Monday Morning, November 2, 1998

The Science of Micro-Electro-Mechanical Systems Topical Conference

Room 324/325 - Session MM+PS-MoM

MEMS Processing and Deep Si Etch Technology

Moderator: L.M. Miller, Jet Propulsion Laboratory

8:20am **MM+PS-MoM1 Overcoming Barriers to MEMS Prototyping and Production**, ***D.A. Koester***, *K.W. Markus,* MCNC **INVITED**

As MEMS continues to grow and expand into new markets, there continues to be a need for low cost proof-of-concept and prototype fabrication. For the past 6 years, MCNC has provided a number of services designed to provide the domestic MEMS community with an array of fabrication and design services intended to help overcome the cost and accessibility barriers to MEMS product development. The cornerstone of this DARPA-supported program, called the MEMS Technology Network (TechNet), is the Multi-User MEMS Processes (MUMPs). MUMPs is a three-polysilicon surface micromachining process offered in a multi-user environment to offset the high cost of fabrication. Since its inception in January '92, MUMPs has fabricated over 1000 designs for more than 140 different R&D groups and has been available to the world-wide community since July of '98. The SmartMUMPS program enables electronics integration of MUMPs by way of flip chip of a standard ASIC designed with a variety of sensing blocks. LIGA technology is also made available through the program. The MEMS Technology Network also provides a spectrum of custom services to the community including deep silicon RIE, backside patterning, stock substrates and access to a Microcosm MEMCAD 4.0 seat.

9:00am **MM+PS-MoM3 Materials, Process, and Integration Issues in SiC MEMS**, ***M. Mehregany***, Case Western Reserve University **INVITED**

SiC MEMS technology holds great promise for applications which are characterized by presence of harsh environments (e.g., high temperatures, large number of vibrational cycles, erosive flows, and corrosive media). Historically, SiC research has focused on the materials and processes needed for high-temperature and high-power microelectronics. These devices require high-quality single crystal films and substrates, which lead most researchers to use 6H-SiC, since nearly defect-free wafers and epitaxial films are available. Unfortunately, high quality comes at a high price; 6H-SiC wafers are very expensive and are available only in small wafer diameters. Thus, applications for 6H-SiC devices are limited to areas which can absorb such high costs, such as (military) aircraft and spacecraft. Our work has been motivated by the necessity to develop a low-cost SiC MEMS technology to penetrate a much more diverse set of markets, including for example automotive. Additionally, we have been motivated to leverage off of the latest fabrication process technologies available from Si to push the SiC MEMS technology further, faster. As a result, we have pursued large-area substrates, i.e., 3C-SiC on Si. Unlike 6H-SiC, 3C-SiC is the only SiC polytype which can be heteroepitaxially grown on Si substrates. Heteroepitaxy on Si gives 3C-SiC a distinct advantage over 6H-SiC in terms of batch fabrication, since high quality, large-area Si substrates are readily available and comparatively very inexpensive. We have pursued the development of bulk and surface micromachining processes using 3C-SiC and poly-SiC, respectively. Heteroepitaxy of 3C-SiC on Si is attractive to MEMS not only for batch fabrication, but also for bulk micromachining. In fact, SiC is undoubtedly an excellent etch stop material for Si bulk micromachining, since Si anisotropic etchants such as KOH and EDP are impervious to SiC. We have used Si bulk micromachining techniques to fabricate a multitude of 3C-SiC structures, ranging from diaphragms for mechanical properties studies, pressure sensors, and optical transmission windows, to cantilever beams and torsional micromechanical structures. Bulk micromachining of 6H-SiC has been demonstrated by others, however the process is much more complicated and the dimensional control and etch stop capabilities are limited at this time. Unlike electronics applications which require high-quality single crystal films, MEMS is much more flexible in that structures can be fabricated from polycrystalline films. SiC MEMS is no exception. We have developed poly-SiC as a basic structural material for SiC MEMS. We have deposited APCVD poly-SiC films atop polysilicon and silicon dioxide sacrificial films on 4 inch diameter Si wafers. We have demonstrated SiC surface micromachining processes, and these have been used to fabricate the first SiC lateral resonant structures. These devices tested at temperatures up to 900C outperformed polysilicon resonators of like geometry with respect to high temperature capability. Of course, the surface micromachining technology using poly-SiC would be extendable to 6H- and 4H-SiC substrate technology, as well as integration with SiC electronics on these substrates. An overview of materials, process, and integration issues in SiC MEMS will be presented, including current device examples.

9:40am **MM+PS-MoM5 Thermally-Actuated Micro-Beam for Large In-Plane Mechanical Deflections**, ***E.S. Kolesar***, *P.B. Allen, J.T. Howard, J.W. Wilken,* Texas Christian University

