

Plasma Science

Room 103 - Session PS1+MM-MoM

Science & Technology of Microplasmas and MEMS Processing

Moderator: M.G. Blain, Sandia National Laboratories

9:40am **PS1+MM-MoM1 The Challenges of Plasma Etching in MEMS Processing**, *G.R. Bogart, J.T.C. Lee, A. Kornblit, H.T. Soh, K.E. Teffau, F.P. Klemens, J.F. Miner*, Agere Systems

INVITED

The rapid advancement in semiconductor technology has allowed for the design and manufacture of more complex microelectromechanical systems (MEMS). Tiny gears and simple microchannels have yielded to more complex integrated systems on a single chip. The applications of this new technology span multiple disciplines and accounts for the wider acceptance of these systems in the market place. While there are numerous methods to generate these micromachines, dry etching provides a level of manufacturing control that wet etching cannot deliver. Additionally, processes that were once limited to wet etching are now being asked of dry etching due to the added control. As an example, large ultra-thin membrane generation, while generally limited to wet etch processes, is now a possibility using dry etching techniques. For optical telecommunications components, the use of thin, silicon on insulator (SOI) wafers allows one to easily combine bulk micromachining with surface micromachining to generate well supported free standing structures. The new requirements that are being placed on dry etching processes have created issues that need to be handled in creative ways. Increasing aspect ratios with 90 degree sidewall angle specifications are competing against demands for higher etch rates, uniformity, selectivity, and other processing metrics. This paper will address some of the challenges that lie ahead for dry etching in the MEMS area.

10:20am **PS1+MM-MoM3 Maskless Etching of Silicon using Patterned Microdischarges**, *K.P. Giapis*, California Institute of Technology, U. S. A.; *M. Sankaran*, California Institute of Technology

Hollow cathode microdischarges have gained recent attention for their high-pressure operation and intense UV radiation. Due to their non-Maxwellian electron energy characteristics, they are capable of producing excited states such as excimers and radicals. For this reason, these discharges could serve as a source of reactive species for materials applications. In this talk, we will present the operation of CF₄/Ar microdischarges and their potential use in silicon etching. Because of the ability to form discharges in small holes and lines, we have used devices as stencil masks to transfer patterns directly into bare substrates. Devices employed were fabricated in copper-polyimide structures with hole diameters of 200 μm . Discharges in flowing gas mixtures (25 sccm CF₄ / 75 sccm Ar) were operated at 20 Torr with DC voltages less than 400 V and currents between 0.01-1 mA. Optical emission spectroscopy was used to detect the presence of etchants such as fluorine radicals. To etch n-type silicon (100), the 2-layer structure was patterned and pressed against the substrate. With the silicon as the cathode of the device, etch rates were found to be larger than 7 $\mu\text{m}/\text{min}$. SEM images showed profiles with a peculiar shape attributed to the expansion of the plasma into the etched void. The plasma expansion was also monitored by I-V characteristics which showed an approximate linear increase in discharge current during the etch time. This technique has also been applied to etching arrays of multiple holes and lines with similar resulting etch rates and profiles. Maskless pattern transfer in this dimensional range presents an alternative to laser drilling and ultrasonic milling.

10:40am **PS1+MM-MoM4 Efficiency of Microfabricated ICP Sources**@footnote 1@, *F. Iza, J.A. Hopwood*, Northeastern University

Recently a micromachined 5 mm-inductively coupled plasma (ICP) source and its use in optical spectroscopy have been reported.@footnote 2,3@ The performance of this device in terms of ion density and power efficiency was poorer than expected in comparison with larger ICP systems. A simple model for micro-ICP sources suggested that increasing the frequency of operation and the coupling between the source and the plasma could lead to improved performance. New microfabricated devices operating at higher frequencies (690 MHz-818 MHz) and with improved coupling coefficients have been fabricated and characterized. Argon plasmas have been generated between 100 mtorr and 12 torr and have been sustained with as little as ~100mW. Probe measurements have been carried out to determine the ion density and electron temperature versus coupling

coefficient, frequency, pressure and power. The electron temperature increases from 3 eV to 4.5 eV as the pressure decreases from 0.4 to 0.1 torr (53.3~13.3 Pa) independently of the frequency of operation and power absorbed by the device. Improved coupling coefficients lead to ion densities of $9 \times 10^{10} \text{ cm}^{-3}$ at 400 mtorr while consuming only 1W. This ion density is three times larger than in previous micro-ICP sources under the same conditions. Increasing the frequency from 690 MHz to 818 MHz, however, does not increase the efficiency as predicted by previous models. A new model that incorporates the power dependence of the plasma resistance will be presented to explain this behavior. @FootnoteText@ @footnote 1@This work is supported by the NSF under Grant No. DMI-0078406. @footnote 2@J.Hopwood, O. Minayeva, and Y. Yin, "Fabrication and characterization of a micromachined 5 mm inductively coupled plasma generator", J. Vac. Sci. B 18, 2446, (2000). @footnote 3@O. Minayeva, and J.A. Hopwood, "Optical Emission Study of a Microfabricated Inductively Coupled Plasma", AVS 47th International Symposium, Paper MM-WeM4.

11:00am **PS1+MM-MoM5 Microhollow Cathode Discharge Flow and Stability**, *D.D. Hsu, M.A. Nierode, D.B. Graves*, University of California, Berkeley

The microhollow cathode (MHC) is a geometry used to sustain atmospheric-pressure glow discharges. Flowing gas through an array of MHCs could be used to process surfaces. For example, nitrogen gas can be flowed through a microhollow cathode discharge (MHCD) in order to incorporate nitrogen onto a polymer, such as polyethylene terephthalate. Convective gas flow through the MHCD is found to affect the stability of these discharges. For example, helium flow greater than 300 sccm through a 200 μm hole at atmospheric pressure allows the MHCD to be sustained at a lower power than a stagnant helium discharge. In addition, the neutral temperature, measured by optical emission spectroscopy, of a helium-nitrogen discharge decreases when going from a stagnant discharge to one with gas flow. Higher flowrates of nitrogen through the hole cause the current to transition from a direct current to a pulsing current. The pressure drop across the hole and the gas flowrate suggest that Poiseuille flow can be used to model flow through an MHC. With pressure, peak temperature, and power deposition data, a fluid model of the discharge can help determine the spatial extent and temperature profile of the discharge. We will discuss the stability limits of these microplasmas as a function of power, pressure, gas flow, and gas composition.

11:20am **PS1+MM-MoM6 Experimental and Numerical Model Investigations of Miniature Microwave Plasma Sources**, *D. Story, T.A. Grotjohn, J.A. Asmussen*, Michigan State University

In the past, the challenge in microwave plasma research was to develop techniques that provide high ion and free radical densities uniformly, over large and ever increasing process areas. Since scale-up was usually an important issue when considering industrial applications, the study of very small microwave plasmas, on the order of a few millimeters, was rarely done. Recently, interest in the development of systems on a chip, MEMS and their related micro system applications, has suggested the possibility of numerous applications for mini and micro plasma sources. Accordingly, this investigation is devoted to the development and the understanding of the behavior of very small microwave plasma sources. We have constructed two microwave plasma systems that create and allow for the experimental investigation of millimeter size plasmas. Plasma are generated across a wide range of input parameters, including pressure variation from below 1 Torr to 1 atmosphere, input power at 2.45 GHz from one watt to 100 watts, and a variety of gas mixtures including argon, nitrogen and hydrogen. Microwave plasma of various sizes (volumes) and aspect ratios are studied. Plasma density, size, shape, ignition, and emission spectra are monitored during each experiment to characterize the miniature plasma over the operating range. Companion global model and two dimensional numerical models will be developed and used to further understand the operation of miniature microwave plasma sources. The experimental and modeling results will identify the experimental operating regime necessary to excite and maintain stable, high density, miniature microwave plasma sources and will also identify the important figures of performance, such as electron temperature versus pressure/power and absorbed power densities versus pressure and plasma size.

