPa have been achieved. While hydrogen desorption is believed to be responsible for this change, XPS analysis also provided evidence of the formation C-N bonds in the annealed films. We will report a complete analysis of the formation, stoichiometry, and stress relief in these films. We will also present the machining and characterization of NEMS resonators in this low-stress material.

MN-MoP3 DNA Detection System using a Microcantilever, K.-A. Yoo, Myongji University, Korea; K.-H. Na, Lite-on Technology Corp. Korea; S.-R. Joung, C.J. Kang, Y.S. Kim, Myongji University, Korea

We propose a novel detection system for analysis various biotinylated DNAs effectively with a microcantilever. The microcantilevers were fabricated employing surface micromachining technique that has attractive advantages in terms of cost efficiency, simplicity and ability of fabricating in array. The fluid cell system for injection of bio-molecular solution is fabricated using a polydimethylsiloxane (PDMS) and a fused silica glass. The microcantilever is deflected with respect to the difference of the surface stress caused by the formation of self-assembled bio-molecules on the gold coated side of the microcantilever. It can detect various biotinylated DNAs according to the specific interactions between the streptavidin and individual DNA sequencing of biotinylated DNA. We confirm that the deflections of bending-up or bending-down of individual microcantilevers are occurred by the bio-molecule adsorption. The microcantilever detected protein A and DNA due to the specific interaction between protein A and DNA. The principle of the interaction is a self-assembly between the bio-

molecules. The microcantilever can be widely used to detect various bio-molecules including specific DNA and can be utilized as a bio-sensor.

MN-MoP5 Novel Fabrication Method of a Master Structure for Replicating an Optical Device Including Vertically Curved Structures, M.W. Lee, K.J. Lim, C.H. Choi, S.B. Jo, S.G. Lee, Inha University, Korea; S.G. Park, Inha University, Korea, Republic of; B.H. O, Inha University, Korea

Replication process is a good way to fabricate a passive optical device. Silicon based fabrication technology provides an efficient way to fabricate a master structure with optically smooth surface roughness. As silicon based technologies are often 2 dimensional processes, replication process requires a sophisticated fabrication steps for a master structure. For that reason, a vertically curved structure which is essential for a passive optical device is hard to fabricate. Some special processes, such as x-ray lithography, laser ablation, and gray-scale mask can overcome the conventional 2-dimensional fabrication process. But the processes need additional process steps, machines and masks. This study demonstrates easy way for fabricating a silicon master structure with vertically curved mirrors. This fabrication method is roughly divided into two steps. At the first step, a silicon wafer was deeply etched by using ICP system, to form the waveguide structures in the master structure. The vertically curved mirror structures at the each ends of the waveguides, are formed by using photoresist reflow process of the second step. After the master fabrication, the master shape was transferred to a PDMS mold. Replication process was carried by using UV curable polymers, and successful vertical redirection of lights at the curved structure was observed with a CCD device. The surface roughness of the replicated structure was also measured, and an optically smooth surface roughness was observed. Detailed fabrication steps and the fabricated device characteristics will be discussed.

MN-MoP6 CO Gas Sensor based on a Doped ZnO Film with a Microhotplate/Floating-Gate MIS Structure, W. Calleja-Arriaga, Inaoe Mexico, MEXICO; J. De la Hidalga-Wade, Inaoe Mexico; A. Heredia-Jimenez, Upaep Puebla-Mexico; G. Rosas-Guevara, I. Juarez-Ramirez, C. Zuñiga-Islas, N. Carlos-Ramirez, P. Alarcon-Peña, L. Tecuapetla-Quechol, M. Escobar-Aguilar, J. Silva, Inaoe Mexico; J.L. Gonzalez-Vidal, Citis-Uaeh Mexico; M.A. Reyes-Barranca, M.L. Olvera, A. Maldonado, Cinvestav Mexico