Numerous electrically-driven microactuators have been investigated for positioning individual elements in microelectromechanical systems (MEMS). The most common modes of actuation are electrostatic, magnetostatic, piezoelectric and thermal expansion. Unfortunately, the forces produced by electrostatic and magnetostatic actuators tend to be small, and to achieve large displacements, it is necessary to either apply a large voltage or operate the devices in a resonant mode. On the other hand, piezoelectric and thermal expansion actuators can be configured to produce large forces and large displacements. Unfortunately, piezoelectric materials are not routinely supported in the fabrication processes offered by commercial MEMS foundries. Consequently, these limitations have focused attention on thermally-actuated devices for generating the large forces and displacements frequently required to position and assemble complex MEMS. This research focuses on the design, finite element analysis and experimental performance evaluation of a MEMS thermally-actuated beam. The motivation is to present a unified description of the behavior of the thermal beam so that it can be adapted to a variety of applications in the microsensor and microactuator arenas. A MEMS polysilicon thermally-actuated beam uses resistive (Joule) heating to generate thermal expansion and movement. When current is passed through the actuator from anchor-to-anchor, the larger current density in the released "hot" arm causes it to heat and expand more than the "cold" arm. Since both arms are joined at their free (released) ends, the actuator tip is forced to move in an arc-like pattern. Removing the current from the device allows it to return to its equilibrium state. To be a useful MEMS device, a thermally-actuated beam will need to produce incremental in-plane mechanical beam tip deflections that span 0-10 microns while generating force magnitudes greater than 10 micro-newtons. The thermally-actuated beam design was accomplished with the L-Edit software program, and the devices were fabricated using the Multi-User Microelectromechanical Systems (MEMS) Process (MUMPs) foundry at the Microelectronics Center of North Carolina (MCNC). A finite element modeling analysis was accomplished with the IntelliCAD computer program. This CAD software incorporates an MCNC fabrication process description file that generates a 3-D solid model of the thermal beam. Additionally, the thermal and electromechanical finite element analyses predicted beam tip deflections and forces consistent with experimental observations. When the "hot" arm's temperature is 600@degree@C (Joule heating), the resulting beam tip deflection is 4.55 microns. For a beam tip force of 14 micro-newtons, the displacement was calculated to be 12.9 microns. The resonant frequency, without damping, was calculated to be 74.7 kHz. The MEMS thermally-actuated beam performance was also experimentally characterized. When the drive voltage was varied between 0 and 8 VDC, tip deflections spanning 0-7 microns were observed. The corresponding tip forces spanned 0-12 micro-newtons. The resonant frequency in ambient air was 68.7 kHz. A measure of the reliability of the thermal beam was established to be greater than 2 million cycles, when continuously operated with a 60 Hz, 4-volt amplitude square wave.

10:00am **MM+PS-MoM6 Development of a Micro EHD Pump Using Laser Micro-machining**, ***C.C. Wong***, *D. Chu, D.R. Adkins,* Sandia National Laboratories

At Sandia, we are developing an active cooling MEMS device for microelectronics applications. This integrated device will incorporate a micro-pump, temperature sensors, micro-channels, and heat exchanger components into a single unit. The first step of this development is to rapidly prototype a micro-pump based on electro-hydrodynamic (EHD) injection principle using laser micro-machining technology. Two initial micro-pumps designs have examined for full fabrication. The first design has two silicon parts stacked vertically on top of each other. Gold is deposited on one side of each stacked plate to serve as electrodes for the electro-hydrodynamic pumping. A Nd:YAG laser is used to drill an array of circular holes in the "well" region of both silicon parts, leaving an open pathway for fluid movement. The Nd:YAG laser is preferred for our fabrication process than excimer laser because of a smaller up-front cost and a less potential environment, safety, and health concern with toxic gases when using excimer laser. Moreover the Nd:YAG laser will allow the operational wavelength to be converted to several frequencies from the

near infrared portion of the spectra (1064 nm @lambda@) to the ultraviolet portion of the spectra (266 nm @lambda@). After the holes are drilled, the silicon parts are aligned and bonded together with polyimide, thus becoming a EHD pump. Fluid flow has been observed when an electric voltage is applied across the electrodes. The newest design has the silicon parts which contain the flow grid oriented "back-to-back" and bonded together. This "back-to-back" design has a shorter grid distance between the anode and cathode plates so that a smaller voltage is required for pumping. A thinned Si spacer was used to maintain consistent grid distance between plates. Experimental results have demonstrated that this EHD micro-pump can generate a pressure head of about 287 Pa with an applied voltage of 120 V. @FootnoteText@ This work was supported by the US DOE under Contract DE-AC04-94AL85000.

10:20am **MM+PS-MoM7 Laminated Plastic Microfluidic Components for Biological and Chemical Systems**, ***P.M. Martin***, *D.W. Matson, W.D. Bennett, D.J. Hammerstrom,* Battelle Pacific Northwest National Laboratory

Laminated plastic microfluidic components are being developed for biological testing systems and chemical sensors. Applications include a DNA thermal cycler, DNA analytical systems, electrophoretic flow systems, dialysis systems, blood sampling, and metal sensors for ground water. This paper describes fabrication processes developed for these plastic microscale components. Most of the components have a stacked architecture, the fluid flows, or is pumped through as many as nine laminated functional levels. Functions include mixing, reaction, and detector channels, reservoirs, and detector electronics. Polyimide, PMMA, and polycarbonate materials with thicknesses between 25 and 100 µm are used to construct the components. This makes the components low cost, inert to many biological fluids and chemicals, and disposable. The components are fabricated by excimer laser micromachining the microchannel patterns and microstructures in the various laminates. In some cases, micropumps are integrated into these components to move the fluids. Vias and interconnects are also cut by the laser, and integrated with micropumps. The laminates are sealed and bonded by adhesive and thermal processes, and are leak tight. The parts withstand pressures as high as 790 kPa. Typical channel widths were 50 µm to 100 µm, with aspect ratios near 5. Performance data will be presented for the DNA thermal cycler and a voltammic chromium metal sensor.

10:40am **MM+PS-MoM8 Deep Anisotropic Etching of Silicon**, ***S. Aachboun***, *P. Ranson,* University of Orleans, CNRS, France

Dry etching of silicon has been largely studied in HDP reactors for many applications such as in Microelectronics. Moreover, deep etching is a relatively new approach that can be widely used in MEMS in the near future. However, as required depths increase, the etch rate and the anisotropy decrease radically with the Aspect Ratio (width/depth). We are interested in etching very deep anisotropic trenches (~100µm) with high Aspect Ratios (~50) and high etch rates (~5µm/mn). A HDP Helicon reactor using a SF6/O2 chemistry and a cryogenic chuck has been used for etching very narrow trenches and holes from 1,2µm to 10µm of width on n-type Si wafers with a SiO2 mask. The first results of this investigation show significant features that demonstrate the feasabilty of this method. Two microns width trenches have been etched over a depth of 50µm at 3µm/mn . The resulting profiles are highly anisotropic and the selectivity Si/SiO2 is over 500. A DOE has been set in order to evaluate the effects of the different parameters and, in order to monitor the plasma and improve the features, Langmuir probe, optical spectrometer and mass spectrometer investigations are planned.