11:40am **PS1+MM-MoM7 Potential and Current Profiles of Nitrogen Gas DC Microplasmas**, *C.G. Wilson, Y.B. Gianchandani, A.E. Wendt*, University of Wisconsin-Madison

We have recently reported on@footnote 1@ DC microplasmas which have been generated between patterned thin-film metal electrodes on the

surface of a wafer. Typical operating pressure and power density are in the range of 1-20 Torr and 1-10 W/cm² at super 2, respectively. The plasma extent can be varied from 1 cm by variations in the electrode area, operating pressure and power. Silicon etch rates of 4-17 µm/min have been achieved. This technology allows multiple independent etching microplasmas to be operated on a single silicon wafer, enabling parallel or consecutive processing. Applications for this include trimming of electronic and micromechanical components, ranging from resistors to resonant gyroscopes. In this paper we will report on characteristics of microplasmas generated by co-planar in-situ electrodes. Breakdown voltage has been found to differ from the Paschen curve, being more uniform over a wider range of pressures. Contour plots of the floating potential of microplasmas have been measured, and the bulk of the voltage drop in the plasma column has been found to be proximate to the cathode. The floating potential is non-uniform at equal heights over the cathode and is lowest close to the center of the electrode. The height of the plasma column is found to scale with operating pressure, ranging in height from 3000-900 µm as pressure changes from 1.2-6 Torr. The internal voltage drop in the plasma column is considerable, and varies with the power density and pressure. At lower pressures, the current is found to be denser at the outer edges of the electrodes, and at higher pressures the current moves to the inner edges, becoming more uniform as the power density increases. We explore the effects of these results on silicon etching performance. @FootnoteText@ @footnote 1@ C.G. Wilson, Y.B. Gianchandani, "Silicon Micromachining Using In Situ DC Microplasmas," Journal of Microelectricalmechanical Systems, Mar. 2001, pp. 50-54.

Thin Films

Room 123 - Session TF+MM-MoM

Thin Film Sensors

Moderator: D.L. Pappas, Duracell

10:20am **TF+MM-MoM3 MEMS Device Platforms as Research Tools for Developing Improved Sensing Films, C.J. Taylor, S. Semancik, R.E. Cavicchi,** National Institute of Standards and Technology

Gas sensing characteristics of metal oxide films are dependent on the preparation method used in their fabrication. To optimize sensing film performance, one must understand how processing parameters influence composition and microstructure, and then correlate these changes with changes in the selectivity, sensitivity and stability of a sensor. We have been using arrays of microhotplates, MEMS devices fabricated with individually addressable heaters and sensing contacts, for both combinatorial studies and gas sensing. The short thermal time constant of the microhotplates makes them excellent microsubstrates for materials research where rapid heating and cooling during deposition are desired (heating rates of 10@super 5@ - 10@super 6@ °C /s are possible). Experiments have been performed using 4- and 16-element arrays as microsubstrates for CVD processing of titanium oxide and tin oxide using the single source precursors titanium(IV) nitrate, titanium(IV) isopropoxide and tin(IV) nitrate. Sensing films have been deposited both isothermally in the temperature range 100 to 450 °C, and using variable temperature deposition. Variable temperature deposition was achieved by applying triangle or square waves of varying frequency and amplitude to the heater. Film microstructure was examined by FESEM and its composition measured by EDS. We report on correlations between processing method, film microstructure and temperature dependent sensing performance for toluene, methanol, isopropanol, carbon monoxide, acetone, and other compounds.

10:40am **TF+MM-MoM4 Correlation Between Gas Response of MIS Field-Effect Sensors and the Bond Strength Between the Metal and the Insulator Layer of the Device, A.E. Åbom, L. Hultman, M. Eriksson,** Linköping University, Sweden

Chemical gas sensors based on the field-effect are used in so called electronic noses as a powerful tool for various applications. The response mechanism is, however, not fully understood. In this work we monitor the material properties in order to understand the sensor properties. The sensors used in this work are Metal Insulator Semiconductor field-effect capacitors. The metal, Pt in this case, is grown by dc magnetron sputtering with varying growth parameters, with the Ar pressure ranging between 3 and 60 mTorr. The response to H@sub 2@ can be described by three steps, @footnote 1@ dissociation of H@sub 2@ molecules on the Pt surface, transport of H atoms through the Pt film and adsorption of H (at the metal-oxide interface) as polarized species (either as dipoles or as

charged species). The polarized H affects the electric field as a shift in the applied voltage. This voltage shift increases with increasing hydrogen concentration in the ambient and reaches a saturation value depending on the amount of adsorption sites at the interface and on the magnitude of the polarization. We have found that the largest obtained voltage shift varies with the deposition process. The lower the saturation response is, the stronger the film is adhering to the substrate, as measured with e.g. scratch adhesion tests in a Hysitron TriboScope. From in-situ XPS studies it is found that no chemical reactions occur between Pt and SiO@sub 2@. We will discuss how the varying bond strength between the two materials is caused either by mechanical interlocking or electrostatic forces. We will further elaborate on whether the amount of adsorbed H at the interface changes between the different samples due to a varying electron density @footnote 2@ at the interface, or if the separation between the charges in the dipole layer is varying. @FootnoteText@ @footnote 1@ Lundström K.I., Shivaraman, M.S., Svensson, C.M., J. of Appl. Phys. 46(9) 1975 @footnote 2@ Norskov, J.K. Phys. Rev. B 26 (6) 1982.

11:00am **TF+MM-MoM5 On the Ammonia Response Mechanism of Field-effect Gas Sensors with Thin Pt Gates, M. Löfdahl, M. Eriksson, I. Lundström,** Linköping University, Sweden

The ammonia sensitivity of Pt gate field-effect chemical sensors shows a strong dependence on the morphology of the thin metal gate. Several investigations have shown that thin Pt gates are necessary to achieve high ammonia sensitivity and that thick gates show a low or even no sensitivity to ammonia. @footnote 1,2,3@ Thin thickness means in this context that the Pt gate metal has to be made so thin that the underlying oxide is partly exposed. However, there exist an optimum, and if the thickness of the metal is made too thin the sensitivity decreases again. In this contribution the morphology of the thin Pt gate has been carefully investigated and characterised by SEM and complementary TEM studies and morphological parameters have been extracted for different processing conditions of the metal film deposition. By correlating the morphological parameters to measurements of the ammonia sensitivity in inert and oxygen-containing ambient the response mechanism is attributed to the existence of Pt-SiO₂ boundaries in the metal. Further experimental investigations show that the Pt-SiO₂ interfaces acts as catalytic sites for the dissociation of ammonia molecules and diffusion of detectable species from these sites determine the response. The diffusion length of the detectable species from the dissociation sites is strongly dependent on the existence of oxygen in the ambient. In an inert ambient the diffusion length can be several mm, whereas in 20 % of oxygen it is only in the order of mm. The most likely candidate for the detectable species is atomic hydrogen. @FootnoteText@ @footnote 1@ A. Spetz, M. Armgath, and I. Lundström, Journal of Applied Physics 63, 1274-1283 (1988). @footnote 2@ J. F. Ross, I. Robins, and B. C. Webb, Sensors and Actuators 11, 73 (1987). @footnote 3@ M. Löfdahl, C. Utaiwasin, A. Carlsson, I. Lundström, and M. Eriksson, Submitted to Sensors and Actuators B (2001).