Doped and undoped zinc oxide (ZnO) single thin films, used as the active element in a gas microsensor, is presented in this work. The gas sensor arrangement is based on a double polysilicon micro-hotplate (MHP) and a polysilicon floating gate MIS transistor (FG-MIS). The ZnO films were doped with 6% of either copper, chromium, or gallium. The ZnO film, with an active area of 80x80 microns, was deposited onto a polysilicon plate that forms the gate of the MIS transistor. This sensing section is heated by a U-shaped polysilicon stripe, which is located beneath the polysilicon plate and electrically isolated from it by nitride/oxide films. The microhotplate is thermally isolated using a deep cavity micromachined in the silicon substrate, and mechanically supported by four polysilicon arms. The sensing film induces a charge in the floating-gate in such a way that the channel conductance is modulated. The sensor structure was characterized by detecting carbon monoxide (CO) at 300 °C. Finally, a complete procedure of fabrication of this sensor structure will be presented at the conference.

MEMS and NEMS

Room 207 - Session MN-TuM

Micro and Nano Fabrication Techniques for MEMS & NEMS

Moderator: A.V. Sumant, University of Wisconsin

8:20am **MN-TuM1 Nanomanufacturing Using Nanotemplates for Directed Assembly of Nanoelements, A. Busnaina**, Northeastern University; *J. Mead*, University of Massachusetts Lowell; *G. Miller*, University of New Hampshire; *C. Barry*, University of Massachusetts Lowell **INVITED**

The electronics industry is looking for new nanoscale technologies that will be energy efficient with high performance, scalable with gain and operational reliability at room temperature that are preferably compatible with CMOS process and architecture. Proposed nanoelectronic devices using technologies beyond currently-deployed are many; mechanical or molecular switches, spin logic, phase logic, molecular devices, cross-bar devices, cross-net devices, etc. Manufacturing of these involves very diverse fabrication and assembly techniques that may involve top-down, bottom or both. There is a need to develop heterogeneous process integration such as combination of hierarchical directed assembly techniques with other processing techniques. High-throughput hierarchical directed assembly and nanoscale components and interconnect reliability will also be essential in going beyond silicon. Another important nanomanufacturing issue is nanoscale defect mitigation and removal and defect tolerant materials, structures and processes in addition to nanoscale metrology tools, such as in-line or in-situ monitoring and feedback. Fundamental understanding and novel technology in high rate, high volume integration and assembly of robust tools and processes are addressed. Nanotemplates and tools are used to accelerate the creation of highly anticipated commercial products and will enable the creation of an entirely new generation of applications. This requires understanding what is essential for a rapid multi-step, high volume/high rate processes, as well as for accelerated-life testing of nanoelements and defect-tolerance.

9:00am **MN-TuM3 Ion Trapping in Microfabricated Ion Trap Arrays, D. Cruz**, UCLA and Sandia National Laboratories; *M. Fico*, A.J. Guymon, R.G. Cooks, Purdue University; *J.P. Chang*, University of California, Los Angeles; *M.G. Blain*, Sandia National Laboratories

In this work we describe the microfabrication and testing of cylindrical ion trap arrays. The ion trap has become an essential tool in several areas of physical science, including mass spectrometry, atomic frequency standards, studies of fundamental quantum dynamics, and quantum information science. Many of these applications benefit from miniaturized ion traps at dimensions several orders of magnitude below the current centimeter and millimeter scale. Our design of the individual trap array element consists of two endcap electrodes, one ring electrode, and a detector/collector plate, fabricated in seven tungsten metal layers by molding tungsten around SiO₂ features (0.5 μm minimum dimension) using standard lithography and plasma etching techniques. Each layer of tungsten is then polished back in damascene fashion. The SiO₂ is removed using a standard MEMS release processes to realize a free-hung ion trap element. Common anchor points of adjacent elements allow for the entire array of traps to be operated in parallel. Four different sized traps were fabricated with inner radius of 1, 2, 5 and 10 μm and heights ranged from 3-24 μm. We focused our testing on the 5-μm sized ion trap array to trap toluene (C₇H₈), mass 92 amu. We discerned the electrical characteristics of the packaged ion trap arrays through vector network analyzer measurements. We ejected the ions by turning off the rf and noted a current signal. We were not able to fully determine that our signal was all due to trapped ions. However, we attained favorable trapping conditions such as a significant pseudopotential well and an ionization rate twice the ion loss rate determined by simulation. 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.