11:00am **MM+PS-MoM9 Application of the Footing Effect in the Microfabrication of Self-Aligned, Free-Standing Structures**, ***A.A. Ayon***, *K. Ishihara, R. Braff, H. Sawin, M.A. Schmidt,* Massachusetts Institute of Technology

The footing or notching effect is observed when dry etching silicon or polysilicon layers on buried dielectric films.@footnote 1@ This effect is usually considered an undesirable feature for most applications, although it is frequently small in conventional reactive ion etching (RIE) tools due to the low density of the plasmas utilized. However, with the new generation of inductively coupled plasma etching tools the notching effect can extend laterally several microns depending not only on operating conditions but also on the aspect ratio@footnote 2@ and extent of overetching time. The suppression of this effect depends in a critical manner on achieving a balance between etching and deposition of passivating films.@footnote 3@ Deviations from such balance promote the appearance of grass or even excessive deposition of passivating films. We review the dependence of footing effect on etching conditions in a time multiplexed deep etcher (TMDE) and suggest specific operating conditions to preclude the appearance of notching even when overetching for as much as 85%. Additionally, we introduce the application of the footing effect in the microfabrication of free-standing structures, by demonstrating the micromachining of self-aligned, wafer-free electrostatic actuators for which etching, releasing, ashing and passivating (dielectric isolation) were done in the same piece of equipment. All processes needed to produce cantilevered structures are done in situ using VLSI compatible plasma chemistries only. The measured pull-in voltage for a 1000 µm cantilevered beam, of the order of 85 V, agrees with predicted values. The novel low-temperature, soft-mask scheme presented here, is compatible with other VLSI processes and can be easily integrated in the microfabrication of intelligent sensors and actuators. This robust new concept allows unparalleled fabrication simplicity while permitting the fabrication of sctructures and devices in an efficient and timely fashion. Electrostatic actuators with or without interdigitated fingers, valves, pumps and relays, to name but a few, are applications that immediately benefit with this technique. @FootnoteText@ @footnote 1@G. S. Hwang and K. P. Giapis, J. Vac. Sci. Technol., B 15 (70) 1997. @footnote 2@T. Nozawa, T. Kinoshita, T. Nishizuka, A. Naral, T. Inoue and A. Nakaue, Jpn. J. Appl. Phys., 34 (2107) 1995. @footnote 3@J. P. Chang, Ph. D. Thesis, Massachusetts Institute of Technology, 1997.

11:20am **MM+PS-MoM10 Test Structure Experiments and Modeling of Very Deep Dry Etching Processes for MEMS Applications**, ***S. Abdollahi-Alibeik***, *J.P. McVittie, K.C. Saraswat,* Stanford University

One successful approach for getting the desired high (~ 4µmm/min) etch rates for MEMS device fabrication is separating the etch and passivation steps in order to eliminate the interference in chemistry. The focus of this work is on the understanding and modeling of the very deep (>100µmm) trench etch process based on this approach. Experiments were done to investigate different aspects of both deposition and etch phases. C@sub 4@F@sub 8@ gas was used for the deposition phase. The deposited material is a CF@sub x@ polymer. It was observed that the deposition rate is highly dependent on the ion flux and ion energy received by the surface. This can be modeled as an increase in the effective sticking probability of the deposition species. While polymer deposition in an overhang test structure is not that conformal, the rate of passivation does not change when the trenches become very deep. The above model and the fact that the trench sidewalls receive little ion flux can explain this discrepancy. In addition, ion reflection also appears to be important since sidewall deposition shows a dependence on the opposite sidewall. For the etch phase SF@sub 6@ gas was used. Lag experiments show that the transport of the etchant species down the trenches depends on the deposition phase. The lag was higher for a larger ratio of etching to deposition time. The fact that ion bombardment of the CF@sub x@ polymer releases F atoms can be the reason for this change in lag behavior. Incorporation of the model into the SPEEDIE profile simulator will be shown.

11:40am **MM+PS-MoM11 Pattern Shape Effects and Artefacts in Deep Silicon Etching**, ***J. Kiihamaki***, *S. Franssila,* VTT Electronics, Finland

Deep silicon etching in inductively coupled plasma (ICP) reactor offers high etch rate, nearly vertical profile, and good selectivity against most common masking materials. Crystal orientation independent ICP etching can replace area consuming KOH wet etch in many micromechanical applications. We have etched various test structures with patterns of different sizes and shapes, using different etch chemistries and etch times. The widths of etched patterns range from submicron to over 100 µm, the etched depths were up to 500 µm. Long narrow features are etched faster than wide short features, indicating three dimensional nature of RIE-lag. Aspect ratio dependent etch rate is also present, further complicating design rule - process interactions. The difference in etch rate of a rectangular hole with respect to a trench of same width increases with aspect ratio and can be up to 20%. Typically narrow trenches have positive angled sidewalls and as trenches get wider the profile turns into retrograde, which implies serious limitations to device design. Positive or vertical profiles can be achieved if etch rate is lowered to 1-2 µm/min. Amount of etchable area also affects profile. Coalescence of closely spaced trenches into a larger feature (due to retrograde profile and/or undercutting) causes both etch rate and profile to change. Large area structures connected to narrow trenches assist the etching of the narrow trenches over considerable distances. To fully utilise the benefits of ICP etching, the design rules must be tailored for each application, because of various pattern shape effects.