11:20am **TF+MM-MoM6 Charge Transport Mechanisms in Epitaxial Tungsten Oxide Films Used for Chemiresistive Sensors, S.C. Moulzolf, R.J. Lad,** University of Maine

Chemiresistive gas sensors fabricated from ultra-thin WO@sub 3@ films containing surface catalysts can be made highly sensitive towards a variety of target gases via manipulation of oxide surface chemistry. However, other important sensor characteristics including baseline stability, response time, and reproducibility are strongly dependent on the specific film microstructure and charge transport within the film. Using in situ Hall effect measurements coupled with structural analysis and gas testing experiments, we have determined a correlation between film deposition parameters, microstructure, and electrical response. WO@sub 3@ films were grown by rf magnetron sputtering on sapphire substrates to produce either epitaxial tetragonal or epitaxial monoclinic phases as deduced by RHEED and XRD. Exact film stoichiometries were controlled via post-deposition annealing treatments in vacuum and/or synthetic air environments. Four-point van der Pauw conductivity and Hall effect measurements as a function of temperature indicate that charge mobility is very small (<2cm@super 2@V@super -1@s@super -1@) and that polaron hopping is the dominant conduction mechanism. The conductivity of the monoclinic phase is an order of magnitude larger than the tetragonal phase and exhibits temperature dependence similar to measurements from single crystal WO@sub 3@. The mobility of the tetragonal phase increases with temperature consistent with scattering from the increased number of grain boundaries and smaller grain size as observed by STM and XRD. Extended annealing in vacuum to reduce the oxide stoichiometry causes higher conductivity and temperature dependent mobility behavior that may be

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attributed to crystallographic shear plane defects in the WO_{3-x} lattice. Upon gas exposure to H_2S or methanol, the tetragonal phase shows higher sensitivity compared to the monoclinic phase but a slower response which correlates with the lower Hall mobility.

Biomaterials

Room 102 - Session BI+MM-TuM

Biomems & Microdevices

Moderator: W. Knoll, Max-Planck-Institut für Polymerforschung, Germany

8:20am **BI+MM-TuM1 Amplification of Biomolecular Interactions into Optical Signals using Liquid Crystals on Nanostructured Surfaces, N.L. Abbott, J. Brake**, University of Wisconsin **INVITED**

Anisotropic interactions between thermotropic liquid crystals and surfaces typically cause liquid crystals to be "anchored" in one or more orientations near surfaces. In this talk, we report the use of surface anchoring phenomena involving liquid crystals for the imaging of biomolecular recognition events on surfaces. The approach is based on the observation that anisotropic forces acting between a liquid crystal and an appropriately designed surface can be perturbed by the formation of biological complexes on the surface. The change in structure of the liquid crystal near the surface is communicated deep into the bulk liquid crystal because the orientational correlation lengths of liquid crystals are typically large (micrometers). We report the design of surfaces with nanometer-scale topography and patterned surface chemistry such that protein molecules, upon binding to ligands hosted on these surfaces, trigger changes in the orientations of 1-20 micrometer-thick films of supported liquid crystals, thus corresponding to a reorientation of ~100,000-1,000,000 mesogens per protein. Binding-induced changes in the intensity of light transmitted through the liquid crystal are easily seen with the naked eye and can be further amplified by using surfaces designed so that protein-ligand recognition causes twisted nematic liquid crystals to untwist. We also use the average gray-scale brightness of the optical appearance of the supported liquid crystal to construct an optical response curve as a function of the amount of bound protein. This approach to detection of ligand-receptor binding does not require labeling of the analyte, does not require the use of a complex apparatus, provides a spatial resolution of micrometers, and is sufficiently simple that it may find use in rapid, direct-read assays performed away from centralized laboratories.

9:00am **BI+MM-TuM3 Micropatterns of Biomolecules on Silicon Hydride Surfaces, J. Pipper, U. Fritz, R. Dahint, M. Grunze**, University of Heidelberg, Germany

Biochips yield a high potential for technological progress in the fields of diagnostics, drug discovery and nanotechnology. They are usually fabricated by photolithographic and softlithographic methods, various printing techniques or the use of micro electrodes. Common substrate materials are glass-, silicon oxide- and gold surfaces. A powerful alternative to these approaches is the photochemically initiated attachment of terminally functionalized 1-alkenes onto silicon hydride surfaces accompanied by Si-C single bond formation. Although the high potential use of silicon microstructures for biosensing applications has been postulated for years, it has not been exploited yet due to a lack of functional groups suitable for the coupling of biological species. Problems in surface derivatization occur as a result of unwanted parallel chemical reactions and a possible fragmentation of the organic compounds during illumination. This dilemma has now been overcome by temporarily masking the chemical functionalities with non-photolabile protective groups. The paper reports on the spatially resolved, photochemical modification of planar and porous silicon hydride surfaces for the immobilization of DNA, proteins and cells. In combination with photoactive compounds, the method of light induced surface derivatization can also be transferred to organic materials.

9:40am **BI+MM-TuM5 Nano-Scale Effects on the Interfacial Fluidity of Organic Films, R.C. Bell, M.J. Ledema, K. Wu, J.P. Cowin**, Pacific Northwest National Laboratory

Interfaces cause fluids in nano-scale spaces to behave very differently than in bulk. We are able to spatially resolve this fluidity with 0.1 nm resolution and show how nanometer films of glassy 3-methylpentane (3MP) are much less viscous at the vacuum-interface than at the 3MP-metal interface using ion mobility to probe the spatially varying flow properties. The amorphous 3MP films are constructed using molecular beam epitaxy on a Pt(111) substrate at low temperatures (<30 K). A 1 eV hydronium ($D_{\text{sub } 3}^+$) ion beam gently deposits ions on or into the films (the latter by depositing more 3MP on top of the ions). The ion motion is monitored electrostatically as the film is heated at a rate of 0.2 K/s above the bulk glass transition temperature of 3 MP (77 K). However, the ions begin to move at temperatures as low as 40 K near the vacuum interface, well

below the bulk glass transition temperature. The viscosity near the vacuum-interface at 80 K is found to be 12 orders of magnitude lower than that expected of a bulk film. Furthermore, the fluidity perturbations were found to persist over 2.5 nm, which was determined by precisely placing the ions at increasing distances from the interfaces and monitoring the effect on the ion's mobility. Computer modeling is employed to further extract information about the nature of these films.