9:20am **MN-TuM4 Monolithic In-Plane Tunable Optical Filter, J. McGee, N. Siwak, R. Ghodssi**, University of Maryland, College Park

The development and commercialization of future communication systems and biological diagnostic equipment will benefit greatly from dense integration of optical and electrical components through the use of in-plane, guided optics. Tunable optical filters are a necessary component for wavelength multiplexing and spectroscopy. However, in-plane devices have been challenging to produce as the very advantages of guided optics, small

size and dense integration, lead to loss mechanisms that degrade the filter well below the performance of out-of-plane devices. Indium phosphide is our chosen material as its direct bandgap provides the possibility of integrated emitters and lasers while operating in the standard telecommunications bands of 1500-1600 nanometers at low loss. We recently demonstrated a functional in-plane tunable filter in indium phosphide and we have now improved its performance by utilizing a ribbed waveguide structure. The use of ribbed waveguides offers the advantage of a large beam less susceptible to divergence while providing single mode operation. In our device, an electrostatically-actuated doubly-clamped beam deflects a ribbed Bragg reflector relative to a stationary ribbed waveguide attached to a second Bragg reflector, forming a variable-length Fabry-Perot cavity. A simulation model we developed predicts a Q-factor of 90 compared to a Q-factor of 44 measured in the original device. The detailed fabrication, characterization, and measurement results will be presented.

9:40am **MN-TuM5 Cooper-Pair Molasses - Cooling a Nanomechanical Resonator with the Quantum Noise of a Single Electron Transistor, K.C. Schwab**, National Security Agency **INVITED**

We are performing ultra-low temperature experiments with a radio-frequency, nanomechanical resonator coupled to a superconducting single electron transistor, a system which has demonstrated the closest approach to the uncertainty principle for continuous position detection, and the closest approach to the quantum ground state of a mechanical system. Recently, we have used the resonator to detect the asymmetric, quantum noise of the SET, which produces the back-action required by the uncertainty principle. In addition, we have discovered an unexpected cooling mechanism, analogous to optical molasses, which is a result of resonant Josephson effects in the transistor: we have observed cooling of a 10 MHz, Q=230,000 mode from 500 mK to 100 mK. Using these techniques and devices, we are anticipating the observation of squeezed, superposition, and entangled states of a mechanical device. @FootnoteText@ @footnote 1@LaHaye, Buu, Camarota, Schwab, "Approaching the Quantum Limit of a Nanomechanical Resonator," Science 304, 74 (2004).

10:20am **MN-TuM7 Granular Adsorbent Loading and Wafer Bonding for Si Microcavity Preconcentrator, H.K. Chan**, University of Michigan; *M. Takeji*, Fuji Electric Systems; *S.W. Pang*, University of Michigan