The Science of Micro-Electro-Mechanical Systems Topical Conference

Room 324/325 - Session MM+VT-MoA

Vacuum MEMS and Microanalysis

Moderator: C.C. Wong, Sandia National Laboratories

2:00pm **MM+VT-MoA1 Polysilicon Sealed Vacuum Cavities for MEMS**, ***J.D. Zook***, *W.R. Herb,* Honeywell*; Y.C. Ahn, H. Guckel,* University of Wisconsin **INVITED**

Sealed vacuum cavities are highly useful in silicon-based micro-electrical-mechanical structures (MEMS). They serve as the reference chambers for absolute pressure sensors and provide enclosures for high-Q mechanical resonators. A process for fabricating sealed vacuum cavities in polysilicon was developed and described by Burns and Guckel in 1988.@footnote1@ The cavities are produced by the sacrificial etching of SiO@sub 2@. The vacuum is generated by the out-diffusion of hydrogen following the polysilicon sealing step. As an additional precaution the devices are coated with silicon nitride. The process was first applied to the fabrication of piezoresistive pressure transducers with a polysilicon diaphragm and a vacuum cavity used as a pressure reference. In 1989 a multi-level polysilicon process was used to fabricate resonant microbeams and to demonstrate that high mechanical Q values require a hard vacuum inside the cavity.@footnote 2@ The micromachined polysilicon resonant microbeams are sensitive strain transducers that provide the basis for temperature, pressure, strain, acceleration and vibration sensors. The polysilicon microbeams are fabricated monolithically on single crystal silicon microstructures, are sealed high vacuum shell enclosures and are characterized by high mechanical Q, typically between 20,000 and 100,000, with recent values as high as 220,000. Two devices have been running continuously for 7 years with no observable change in Q, i.e., no change in the vacuum level. The most recent use of the vacuum encapsulation process has been for fiber optic sensors which combine the advantages of silicon microfabrication with those of optical fiber communication.@footnote 3@ The microbeams are optically excited into resonance by either an optothermal mechanism or a photovoltaic mechanism. They can be driven by modulated light or can be self-resonant. The vibration of the beam modulates the light reflected back into the fiber, which is then detected using a photodetector. Fiber optic sensors also have advantages for aerospace because of their light weight and EMI immunity. A network of 16 optically resonant microbeam temperature sensors driven and read by the same laser was recently demonstrated. Optically driven self-resonant microbeams have been operating continuously for 4 years without measureable change in Q. The most recent demonstration of the vacuum integrity of the polysilicon cavities has been the high temperature operation of the microbeams. Operation up to 510 C for several hours resulted in no loss of vacuum as evidenced by the Q of the resonators after they were returned to room temperature. Thus polysilicon-based vacuum-encapsulated devices are potentially suitable for fiber-optic-based sensors that withstand harsh environments, including high temperature. The value of Q is determined not only by residual gas in the cavity but also by the end losses and by electrical losses induced by the vibrating polysilicon capacitor composed of the microbeam and the bias electrode. By measuring Q as a function of dc bias, the electrical contributions to Q can be subtracted, providing an upper limit on the partial pressure of residual gas in the vacuum cavity. @FootnoteText@ @footnote 1@D. W. Burns, Ph. D. Thesis, Dept. Mat. Sci., UW, Madison, WI (1988). @footnote 2@J. J. Sniegowski, Ph. D. Thesis, Dept. Nuc. Eng. and Eng. Phys., UW, Madiso n,WI (1989). @footnote 3@J. D. Zook, D. W. Burns, W. R. Herb, H. Guckel, J. W. Kang and Y. C. Ahn, S ensors and Actuators A52 (1996) pp. 92-98.

2:40pm **MM+VT-MoA3 Wafer Level Vacuum Packaging for MEMS**, ***R.W. Gooch***, *T.R. Schimert, W.R. McCardel, B.A. Ritchey,* Raytheon Systems Co.

Many types of MEMS devices require a vacuum environment for operation. Some such as uncooled bolometer IR detectors and imagers, and resonant reed devices require 10 mTorr or lower for optimal performance. Packaging cost associated with traditional materials, packages, and processes needed to achieve the vacuum requirements remains the primary barrier to high volume products. Wafer level vacuum packaging transfers the packaging operation into the wafer fab. It is a product neutral enabling technology for commercialization of MEMS for home, industry, automotive, and environmental monitoring applications. Proof of principle has been demonstrated with bolometer IR detectors on 1- inch piece parts sawed from Si wafers. The lid part contained an etched cavity and was joined to the device part with a solder seal. Less than 10mTorr pressure was measured in a cell volume of 4 cubic mm. A 120x160 IR bolometer array and a resonant reed MEMS device are being designed to be packaged in wafer form using this process. Progress toward these goals will be described. This work is supported in part by DARPA/ ETO, Elias Towe program manager and Al Pisano MEMS program manager.

3:00pm **MM+VT-MoA4 A Dual Sensor Vacuum Gauge: Advanced Micromachined Thin Film Pirani Sensor Combined with a Piezoresistive Sensor**, ***D.H. Baker***, *R.A. Outlaw,* Teledyne Hastings Instruments*; D. Rosenblatt,* Rosenblatt Associates

A new dual sensor vacuum gauge which employs an advanced thin film micromachined Pirani sensor combined with a B ion implanted Si piezoresistive sensor has been developed. The two sensors are mounted on a single header and welded into a small volume (2 cc), 316 stainless steel envelope which can withstand an over pressure of 1000 psi. The instrument is UHV compatible and can detect pressure from 1000 Torr down to less than 1x10@super -5@ Torr. It is shock resistant, altitude insensitive, and bakeable to 250°C. The gas composition insensitive piezoresistive sensor permits cross over to the gas composition sensitive Pirani, thus establishing the calibration of the Pirani in the gas environment at the crossover pressure. Design criteria leading to the present sensor configurations are discussed. In particular, material selection and heat transfer solutions for the fully contiguous membrane and the various suspended geometric designs are presented. Membrane stress levels characteristic of each design are also discussed. Finally, thermal and electronic noise limitations are considered to establish the ultimate sensitivity of the instrument.