10:00am **BI+MM-TuM6 Interfacial BioMEMS: Bridging the Micro to the Macro, T. Desai**, University of Illinois at Chicago **INVITED**

A great deal of consideration has been given in recent years to the biological uses of micro-electro-mechanical systems (MEMS). However, such devices are not yet found in many clinical settings due to lack of appropriate interfacing between these devices and the biological world. This talk will describe approaches to engineer interfaces that enhance the biocompatibility and functionality of implantable MEMS based devices. First, the surface modification of silicon-based devices on the nanometer and micron scale to ensure device functionality and integration will be described. Such chemical modifications must be incorporated onto silicon substrates to modulate the interfacial response, while at the same time ensuring compatibility with microfabrication and micromachining processing. Secondly, microfabrication techniques that can be used to selectively attach and spatially localize chemical species in order to control interfacial reactions with the body will be discussed. By integrating surface modification protocols with MEMS processing, one can create device surfaces that interact appropriately with multiple populations of cells and the surrounding tissue. The identification of principles for engineering microdevice surfaces will aid in developing therapeutic bioMEMS, lab on a chip platforms, and drug delivery systems that can more effectively interface with the biological world.

10:40am **BI+MM-TuM8 Dynamics of Biomolecular Recognition on Calibrated Beads in Microfluidic Channels, G.P. Lopez, T. Buranda, J. Huang**, The University of New Mexico; V.H. Perez-Luna, Illinois Institute of Technology; L.S. Sklar, The University of New Mexico

We have developed a new approach for the analysis of biomolecular recognition in microfluidic systems. The method is based on real-time detection of biomolecular binding to receptor-bearing microspheres comprising affinity microcolumns. The microcolumn format ensures efficient analyte contact with receptors and rapid mixing. Molecular assemblies on microspheres can be characterized and calibrated using flow cytometric techniques prior to packing. Model assays demonstrated include direct fluorescence methods of quantitatively detecting recognition of model analytes by protein receptors and ligands displayed in well-characterized affinity matrices. We establish a model system for detection of recognition between a monoclonal antibody and the FLAG@super TM@ epitope tag. The assay can detect sub-femtomole quantities of antibody with good signal-to-noise ratio and a large dynamic range spanning nearly four orders of magnitude in analyte concentration. Kinetic and equilibrium constants for the reaction of this receptor-ligand pair are obtained through modeling of kinetic responses of the microcolumn and are consistent with those obtained by flow cytometry. Because of the correlation between kinetic and equilibrium data obtained for the microcolumns, quantitative analysis can be done in minutes, prior to the steady state endpoint of the recognition reaction. The approach has the potential to be generalized to a host of bioaffinity assay methods including analysis of small molecule analytes, protein and nucleic acid complexes, and microsystem-based multi-analyte determinations.

11:00am **BI+MM-TuM9 Microfluidic Patterning of Biopolymer Matrices for Cellular Pattern Integrity, W. Tan, T. Desai**, University of Illinois at Chicago

The ability to design and create biologically relevant patterns via microfluidic patterning on surfaces provides new capabilities for cell biology, the production of biosensors and tissue engineering. However, cellular patterns, defined by microfluidic methods, often lose integrity over time due to cell growth and migration immediately upon removal of the PDMS stamp. In this study, biopolymer matrices were used in conjunction with cellular micropatterning to control cell attachment, growth, and long-term maintenance of these patterns. The incorporation of appropriate matrix materials with microfluidic cell patterning methods results in highly compliant patterns of adherent human endothelial cells (HUVECs) and fibroblasts after several days in vitro. Furthermore, cell type and chemical components in these biopolymer matrices influence the ability of the biopolymer matrices to control cell growth, proliferation and compliance to the patterns. Cell growth and migration in micropatterned biopolymers

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such as agarose, collagen, collagen-GAG mimics, and collagen-fibronectin are quantitatively measured and compared, and cell-matrix interactions are also examined over time. Results suggest that the use of an appropriate biopolymer matrix helps to control cell growth and maintain pattern integrity for long periods of time. This is essential for conducting stable biological experiments, as well as achieving control over tissue engineering constructs with multiple cell types.

11:20am BI+MM-TuM10 High Throughput Techniques for Non Invasive Cancer Cell Detection, W.C. Wilson, L.F. Pardo, X.Z. Yu, T. Boland, Clemson University

The usefulness of patterned surfaces, which specifically bind antagonists has been recognized for a wide variety of biomedical applications ranging from drug screening to tissue engineering. Current technologies for creating patterned surfaces suffer from many drawbacks. For optimized results, technologies that are flexible, use a large number of different proteins, high-throughput and inexpensive are warranted. Ink jet technology has shown promise in meeting these criteria and commercial systems are being developed. High throughput and quantitative assaying of the patterns is equally challenging. For example, in early cancer detection, it is desirable to detect a few abnormal cells within millions of normal cells. It is unlikely that PCR based techniques or gene chips will be economically feasible tools for early detection since most of the cost will be associated with analyzing normal DNA. Economical high-throughput screening and concentration technologies may be able to discriminate and select abnormal cells for further analysis. We developed a piezo driven protein and cell printer in our laboratory, able to simultaneously deposit picoliter drops of cell or protein solutions out of nine nozzles. The printer can deliver a single cell per drop to a surface with submicron resolution. Furthermore, it is equipped with a robotic arm and conveyer belt allowing for truly high-throughput printing. Examples of its use including for anti angiogenesis drug screening will be presented. Quantitative assaying is done using a cell scanner. The cell scanner has a resolution of less than 2 μm , is fully computer controlled, high-throughput and an economically attractive when compared to epifluorescent microscopes. Results will be presented with fluorescently labeled cells demonstrating the potential of the cell scanner for high-throughput discrimination and selection of prostate cancer cells.

11:40am BI+MM-TuM11 Electrochemically-Activated Switching of Surface Chemistry Using Tethered Molecular Machines, B.C. Bunker, D.L. Huber, J.G. Kushmerick, M. Kelly, C.M. Matzke, Sandia National Laboratories; J.F. Stoddart, J. Cao, J.O. Jeppesen, J. Perkins, University of California, Los Angeles

Sandia National Laboratories is integrating "smart" coatings into microanalytical systems for transporting, separating, and detecting species such as proteins. This paper describes the first demonstration of the use of electrochemically-activated molecular machines to switch surface chemistries. The "motor" for the machines being studied consists of an open aromatic ring system (cyclobis(paraquat-p-phenylene)) referred to as the "blue-box" due to its strong optical absorption properties. Reversible oxidation or reduction of the blue box makes it attract or repel aromatic threads such as functionalized naphthalenes or tetrathiafulvalene (TTF). Researchers at UCLA have succeeded in attaching a disulfide-terminated tail to the blue box which is used to tether the blue box to gold surfaces. Ellipsometry and atomic force microscopy measurements indicate that monolayer films of the blue box are produced. Electrochemical measurements indicate that while the voltages required to reduce the blue box are similar to voltages known to induce switching of the box in solution, adsorption of naphthalene threads is irreversible. Reversible switching is only seen for TTF threads that can themselves be oxidized. Contact angle measurements show that reversible changes in surface chemistry can be induced using appropriate threads. A simple microelectronic device has been constructed to demonstrate how the molecular machines can be used to move liquids or dissolved species within microfluidic systems.