A new technique for loading 180 to 212 μm diameter granular adsorbents and Au-Si eutectic bonding at 400 °C has been developed for filling and hermetically sealing 450 μm deep Si cavity microheaters for thermal regeneration of graphitized carbon adsorbents. This development has enabled the first wafer-level integrated, microfabricated preconcentrator for trapping parts-per-billion concentrations of volatile organic compounds (VOCs). Previous Si microheaters for preconcentrators have included anodically bonded glass and Si and Al solder bonded Si-Si using rapid thermal annealing at 850 °C, all of which were assembled at the die level due to the difficulty in loading carbon granules using dry filling methods. The newly developed adsorbent-solvent method uses the principle of solvent surface tension to confine the granules into the cavities. This method is demonstrated across a 100 mm wafer for cavities accommodating 0.8 to 1.5 mg of carbon granules. The remaining solvent in the porous carbons has to be removed to avoid outgassing during the wafer-level hermetic sealing step using Au-Si eutectic bonding. Solvent removal under vacuum and elevated temperature have been investigated for the new adsorbent-loaded cavity microheaters. The resulting bonded wafers after loading of carbons and removal of the solvent have bonding strengths of >3 MPa under a tensile load for Au-Si eutectic bonding at 400 °C. The result of the new adsorbent-solvent loading, solvent removal, and Au-Si eutectic bonding is the first wafer-level integrated microscale carbon granule preconcentrator which traps VOCs at room temperature and heats to 300 °C to thermally regenerate the carbons and desorb the VOCs for analysis with a microscale gas chromatograph.

10:40am **MN-TuM8 Copper Electroplating to Fill Blind Vias for 3D Integration, S. Spiesshoefer, S. Polamreddy, R. Figueroa, J. Patel, T. Lam, L. Cai, S. Burkett, L. Schaper**, University of Arkansas

The continued demand for lower cost electronic products with decreased size, higher performance and increased functionality require improvements in the system level integration of logic, memory, and other functional integrated circuits. The formation of vertical interconnects in silicon may be one approach to provide this integration. This method involves stacking of individual die to form a highly interconnected 3D structure. One way to create an efficient 3D stack is to place electrically conductive vias through

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the body of the silicon to bring the connections from top to bottom. Copper is the metal used to fill the through silicon via (TSV) structure because of the high conductivity and the common use in multilevel wiring. A process will be described in this paper to electroplate copper into small diameter (5-10 μm) vias of aspect ratio > 3 . The objective of this project is to develop an electroplating process to obtain a void-free copper filled blind via. Prior to plating, vias are formed by both reactive ion etch (RIE) and deep RIE processes. The resulting via profile varies depending on the etch process chosen. Vias are lined with insulation, barrier, and seed films. The insulation, SiO_2 , is deposited by plasma enhanced chemical vapor deposition (PECVD) while the barrier (TaN) and Cu seed layers are deposited by sputtering. A complete and conformal copper seed layer is essential for the electroplating process. A combination of three electroplating techniques is used in this study. They consist of optimized bath composition (additive control), fountain plating, and reverse pulse plating. The goal during electroplating is to achieve a bottom-up fill, also referred to as a super fill. The process will be described that results in void-free electroplating to fill an array of blind vias as well as the related processing issues.

11:00am MN-TuM9 A Fully Integrated Micro Plasma Electron Source in Silicon, E. Wapelhorst, J.P. Hauschild, J. Müller, Hamburg University of Technology, Germany

This paper presents the concept and the fabrication of a novel, fully integrated electron and UV light source using a micro plasma. The electron source is primarily developed for use in a micro mass spectrometer. This novel system is fabricated using standard processes in silicon e.g. DRIE based on the working principle of a micro plasma ion source as shown in [figure 1](#). Furthermore, the RF-efficiency is increased by direct RF coupling through vias. An application of this type of electron source for ionization purposes in a micro mass spectrometer has been presented in [figure 2](#) which uses the concept of [figure 1](#). The electron source consists of three units: The filament, the plasma chamber, and the electron accelerator. The pressure in the plasma chamber is set to 100 Pa. To ignite a stable RF plasma in the chamber, a current pulse is driven through the filament to free electrons while the RF signal is directly applied to the RF coupler. After ignition the filament is switched off. By applying a voltage between the extraction grid and the acceleration grid electrons can be extracted from the plasma and accelerated to a defined kinetic energy. Due to its shape, the acceleration area has a focusing effect on the electron beam. The pressure in the acceleration area is less than 1 Pa. The two described pressure regimes are installed by the extraction grid which acts as pressure aperture. The system emits an electron beam with adjustable and defined energy, e.g. a 100 μA beam and an electron energy of 70 eV. Benefits of the system are the high electron current, small dimensions (diameter of the plasma chamber is less than 1mm), the low gas and power consumption, uncritical vacuum requirements because of the small size, and the adjustable electron energy. [FootnoteText](#) [figure 1](#) Plasmagestuezte Ionenquelle in Mikrosystemtechnik fuer den Einsatz in einem MMS, P.Siebert, TUHH, 2001 [figure 2](#) A Micro Mass Spectrometer, G.Petzold et al. MicroTAS, 2001.