4:00pm **MM+VT-MoA7 The Knudsen Compressor as a Micro and Macroscale Vacuum Pump Without Moving Parts or Fluids**, ***S.E. Vargo***, *E.P. Muntz, G.R. Shiflett,* University of Southern California*; W.C. Tang,* Jet Propulsion Laboratory

Microelectromechanical systems (MEMS) are rapidly becoming integral components of space missions and are finding an increasing utilization in commercial applications. Several current lander, probe and rover missions under study at NASAs Jet Propulsion Laboratory (JPL) focus on utilizing MEMS based instruments for science data gathering. These small instruments and NASAs new commitment to faster, better, cheaper missions has brought about the need for novel approaches to satisfying mission requirements. For example, a miniaturized mass spectrometer is currently under development at JPL that is designed to provide in-situ gas composition analyses of planetary atmospheres. This device utilizes a micromachined quadrupole array to provide comparable performance to a commercial large-scale unit but with much less mass, power and volume. However, the miniaturized mass spectrometer system lacks a vacuum pump that can meet future mission requirements. One attractive candidate for a vacuum pump is the Knudsen Compressor that is under collaborative development at the University of Southern California (USC) and JPL. The Knudsen Compressor is a vacuum pump that operates on the rarefied gas dynamic phenomenon of thermal transpiration, which is the development of a pressure difference between two volumes of gas via a temperature difference between the ends of small channels joining the volumes. A laboratory-scale Knudsen Compressor has previously been tested at USC@footnote 1@ with its success leading to the design and fabrication of a micromechanical version. This device has two overwhelmingly attractive features over miniaturized or mesoscale vacuum pumps - no moving parts and no fluids. The Knudsen Compressor is applicable in MEMS instruments as well as to larger, more standard pumping applications.@footnote 2@ The paper will include calculations of pumping speed, power usage, size and ultimate pressure for several applications of the Knudsen Compressor. @FootnoteText@ @footnote 1@Vargo, S.E. and Muntz, E.P. (1997): An Evaluation of a Multiple-Stage Micromechanical Knudsen Compressor and Vacuum Pump. In: Rarefied Gas Dynamics, Proceedings of the 20th International Symposium on Rarefied Gas Dynamics, Peking University Press, p995-1000, Beijing. @footnote 2@Pham-Van-Diep, G., Keeley, P., Muntz, E.P., Weaver, D.P. (1995): A Micromechanical Knudsen Compressor. In: J. Harvey and G. Lord Ed. Rarefied Gas Dynamics, Oxford University Press, 715-721.

4:20pm **MM+VT-MoA8 Novel Microvalve with Low Leakage**, ***M. Hirano***, *K. Yanagisawa,* Nippon Telegraph and Telephone Corporation, Japan; *S. Nakano,* NTT Advanced Technologies Corporation, Japan; *M. Shoji,* Nippon Telegraph and Telephone Corporation, Japan

Microvalve, being capable of precisely controlling fluid flow, is necessary in various industrial fields such as chemical analysis. This paper reports the novel microvalve with very low leakage, which was fabricated by silicon micromachining techniques. The valve is a micromachine constructed on a silicon substrate chip, and it uses a valve cap supported by a suspension spring and a valve seat with a 50-µmm-diameter bore to control fluid flow. Normally closed valve is obtained by applying compressive stress to the suspension spring @footnote 1@. Piezoelectric actuator bends the suspension spring, resulting in opening and closing the valve. The silicon substrate chip, on which the microvalve was fabricated, was suitably mounted on the specially designed holder, to which inlet and outlet lines are connected. The leak or flow conductance of the microvalve was precisely determined by measuring the pressure change in the gas flow system designed for precisely determining the leak and flow rate @footnote 2@. The measurements show that the valve has a very low leak rate of 5.8 x 10@super -10@ Pam@super 3@/s. This reduced leakage was due to tight contact between the cap and seat, which was obtained by nanometer-scale flat valve surfaces and self-alignment of the cap and seat-bore based on the fabrication techniques we have developed @footnote 1@. It is concluded that the flat surfaces result from the flat substrate of the sacrificial SiO@sub 2@ film deposited by RF magnetron sputtering, and from the homogeneous dry-etching at our amorphous surfaces by ion-beam milling. @FootnoteText@ @footnote 1@ K. Yanagisawa, H. Kuwano, and A. Tago, Microsystem Technologies 2. 22 (1995). @footnote 2@ M. Hirano, K. Yanagisawa, H. Kuwano, and S. Nakano, Trans. IEE of Japan 117-E, 622 (1997).

The Science of Micro-Electro-Mechanical Systems Topical Conference

Room 324/325 - Session MM+NS+SS-TuM

Micro-Science and Tribology of MEMS

Moderator: N.E. McGruer, Northeastern University

8:20am **MM+NS+SS-TuM1 Making a Bridge to the Nanoworld**, ***S.R. Manalis***, *S.C. Minne, J.D. Adams, K.B. Crozier, H.T. Soh, T.A. Sulchek, K. Wilder,* Stanford University; *G.G. Yaralioglu, A. Atalar,* Bilkent University, Turkey; *C.F. Quate,* Stanford University **INVITED**

Our vision for micro-electro-mechanical-systems (MEMS) is to provide a window to the microscopic world. Scanning probe microscopes with automated cantilever arrays now image surface areas in excess of one square millimeter with atomic resolution. We will present new types of cantilevers and transducers that improve the speed, sensitivity, and simplicity of scanning probe microscopes. Samples are imaged at video rates with an integrated piezoelectric actuator that bends the cantilever over surface topography at high speeds. The deflection sensor, which consists of a micromachined light modulator, monitors cantilever bending with a sensitivity near one percent of an atomic diameter. We also present approaches for microfabricated biological sensors based on mechanical, electrical, and optical methods of transduction.