Tuesday Evening Poster Sessions, October 30, 2001

Biomaterials

Room 134/135 - Session BI-TuP

Surface Characterization and Non-Fouling Surfaces Poster Session

BI-TuP2 Short-term Oxidation of Polymer Films Deposited from Pulsed Radiofrequency Allylamine Plasmas, J.D. Whittle, G.R. Kinsel, R.B. Timmons, University of Texas at Arlington

Plasma deposited films are seen as a promising route to the synthesis of novel functional coatings for a large number of potential applications. Allylamine deposited films in particular are of great interest in the biomaterials field as surfaces for protein adsorption. Studies of the long-term aging of these plasma polymers have shown that the oxygen content of the films changes over extended periods of time. Earlier work has shown that the oxygen content of allylamine films deposited from continuous wave plasmas increased from around 2% for a fresh sample, to around 10% after a year of aging in the laboratory, with the greatest change in composition being within the first 48h. In addition, some loss of nitrogen from the films has also been observed. In this study, we concentrate on the changes in chemistry over the first few days, and in particular the first 12 hours following deposition. The surface chemistry is investigated by X-ray photoelectron spectroscopy (XPS) and Matrix Assisted Laser Desorption/Ionization Mass spectrometry (MALDI-MS). Using XPS we investigate the stability of the plasma polymer surfaces in the UHV environment using different substrates for deposition to determine what the source of the oxidative species may be. A small amount of oxygen is always present in these plasma polymers, which may be due to the unavoidable exposure to the atmosphere between completing the deposition, and insertion of the sample into the spectrometer. Further, by analyzing samples exposed to the laboratory atmosphere for specific lengths of time, we show how the surface chemistry evolves in the first few hours following deposition. We also examine the effect of plasma power and pulsing duty cycle on the post-deposition properties of the films.

BI-TuP3 Fast Impedance Spectroscopy Measurements on Supported Lipid Bilayer Membranes with and without Incorporated Ion Channels, G. Wiegand, S. Beyer, N. Arribas-Layton, P. Wagner, Zyomyx Inc.

A substantial part of the mammalian proteome is represented by proteins that are either associated or incorporated into lipid bilayer membranes. Our goal is to provide appropriate platform assays and transducer technologies for the functional analysis of membrane proteins. Our special focus is on ion channels due to their pharmacological relevance. Because ionic flux thru an ion channel generates an electrical signal, electronic transducer technologies are the most direct detection method for ion channel analysis. We developed a method of fast impedance spectroscopy that combines the power of a spectroscopic technique providing high information content with the millisecond time resolution of a fast analytical tool. In biophysical experiments, time dependent quantities such as the membrane resistance and the membrane capacity are obtained from the measured sequences of impedance spectra. Supported lipid bilayers provide membrane matrices for protein incorporation that are coupled to solid surfaces. Supported bilayer applications take advantage of the high membrane stability imparted by the solid support, and of the improved accessibility for analytical tools due to the two-dimensional geometry. As a result of the chip compatibility, supported membrane systems are potentially useful in high-throughput technologies. By application of fast impedance spectroscopy, dynamic properties of supported lipid bilayers with and without incorporated ion channels are studied during formation, relaxation and in various states of conduction.

BI-TuP5 Optical Inverted Microscope with a Scanning Near Field Optical Microscope to Study Biological Material, A. Cricenti, R. Generosi, M. Luce, P. Perfetti, ISM-CNR, Italy

A scanning near field optical microscope (SNOM) has been added to a standard inverted optical microscope with the dedicate aim of characterizing the inner parts of biological molecules. Therefore, in addition to the requirements of reliability and mechanical stability we have carefully looked to analyzing a sample with all available geometries for input/output of photons, in order to get as many information as possible. The SNOM unit consists of a support mounted on the optical microscope arm containing a piezoelectric scanner. The reflectivity of the sample can be measured by applying different methods: the sample can be illuminated on top by an external source, as well as by the optical fiber used for the

detection of the reflectivity signal. Absorption experiments can be easily performed by detecting the transmitted signal through the optical apparatus of the inverted microscope. Also fluorescence signal can be simultaneously detected. Reflectivity, transmissivity and fluorescence measurements will be presented on several biological systems, with a resolution well below the diffraction limit.

BI-TuP6 Investigation of Bone Tissues using Infrared Spectroscopic Ellipsometry, G.M.W. Kroesen, J.-C. Cigal, E. Stoffels, B. van Rietbergen, R. Huiskes, Eindhoven University of Technology, The Netherlands

Small fractures on the bone surface, called micro-cracks, are formed throughout the lifetime as a result of e.g. mechanical stress. In individuals of advanced age, these defects are no longer efficiently repaired by the organism. Increasing density of micro-crack is one of the important factors which lead to osteoporosis: the severe loss of bone mass and attendant fragility of the skeleton. The size of micro-cracks is in the order of 10 microns, and they are difficult to detect in vivo. Apart from these fractures, the chemical composition of the bone surface is expected to change in the course of ageing. Spectroscopic ellipsometry is a powerful but non-destructive technique of analysing complex surfaces, and it seems very suitable in a study of bone tissues. We developed a spectroscopic ellipsometer combined with a Fourier transform spectrometer in the middle infrared range (wavelength of 2.5 to 10 microns). This device allows to collect accurate data on the chemical composition of the bone surface. In addition, it can provide information about the surface roughness, which is useful in determining the density of micro-cracks. Ellipsometry is a purely physical method, and this novel application to the complex biological environment poses many technical challenges. We will present preliminary results on ellipsometric analysis of bone surfaces, including infrared spectra of several bone samples. In the subsequent study we will investigate how the ageing of the bone tissue is reflected by its infrared properties.

BI-TuP7 Changes in Bone Surface after Exposure to an Electric Discharge, J.H.R. Feijen, C.Y.M. Maurice, E. Stoffels, G.M.W. Kroesen, B. van Rietbergen, R. Huiskes, Eindhoven University of Technology, The Netherlands

Human bones are subject to a continuous process of regeneration. Due to mechanical stress, cracks on a microscopic scale are generated in bone tissue, but in the healthy situation these cracks are repaired before they can lead to serious damage. In the case of disturbed bone regeneration, however, due to osteoporosis, drugs that inhibit bone resorption or bone cancer, the mechanical integrity of bone is impaired by accumulation of micro cracks or large metastatic defects. Treatment of bone diseases in vivo is nowadays very difficult. We consider an alternative method of bone surface processing, using non-equilibrium (cold) electric discharges. These plasmas combine high reactivity with non-destructive character. In this study we attempt plasma treatment and observe its impacts on the surface of bone tissues. These impacts are change in roughness, etching of some layers, removal of cells, etc. Since the concept of exposing living tissues to electric discharges is new, the presented results are preliminary and the medical implications are not yet resolved. For this experiment we employ a low-pressure inductively coupled plasma (ICP), supplied with diagnostics. A Langmuir probe, an energy-resolved mass spectrometer and a Doppler shifted laser-induced fluorescence (DSLIF) techniques are used to monitor the parameters of the plasma. With an Environmental Scanning Electron Microscopy (ESEM) we record images of the surface before and after exposure to the plasma. Several gases will be investigated, like oxygen, hydrogen and argon, and plasma treatment under various conditions (varying pressure, power and electric bias) will be performed. In the continuation of this work, cold atmospheric discharges will be used for bone treatment.