11:20am MN-TuM10 Deep Reactive Ion Etching of Membrane-Based Devices Using a Low-Frequency Bias, R.J. Shul, J. Stevens, R.P. Manginell, M.G. Blain, S.G. Rich, S.A. Zmuda, L.J. Sanchez, Sandia National Laboratories; M. DeVre, J. Shin, S.L. Lai, Unaxis USA Inc.

Deep reactive ion etching (DRIE) of Si or the Bosch process is essential in the fabrication of many membrane-based devices, especially chemical and biological sensors. The process relies on the ability of the DRIE process to essentially stop on the membrane film, typically a dielectric film such as SiO_2 or SiN. The ability to stop on this film is due to the high etch selectivity of Si to the membrane film. Since the SiO_2 or SiN membrane is an insulator, positive charge can build up on this film upon exposure to the plasma and notching at the foot of the feature can result. The positive charge causes ions accelerated from the plasma to assume a divergent path at the Si-insulator interface and causes preferential, lateral etching of the Si at the interface, allowing a notch to form. The notch can be several microns in both the lateral and vertical dimensions and can destroy the device. Notching can be minimized and often eliminated when a low-frequency or pulsed-bias is used. In this study, we will report on the results of deep Si etching of membrane devices using a low-frequency 100 kHz bias. We will compare the results for specific device applications where both high-frequency and low-frequency biasing has been incorporated and demonstrate the relative advantages of low-frequency techniques. 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.

11:40am MN-TuM11 Aspect Ratio Dependent Etching Lag Reduction in Deep Silicon Etch Processes, S.L. Lai, D. Johnson, R.J. Westerman, Unaxis USA, Inc.

MEMS device fabrications often involve 3-D structures with high aspect ratios. Moreover, MEMS designs require structures with different dimensions and ARs to co-exist on a single microchip. There is a well-documented aspect ratio dependent etching (ARDE) effect in deep silicon etching (DSE) processes. The ARDE effect can be manifested in two ways: firstly, the etch rate decreases as the aspect ratio increases for a specific feature; secondly, for features with different dimensions etched simultaneously, bigger features are etched at faster rates. For example, when a 2.5 μm -wide trench is etched simultaneously with a 100 μm -wide trench in a conventional DSE process, the resultant trench depth of the latter can be more than double that of the 2.5 μm -wide trench. Indeed, the ARDE effect causes many undesired complications to MEMS device fabrication. One of the approaches to cope with ARDE is to employ etch stop layers, such as oxide, to compensate the lag. However, disadvantages, such as notching at the silicon/oxide interface, emerges sometimes when an etch stop layer is used. At Unaxis, we have developed a proprietary technique to eliminate the ARDE effect encountered in DSE processes. Our novel technique is based on a new physical model for ARDE lag reduction. This paper presents the theoretical model and the experimental results on ARDE reduction. With controls over the passivation and etch steps employed in a TDM etch process, we have demonstrated that normal ARDE can be changed to inverse ARDE, while maintaining good etch profile in all features. DSE processes can be optimized such that ARDE is completely eliminated. In the experiments, the ARDE lag was reduced to below 3% for trenches with widths ranging from 2.5 to 100 μm ; for trenches with widths ranging from 4 to 30 μm , the ARDE lag was below 2%. Such results were achieved at etch rate exceeding 2 $\mu\text{m}/\text{min}$.

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