9:00am **MM+NS+SS-TuM3 Nanotribology of Vapor-Phase Lubricants and Their Potential Applications to MEMS@footnote 1@**, ***J. Krim***, North Carolina State University **INVITED**

The concept of lubricating high temperature surfaces with organic vapors has existed for at least forty years, with substantial efforts beginning in the 1980's and continuing on to the present day. Vapor-phase lubricants are advantageous for use at high temperature, as well as in situations where the vapor can be used as a reservoir for replenishment of areas where the lubricant has been depleted in the course of device operation. While work in the area of vapor-phase lubrication has to date focussed on the lubrication of macroscopic systems, vapor lubrication mechanisms may ultimately prove to be of critical importance to sub-micron mechanical systems in cases where lubricant delivery and/or replenishment by other methods proves impractical. In order to examine the viability of vapor-phase lubrication at length scales commensurate with submicron-scale machinery, we have constructed a Quartz Crystal Microbalance which operates in combination with a Scanning Probe Microscope so as to form a simple nanometer-scale mechanical system whose response to a number of vapor-phase lubricants can be monitored for nanotribological performance. Our observations of organic and water-vapor films recorded with this device will be discussed. @FootnoteText@ @footnote 1@Work supported by NSF and AFOSR

10:20am **MM+NS+SS-TuM7 Vacuum Deposited Fluorinated Alkyl Siloxane Films for Adhesion Control in MEMS Devices**, ***T.M. Mayer***, *M.P. de Boer, N.D. Shinn, P.J. Clews, T.A. Michalske,* Sandia National Laboratories

Monolayer films of polymerized alkyl siloxanes have been employed for surface passivation and adhesion control in MEMS devices. However, reproducible film formation and properties have been difficult to achieve due to process sensitivity to substrate preparation conditions, presence of small quantities of adsorbed water, and the high aspect ratio structures typical of MEMS devices. In contrast to the normal solution coating process using alkyl trichlorosilane precursors, we have developed a vacuum-based film deposition process, using volatile fluorinated alkyl trichloro silane precursors. Reproducible substrate conditions are obtained by UV-ozone oxidation followed by sequential or simultaneous exposure to the chlorosilane precursor and water vapor. Efficient transport of reactants into high aspect ratio structures is accomplished by maintaining Knudsen flow conditions at low pressures. We measure kinetics of film growth by in-situ ellipsometric and quartz-crystal microbalance techniques, and evaluate film composition and structure by XPS and IR spectroscopies. We also measure the work of adhesion and surface energy of coated cantilever beams under equilibrium fracture mechanics conditions. We compare results to uncoated structures, and to structures coated from solution with alkyl and fluoro-alkyl siloxane films. Sandia is a multiprogram laboratory operated by Sandia Corp., a Lockheed Martin Company, for the U. S. Dept. of Energy under contract DE-AC04-94AL85000.

11:00am **MM+NS+SS-TuM9 Adhesion Hysteresis of Polysilicon Beams in Controlled Humidity Ambients**, *M.P. de Boer,* ***T.A. Michalske***, *M.R. Tabbara,* Sandia National Laboratories; *R. Maboudian,* University of California, Berkeley; *T.M. Mayer,* Sandia National Laboratories

Auto-adhesion, or spontaneous sticking between MEMS structures, is currently a major limitation in bringing this new class of engineering devices to the broader market. MEMS are particularly susceptible to auto-adhesion because the structural members: 1) are constructed in close proximity to each other, 2) are highly compliant due to their extreme length to thickness aspect ratio and, 3) have large surface to volume ratios which increase the relative importance of adhesive surface forces. If the miniature structural members are brought together by surface (capillary, electrostatic) or inertial (shock, rapid air flow) forces, they may remain adhered after the external force is removed. If the structures remain adhered, bonding may increase over time, giving rise to the phenomena known as adhesion hysteresis. In this work we develop mechanical analysis for and report on measurements of adhesion hysteresis in surface micromachined polysilicon beams subject to dry and wet ambients. The electrostatically activated beams used in this study were tested directly after supercritical drying or after the application of hydrophobic molecular coatings such as octadecyltrichlorosilane (ODTS) or perfluorodecyltrichlorosilane (FDTS). Results indicate that both uncoated and coated beams show strong increase in adhesion after an incubation period in humid environments. This incubation time is shorter and occurrs at lower RH for uncoated beams than coated beams. For the case of uncoated beams, we are able to show that a model based on individual asperity contact forces can be used to predict the overall adhesion behavior in micromachined beams. The behavior of coated beams is compared with ellipsometric measurements indicating water adsorption on these nominaly hydrophobic surfaces after extended exposure at high RH conditions.

Biomaterial Interfaces Group
Room 326 - Session BI+AS+MM+NS+SS-TuA

Nanoscale to Mesocale Biomaterial Structures

Moderator: M.J. Tarlov, National Institute of Standards and Technology

2:00pm **BI+AS+MM+NS+SS-TuA1 Self-Assembly of a Multidomain Protein: Fibronectin at Lipid Model Interfaces**, ***V. Vogel***, *G. Baneyx,* University of Washington **INVITED**

Fibronectin, an adhesion protein with multiple recognition sites, mediates cell attachment to synthetic and biological surfaces. In solution, fibronectin exists in a globular state where most of its recognition sites are buried in the protein core. Surface adsorption induces conformational changes in the protein that expose many of these sites. Furthermore, it is known that on the surface of cells fibronectin assembles into detergent insoluble fibers, which are considered to be the main functional form of the protein. Fibronectin is hence a prime example of a protein with multiple recognition sites that can be regulated through environmental control. Unfortunately, the molecular pathways of activation and self-assembly are still poorly understood. We have recently found that fibronectin can self-assemble into fibrillar networks at receptor-free phospholipid monolayer interfaces under physiological conditions. This is a crucial observation since the paradigm in biology is that fibril assembly of fibronectin is mediated by membrane-bound receptor molecules. Availability of a simplified model system allows investigation of the molecular pathways by which appropriate surfaces can activate fibronectin and facilitate self-assembly.