BI-TuP8 Glass Ionomer Cements: Probing Uptake from Solution using Surface Sensitive Techniques, B.M. Hutton, G. Palmer, P.C. Hadley, University College London, UK; T.A. Steele, A.J. Eccles, Millbrook Instruments Ltd., UK; R.W. Billington, G.J. Pearson, Queen Mary, UK; F.H. Jones, University College London, UK

Glass ionomer cements (GICs) are dental filling materials with the ability to take up ionic species (e.g. F@super -@) from solution and store them within the cement matrix for subsequent re-release. This offers the potential for controlled drug release in-vivo. Previously, ion uptake and release by such materials has been determined primarily by measuring ion concentration in solution. Although this approach provides useful data on the concentration of ionic species as a function of time, little is learned about the mechanism of uptake and release. In the current work, X-ray photoelectron spectroscopy (XPS) and secondary ion mass

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spectrometry (SIMS) have been used to examine ion uptake. Cements were analysed after the introduction of fluoride either by doping or by immersion in KF(aq) of various concentration. Immersion of a GIC based on poly(acrylic acid) and a calcium aluminosilicate glass was found to result in the formation of a calcium and fluoride rich surface layer, while doping by mixing with KF solution during GIC preparation resulted in no such surface layer. The formation of CaF₂-like layers on immersion was entirely unexpected on the basis of previous solution-based experiments and may explain differences in measured uptake kinetics. The use of these techniques has been extended to examine the inclusion of molecular active species (amprolium hydrochloride and chlorhexidine acetate) within the GIC matrix. Both species could be detected in GIC samples irrespective of whether they were included by mixing or by immersion in solution. However, relative peak intensities indicated that the binding of the active molecule is dependent on the method of inclusion. Significant applications in the study of the uptake and release mechanisms of active species such as antibacterial and antifungal agents are envisaged. @FootnoteText@ @footnote 1@ Hadley P, Billington RW, Pearson GJ. Biomaterials 1999;20:891-897.

BI-TuP9 Morphological Analysis of the Collagen Structure of Regenerated Rat Tendons Following Laser Photo-stimulation, V. Baranauskas, Universidade de Campinas - Brazil, Brazil; N.V. Parizotto, Universidade Federal de Sao Carlos - Brazil

Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) were used to comparatively study the structure of regenerated rat tendons submitted to laser photo-stimulation after a surgical injury that extracted around 6 mm of Achilles' tendon (tenectomy) of the left forefoot. The experiment was performed using male Rattus norvegicus albinus, lineage Wistar, with body weights in the range of 250 to 300 g, and 90 days old. The healing course was monitored at 7, 14, 21 or 28 days after the injury. The animals were divided into four groups. One group was used as a control and the other three were subjected to irradiation of doses of 0.5, 5.0 and 50 Jcm⁻², respectively. A He-Ne laser of 6 mW power (L = 632.8 nm) was used. AFM and SEM images, at different magnifications, allowed the identification of the time-dependence of the recovery, measured by the organization of the collagen fibers. Comparison of natural recovery processes with the laser photo-stimulation procedures will be discussed.

BI-TuP10 Evolution of a UHV Compatible Heater for TSG Preparation, M. Hasselblatt, B. Jackson, M. Heidecker, P. Wagner, Zyomyx, Inc.

Template Stripped Gold (TSG) surfaces have been used extensively as a source for ultra-flat substrates. The preparation of these thin gold films relies on accurate temperature control. Heating in UHV / HV is always difficult since an accurate temperature measurement requires excellent thermal contact with the sample due to the lack of convection. Working with layered crystals like mica makes matters worse. Here we present the evolution of an ultra-high vacuum compatible heating plate that is optimized for the preparation of TSG. Initial versions of this heater were successfully built by casting Omegabond 600 High Temperature Chemical Set Cement into a Teflon mold including pre-coiled Omega Nickel Heating Wire and thermocouple wire. The current design features an exceptional degree of thermal homogeneity over approx. 9 square inches with variations of less than 1% and also a modular design to improve serviceability.

BI-TuP11 Imaging Biomolecules for Skin Cancer Demarcation, M.B. Ericson, A. Rosén, Chalmers University of Technology - Göteborg University, Sweden; A.-M. Wennberg, C. Sandberg, Sahlgrenska University Hospital - Göteborg University, Sweden; F. Gudmundsson, Chalmers University of Technology - Göteborg University, Sweden; O. Larkö, Sahlgrenska University Hospital - Göteborg University, Sweden

Protoporphyrin IX, Pp IX, is a photoactive porphyrin molecule formed in the cell heme synthesis. It has been shown that Pp IX is formed to a larger extent in tumor cells due to enzymatic and metabolic differences compared to normal cells. This effect can be enhanced by exposing the cells to an excess of aminolevulinic acid, ALA, a precursor in the heme synthesis. By imaging the fluorescence from Pp IX molecules in the skin, the extension of skin tumor can be visualised with respect to the enhanced Pp IX production. This technique is based on photodynamic therapy, PDT, which is a new clinical treatment for cancer that has developed over the past 25 years. In a clinical study of 40 patients with basal cell carcinoma, a malignant type of skin cancer, the Pp IX fluorescence was recorded by a CCD camera set-up. The lesions were treated with ALA cream and thereafter the fluorescence was visualised by using filtered mercury lamps as excitation light-source. The contrast in the fluorescence images was

evaluated as a function of ALA application time in order to optimise the technique. The study showed a correlation between the fluorescence images and histological pattern however the individual variations were large. Further studies are planned in order to further improve the technique.

BI-TuP12 Characterization of the Crotalus Durissus Terrificus Venom by Atomic Force Microscopy, V. Baranauskas, J. Zhao, Faculdade de Engenharia Eletrica e Computacao - UNICAMP, Brazil; D.M. Dourado, UNIDEP - Brazil; M.A. Cruz-Hofling, Instituto de Ciencias Biologicas - UNICAMP - Brazil, Brazil

Atomic Force Microscopy (AFM) was used to study the morphology of crude venom from the South American rattlesnake Crotalus durissus terrificus. The effects of the crotalic venom on humans are systemic, leading to suffocation in fatal cases due to the neurotoxic, myotoxic and coagulative action of the components of the venom. We used adult snakes from the Pantanal region, Mato Grosso do Sul, Brazil, that remained without food for 30 days before the venom was extracted. The venom was collected manually by a specialist and dried at room temperature. Atomic Force Microscopy images, obtained using low vertical forces, allowed characterization of the surface morphology of the samples at sub-micron resolution. Coiled and porous structures are observed. Characterization of the venom by AFM is potentially of great importance because it may allow the comparison of its natural components. Critical discussion of the experimental results and characterization of the samples by AFM are given.

BI-TuP13 Role of Interfacial Water Structure on the Protein Resistant Properties of Oligo(ethylene glycol) Monolayers, B. Subramanian, J. Yan, G.P. Lopez, The University of New Mexico

Understanding the mechanism of protein adsorption at surfaces is an important issue in the field of biomedical materials, cellular adhesion and clinical diagnostics. Self-assembled monolayers (SAMs) of oligo(ethylene glycol)-terminated alkanethiols on gold are known to be protein resistant and represent a good model system to study the interactions of proteins with organic surfaces. Although these SAMs are resistant to protein adsorption, the mechanism by which these monolayers prevent protein adsorption is not yet established. Recently, it was suggested that protein resistance of these monolayers is a consequence of the formation of a structured interfacial water layer, which prevents direct contact between the surface and the protein. It was further suggested that, this might be a common mechanism for other monolayers, which show resistant to protein adsorption. It has been observed that, interfacial water undergo sharp changes in its properties (e.g., density, surface viscosity) at 15, 30, 45, and 60°C. These changes are attributed to the change in the structure of interfacial water at that temperature. We examine whether the change in the interfacial water structure at these characteristic temperatures affect the protein resistant properties of these monolayers, by carrying out protein adsorption on mixed monolayers of hexa(ethylene oxide)-terminated alkanethiols and methyl terminated alkanethiol (@chi@ @sub EG6@ = 0.44) as a function of temperature. The results show that, there is a sharp change in the protein adsorption behavior at 30±1°C. Below this temperature, there is no protein adsorption and above this temperature there is approximately a monolayer of protein adsorbed on the SAM surfaces. These results strongly support the view that interfacial water structure plays an important role in the protein resistant properties of oligo(ethylene glycol) SAMs.

BI-TuP16 Oligo(Ethylene Glycol)-Terminated Self Assembled Monolayers: Protein Resistance and the Effect of Assembly Temperature, C. Boozer, S. Chen, L. Li, S. Jiang, University of Washington

The rational design of protein resistant surfaces is a critical step in the ongoing development of biomaterials and biosensors, yet we lack a fundamental understanding of how such surfaces work. Here, we report a systematic study of the behavior of oligo(ethylene glycol)-terminated self-assembled monolayers (SAMs) prepared at a range of temperatures. The monolayers were formed by self-assembly of (EG)₆-terminated thiols, in a heated (or cooled) methanol solution, on both single crystal and polycrystalline gold films. The films were characterized using atomic force microscopy (AFM), X-ray photoelectron spectroscopy (XPS), and infrared adsorption (FTIR). Protein adsorption on the OEG-terminated SAMs was studied using a home-built surface plasmon resonance (SPR) sensor. It was found that the ability of the OEG-terminated SAMs to resist protein adsorption from a buffer solution correlates with the temperature at which they were prepared. Protein adsorption studies were performed with both bovine serum albumin and fibrinogen, and in both cases we found that

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protein resistance of the films was greatly diminished by increasing assembly temperature. A possible mechanism will be presented.

BI-TuP17 Determination of Adsorption Thermodynamics for Lysine Residues on Functionalized SAMs Using Surface Plasmon Resonance, V.N. Vernekar, R.A. Latour, Clemson University

Although protein adsorption is key to many bioengineering problems, it is still not well understood. New comprehensive approaches to this problem are needed. In this study we take a systematic approach to address protein-surface adsorption by studying submolecular interactions of peptide residues with model surfaces. We hypothesize that combining the intermolecular thermodynamic contributions for peptide residue-surface adsorption with intramolecular residue-residue interactions will provide an approach to accurately predict overall protein adsorption. Accordingly, the objective of this study was to develop experimental techniques to measure residue-surface adsorption using surface plasmon resonance spectroscopy (SPR). The model residue-surface system selected for this initial study was poly-L-lysine (PL) and OH & COOH terminated Au-alkanethiol self assembled monolayers (SAMs). Preliminary studies were conducted to develop surface preparation and cleaning protocols necessary to obtain a stable SPR signal during the adsorption process. Adsorption studies were then conducted to measure the difference in signal as a function of surface functionality and PL concentration. Results show that the amount of adsorbed PL increases with increasing solution concentration, with the COOH-SAM adsorbing more PL than the OH-SAM for each concentration. These studies provide experimental data that is needed for calculating thermodynamic parameters for adsorption (Gibbs free energy, enthalpy, entropy) for each of these model systems. These values will be compared to results predicted from computational chemistry studies by others in our group for these same residue/surface systems.

Microelectromechanical Systems (MEMS)

Room 130 - Session MM-ThM

Characterization of MEMS Materials

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

8:20am **MM-ThM1 Mechanical Properties of MEMS Materials, W. Sharpe, Johns Hopkins University** **INVITED**

The "mechanical" part of "microelectromechanical systems" (MEMS) requires knowledge of mechanical properties to predict relations between forces and displacements. Young's modulus and Poisson's ratio are needed for elastic response, and the strength of the material is needed to determine the allowable forces or displacements. Tensile testing is the preferred approach for structural materials because its uniform stress and strain fields enable direct determination of mechanical properties according to their definitions. Tensile testing of small thin-film specimens presents three challenges - preparation and handling of the specimen, measurement of small forces, and measurement of strain in the specimen. The author and colleagues at Hopkins have developed techniques and procedures for tensile testing of polysilicon, silicon nitride and silicon carbide. It is easier to measure mechanical properties of MEMS materials indirectly by modeling microdevices and extracting properties. One can fabricate a comb-driven resonant structure and use the measured resonant frequency to determine the modulus. Thin membranes of different shapes can be pressurized, and the measured displacements used to determine both Young's modulus and Poisson's ratio. Cantilever or fixed-end beams can be deflected electrostatically to measure modulus. However, none of these indirect approaches permit measurement of all the three properties (modulus, ratio, strength) simultaneously as does the tensile test. This presentation summarizes the current state-of-the-art in terms of test methods and the values of the polysilicon and other materials used in MEMS.

9:00am **MM-ThM3 The Beam vs. Plate Distinction for Si Strips Mechanically Loaded in Bending, S.K. Kaldor, Columbia University; I.C. Noyan, IBM T.J. Watson Research Division**

Silicon structures used in microelectromechanical systems (MEMS) are generally anisotropic and possess dimensions that make it difficult to determine whether a beam or plate solution is more appropriate. Since a plate has an increased stiffness over that of a beam, errors of up to 10% in predicted displacements and stresses can occur if the proper bending solution is not employed. For single crystal Si samples loaded in four-point bending, we report both finite element modeling results and x-ray curvature measurements that illustrate the effects of boundary conditions (bending jig rollers used to apply displacements), specimen anisotropy, and specimen dimensions. We find that the transverse, or anticlastic, bending effects, which are ignored by 2-D solutions, should be considered as they can result in non-uniform loading across the sample width, and they are important in deciding whether a beam or plate solution should be used. While the sample's width-to-thickness ratio is typically the only criterion used to differentiate between beam and plate structures, we show that it is necessary to consider not only the sample's width and thickness but also the amount of applied bending; this was first considered by Searle¹ in 1908. We show that the Searle parameter, $\text{width}^2 / (\text{thickness} \times \text{bending radius})$, can be used to accurately differentiate between beam and plate structures. Furthermore, the difference in stiffness between a beam and a plate depends on the Poisson's ratio of the bent material. Since Poisson's ratio in Si can vary from 0.06 to 0.36 with crystallographic orientation, controlling the bending direction of a single crystal is a possible method for tailoring the specimen's flexural rigidity. ¹G.F.C. Searle, "Experimental Elasticity," 2nd ed. Cambridge UP, 1920.