2:40pm **BI+AS+MM+NS+SS-TuA3 Nanofabricated Substrates for Probing Single Biomolecules by Surface Enhanced Raman Scattering**, ***S. Petronis***, *L.K. Hedberg, H. Xu, M. Käll, B. Kasemo,* Chalmers Univ. of Technology and Univ. of Gothenborg, Sweden

The effect of Raman scattering enhancement when coherent laser light interacts with molecules attached to rough surfaces and microscopic metal domains has been known for more than two decades and is called Surface Enhanced Raman Scattering (SERS). The intensity of the Raman signals for such molecules is frequently enhanced by a factor 10@super 5@-10@super 6@ at best.@footnote 1,2@ However recently much larger enhancement factors, in the range 10@super 14@-10@super 15@, have been observed for molecules adsorbed on colloidal silver particles of specific dimensions.@footnote 3,4@ This giant enhancement allows the recording of vibrational spectra from a single molecule for the first time, instead of the ensemble averaged spectra from many molecules, which are normally obtained in optical spectroscopies. Here we report on an attempt to use nanolithography to fabricate structures of silver in the size range 100 - 200 nm and having different shapes in order to explore the size and geometry dependence of the SERS effect. Microfabricated structures which give the highest enhancement could be used for probing different biomolecules and perhaps designing a biosensor. SERS active substrates were prepared as arrays of silver particles on a Si wafer. Within each array the silver particles had a constant shape, size and separation. Three particle shapes (circular, triangular and square), two particle sizes (100 nm and 200 nm), and five different particle separations (10, 50, 100, 150 and 200 nm) were produced by electron beam lithography with a double-layer resist system and "lift-off" procedure. A reference area of uniformly deposited Ag film mimicked an infinite silver surface. The final structures and the chemical composition of the silver particles were characterized by Scanning Electron Microscopy (SEM) and Auger electron spectroscopy (AES), respectively. Preliminary Raman scattering experiments have been performed on the dye-molecule Rhodamin 6G adsorbed on the nanofabricated substrates. A giant enhancement of the Raman signal was observed on all patterns, but not on the Ag film or the Si surface. @FootnoteText@ @footnote 1@M.Moskovits, Rev. of Mod. Phys., vol. 57, No 3, 1985, pp 783-826 @footnote 2@A.G.Mal'shukov, Phys. Rep., vol 194, Nos 5&6, 1990, pp 343-349 @footnote 3@K.Kneip et al., Phys. Rev. Lett., vol. 78, No 9, 1997, pp1667-1670 @footnote 4@S.Nie, S.R. Emory, Science, vol. 275, No 21, 1997, pp 1102-1106

3:00pm **BI+AS+MM+NS+SS-TuA4 Nanostructured Surfaces for Biorecognition - A Novel Templating Approach**, ***H. Shi***, *B.D. Ratner,* University of Washington

Materials that specifically recognize proteins may find a variety of applications in separations, sensors and medical materials. Molecular imprinting provides an intriguing approach to plastic antibodies against small molecules, but the use of proteins as templates has been less successful in making protein recognition materials. In this study, nanostructured surfaces with tailored protein-binding cavities are prepared by an imprinting technique based on RF-plasma deposition of organic thin films. A polysaccharide-like surface with protein-imprinted nanopits allows only the template protein to fill the pits, and to bind strongly, because the nanopits are complementary to the template protein in shape and in the distribution of functional groups. The bound protein in its pit is prevented from exchange with protein in the solution due to a strong binding and steric hindrance, while the non-template protein that is weakly adsorbed on the surface is displaceable. Atomic force microscopy (AFM) and transmission electron microscopy (TEM) showed that nanometer-sized pits, in the shape of imprinted proteins, were created on the surfaces of our protein-imprinted polymer films. Imprinting fidelity was confirmed by AFM analysis of imprints of monodisperse colloidal gold nanoparticles. Electron spectroscopy for chemical analysis (ESCA) and time-of-flight secondary ion mass spectrometry (TOF-SIMS) indicated that template proteins were washed off the surfaces of protein imprints while sugar molecules were covalently incorporated. Radiolabeled -protein adsorption showed that a protein imprint recognized its template protein from a binary mixture with a high specificity. This study illustrates a novel templating strategy for biological molecules that can be exploited for fabrication of biorecognition materials.

3:20pm **BI+AS+MM+NS+SS-TuA5 Sensing and Analyzing Single Molecular Interactions with Microfabricated Devices@footnote 1@**, *J.-B.D. Green,* ***G.U. Lee***, Naval Research Laboratory **INVITED**

There is an intense effort to create new tools for manipulating and characterizing single macromolecules because of the power that these techniques can bring to the analysis of biological macromolecules. Due to the high force and displacement sensitivity of the atomic force microscope (AFM) it has been used to measure inter- and intramolecular forces between model ligand-receptors, i.e., streptavidin-biotin, complimentary strands of DNA, and biologically relevant supra-molecular structures, i.e. titin. With the success of these measurements, there are efforts to obtain even more detailed force measurements and to establish these techniques in the biotechnology laboratory. Our efforts focus on: 1. Designing force transducers with force (10@super -12@N), time (10@super -5@s) and spatial (10@super -9@m) resolutions that push the thermal noise envelope. 2. Developing immobilization strategies that produce more reliable force measurements. We will discuss two new microfabricated devices under development in our laboratory. The first microfabricated apparatus offers an excellent platform for detailed measurements of intermolecular interactions and possibly even analysis of combinatorial arrays. The second is an ultra-sensitive detector based on piezoresistive force transduction and magnetic microparticles. The future of these and similar devices will be considered. @FootnoteText@ @footnote 1@This work has been conducted in collaboration with Alexey Novoradovsky, Jonah Harley, Mohan Natesan, Steven Metzger, David Baselt, and Richard Colton.

4:00pm **BI+AS+MM+NS+SS-TuA7 Nanomechanical Properties of Cellular Components Determined by Interfacial Force Microscopy**, ***P.R. Norton***, *K de Jong, J.F. Graham, N.O. Petersen,* University of Western Ontario, Canada

The cell membrane is the contact surface between the cell's internal environment and the outside world. Increasingly it is recognized the there is strong active coupling between mechanical properties and cellular functions in properties such as locomotion and adhesion and in cytoskeletal diseases such as muscular dystrophy.@footnote1@ There is therefore an urgent need to understand the mechanical properties of cells and cellular subcomponents at length scales << 1µm. We will describe our initial experiments to achieve this goal. We have used three different imaging techniques in our investigation of the nanomechanical properties of larynx cells. First, immunofluorescent labelling was used to permit visualization of specific cell components in the confocal microscope, for example to determine whether the cell nucleus was removed in a shearing process. The same cell was then imaged in the atomic force microscope (AFM), permitting identification of components involved in motion such as microspikes. The nanomechanical properties of cells were then studied by nanoindentation using the interfacial force microscope (IFM).@footnote2@ While we have not yet succeeded in imaging and measuring the same cell used in the confocal and atomic force microscopies, we have demonstrated the feasibility of our approach and have obtained quantitative force-distance curves on different regions of a single cell fixed in paraformaldehyde, sodium periodate and lysine, which cross-links the proteins. From these data we can derive the elastic modulus, hardness etc of the specific region of the cell. The modulus of such a cell was ~ 3GPa, comparable to a soft polymer. Similar measurements are planned on unfixed cells. @FootnoteText@

@footnote1@Chen, C.S., et al. Science 276, 1425 (1997) @footnote2@Warren, O.L., et al. Physics in Canada 54, 122 (1998)

4:20pm **BI+AS+MM+NS+SS-TuA8 Unbinding Force of NTA-M@super 2+@--Histidine Complexes. The His-Tag Immobilization Force***, J.G. Forbes, P. Yim,* University of Maryland, College Park

A sequence of six or more histidines will bind tightly to a Cu, Ni, or Co complex. The compound typically used to immobilized the metal is N-(5-amino-1-carboxypentyl)iminodiacetic acid (NTA). Most proteins will not bind to the complex unless there is a sequence of histidines, which is readily added using recombinant DNA techniques. The histidine tag may be removed from the metal complex with a high concentration of imidazole or by protonating the histidines at a pH below 6. We have studied the the unbinding strength of this interaction with the atomic force microscope (AFM). To perform this measurement, we have functionalized silicon nitride AFM tips with NTA-M@super 2+@. A glass slide was coated with recombinant DNAse I with a his-tag on the C-terminus. Unbinding force measurements were made in phosphate buffered saline (PBS) to reduce electrostatic interactions. We find that the unbinding force for the NTA-M@super 2+@/His-tag interaction to be ca. 85~pN for each of the metal complexes. Interestingly, 0.5~M imidazole does not remove the interaction, but only changes the distribution of the measured forces. This is a result of the non-equilibrium condition of the tip being forced into the protein coated surface. The interaction is almost completely removed by lowering the pH to 5.0 where the histidines are protonated and can no longer coordinate with the nickel. The remaining interaction forces are due to the histidines which are exposed when the tip presses into the surface. These results provide a quantitative measurement of mechanical strength of binding of proteins to surfaces functionalized with NTA-M@super 2+@.

4:40pm **BI+AS+MM+NS+SS-TuA9 Sieving of DNA Molecules in Nanofluidic Channel***, J. Han, H.G. Craighead,* Cornell University

Entropic trapping and sieving effect of long DNA molecules was studied in variable thickness nanofluidic channels. We used photolithography and etching techniques to define fluid channels on Si wafers, and anodic bonding method to seal the channel with a thin pyrex glass coverslip. The channel consists of alternating regions with two different channel thicknesses(~100nm and 1.6μm). We studied electrophoretic motion of lambda phage DNA in this channel by epi-fluorescence microscopy. Since the radius of gyration of a typical long DNA molecule is larger than the smaller gap of the channel, the shallow part of the channel can be an entropic barrier for DNA motion.Therefore, DNA molecules were retarded when they entered into the thin region from the thick region. We measured the mobility of DNA molecules in these channels and observed that below a certain electric field, mobility of DNA molecule decreased to near zero drastically, showing that DNA molecules be entropically trapped and sieved. The threshold electric field was mainly dependent on the geometry of channel(e.g. gap size) and the length of DNA driven. This suggests a new type of separation device for DNA and other polymers.

5:00pm **BI+AS+MM+NS+SS-TuA10 Detection of Molecular Ion and Quantification of Pentapeptide on Plasma Hydroxylated Fluoropolymer by Time of Flight Secondary Ion Mass Spectrometry***, J.A. Gardella, L.M. Sun,* State University of New York, Buffalo

Abstract: Poly(hexafluoropropylene-co-tetrafluoroethylene) (FEP) was modified by a hydrogen/methanol radio frequency glow discharge plasma. Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS) was employed to characterize the modified FEP surface and three pentapetides (YGGFM, YGGFL, YIGSR) which were microsyringe deposited on the modified FEP film. New fragments of OH (CF@sub 2@)n in negative ion SIMS of the modified FEP film indicated that -OH functional group had been incorporated on the FEP surface after plasma treatment. In the positive ion SIMS of three pentapeptides on the hydroxylated FEP film, protonated molecular ions were dominant signals from the peptides whereas not many fragments were observed either from the peptides or the impurity. Sodium and potassium adduct molecular ions were detected as well as oxidized protonated molecular ion of YGGFM in the positive ion SIMS spectrum. Negative ion SIMS of YGGFL yielded a deprotonated molecular ion. The mixture of these three pentapetides was also studied by TOF-SIMS. The relative intensity of protonated molecular ions of YGGFL, YGGFM and YIGSR showed the possibility of quantification on the hydroxylated fluoropolymer by TOF-SIMS. As a study of substrate effects, TOF-SIMS spectra of these peptides on oxidized Ag substrate were recorded. Comparing SIMS results of pentapetides on Ag and on modified FEP film, fewer fragments occurred from the FEP film than that from the Ag substrate. A substrate like the FEP fluoropolymer might be beneficial for the quantification of peptides because of the intensity of parentlike species in SIMS measurement.

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