9:20am **MM-ThM4 Amorphous Diamond MEMS, J.P. Sullivan, T.A. Friedmann, M.P. de Boer, M.T. Dugger, M. Mitchell, R.G. Dunn, R. Ellis, Sandia National Laboratories; D.A. LaVan, Massachusetts Institute of Technology**

Microelectromechanical systems (MEMS), including electrostatic comb drives, simply-supported beams, and tensile test specimens, have been fabricated from amorphous diamond (aD), a pure carbon material with mechanical properties similar to crystalline diamond. Measurements using aD MEMS revealed that the material has high strength (8 GPa), fracture toughness (8 MPa.m^{1/2}), and elastic modulus (800 GPa). These properties, combined with good inherent wear resistance, makes the

material useful for achieving long lifetime MEMS that have rubbing surfaces or experience impact loading. Hydrophobicity and bio-compatibility of aD were also evaluated. The water contact angle was found to range from 84° for the as-prepared MEMS material up to 94° after annealing to 850°C. The increase in contact angle with annealing is similar to that found for crystalline diamond surfaces, which is due to O desorption that leaves an H-terminated surface. The hydrophobic nature of aD greatly reduces stiction in MEMS, thus permitting release without the use of applied hydrophobic coatings or supercritical drying. Bio-compatibility was tested through the use of cultured cell growth, using bovine capillary endothelial cells, on bare and fibronectin-coated aD surfaces. Limited cell growth and adhesion was found for the uncoated aD surface, while good growth and adhesion was found for the fibronectin-coated aD. This is desirable for the creation of bioMEMS. Finally, the very high elastic modulus of this material is desirable for achieving mechanical structures with high resonant frequency. A key requirement for mechanical oscillators used for electrical signal processing is the need for high quality factor, Q. The Q for aD MEMS oscillators operating in vacuum will be reported and compared to that found for silicon oscillators. Sandia is a multiprogram lab operated by Sandia Corp., a Lockheed Martin Co., for the U.S. D.O.E. under contract DE-AC04-94AL85000.

9:40am **MM-ThM5 Fabrication Techniques and Integration Processes for a New Ultrananocrystalline Diamond (UNCD) -Based MEMS Technology and Characterization of UNCD Mechanical Properties, O. Auciello, A.V. Sumant, D.M. Gruen, J.A. Carlisle, J. Birrell, N.A. Moldovan, D.C. Mancini, M. Angadi, Argonne National Laboratory; H.D. Espinosa, B.C. Prorok, Northwestern University**

State-of-the-art Si-based MEMS components exhibit serious performance limitations due to the relatively poor mechanical and tribological properties of Si. Diamond and diamond-like materials are investigated for MEMS applications, but they also have microstructural /properties, and/or processing limitations. A novel diamond coating technology developed at ANL yields phase-pure UNCD coatings with 2-5 nm grains and smooth surfaces, in addition to hardness of 97 GPa and friction coefficient of ~ 0.01, both similar to pure diamond. The unique growth process (involving C60 or CH₄ /Ar microwave plasmas), based on C₂ dimer insertion into the growing film, results in low activation energy for growth of UNCD on various substrates down to a record low temperature of ~350 °C. We demonstrated the fabrication of high-resolution UNCD-based 2-D and 3-D MEMS components, such as micro-gears, pinwheels, cantilevers, strain-gauges, and a microturbine, via growth of UNCD on Si and sacrificial SiO₂ layers, and selective etching. UNCD coatings can be grown conformally on high aspect ratio Si structures. UNCD coatings exhibit excellent mechanical and tribological properties, in addition to extremely low threshold voltage for electron field emission, which allows to produce MEMS sensors using the uniquely combined mechanical/electron emission properties of UNCD. We will discuss fabrication issues and UNCD properties applicable to MEMS. Work supported by the U.S. Department of Energy, BES-Materials Sciences, under Contract W-31-109-ENG-38.

10:00am **MM-ThM6 Silicon Carbide Films by Low Temperature CVD for MEMS Applications, D. Gao, C.R. Stoldt, W.R. Ashurst, C. Carraro, R. Maboudian, University of California, Berkeley**

The single source CVD precursor, 1,3-disilabutane, is used to grow polycrystalline cubic silicon carbide (SiC) films for MEMS applications at temperatures below 1000 °C. Using this process, SiC films are integrated into surface and bulk micromachining technologies to obtain SiC-based micromechanical structures. SiC cantilever beam arrays and strain gauges are fabricated and used to characterize film stress and stress gradients. Also, released polysilicon microstructures are coated with thin SiC films, and exhibit superior physicochemical characteristics. For instance, SiC-coated lateral resonators are functional after HF and hot KOH treatments and display increased resonant frequencies.

10:20am **MM-ThM7 Thermal Characteristics of Microswitch Contacts, X. Yan, N.E. McGruer, G.G. Adams, Northeastern University; S. Majumder, Analog Devices, Inc.**

Electrostatically actuated microswitches and relays developed at Northeastern University are approximately 100 x 100 μm in size and have been tested beyond 10⁹ cycles with a current of 2 mA per contact. For gold-gold contacts, the microswitches fail in a permanently closed mode in less than 10 cycles for currents exceeding 300 mA. At currents of approximately 1 A, the drain electrode melts, resulting in a permanently open switch. A number of authors have reported on various aspects of heat conduction through larger contacts. Hyman and Mehregany

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have discussed the contact physics of microcontacts, and modeled their thermal behavior. @footnote 1@ However, they do not consider the effect of the thin film traces leading up to the contacts in most MEMS switch designs. Finite element modeling and experiments have been used to study the thermal characteristics of microswitches. Because of the asymmetry in the contact geometry, the highest temperature is located in the thin film contact trace rather than at the contact interface. Contributions from convection and radiation are negligible, and conduction through the gas is marginally important. The hottest spot moves away from the contact as the contact radius increases, from 0.3 μm for a 100 nm contact radius to 2.7 μm for a 500 nm contact radius. Measurements show a sharp decrease in the contact resistance at a switch voltage of about 0.08 V and a current of 0.15 A, which may be due to softening of the contact surfaces and/or removal of surface contaminants. The result is in rough agreement with the onset of softening predicted by the model. The contact trace melts at a switch current of 1 A. The melted region is between 3.5 and 6 μm away from the center of the contact, slightly further than is predicted by the model. @FootnoteText@ @footnote 1@ Daniel Hyman and Mehran Mehregany, Proc. of 44th IEEE Holm Conf. on Electrical Contacts (Arlington, VA, October 26-28, 1998, pp.133-140).

10:40am **MM-ThM8 MBE-grown Single-crystal Ferromagnetic Shape Memory Ni@sub 2@MnGa Thin Films, J.W. Dong, J. Lu, J.Q. Xie, Q. Pan, J. Cui, S. McKernan, R.D. James, C.J. Palmstrom**, University of Minnesota

Ni@sub 2@MnGa is a ferromagnetic shape memory alloy which goes through a thermodynamically reversible martensitic phase transformation and demonstrates ferromagnetic properties. In bulk, Ni@sub 2@MnGa with the stoichiometric composition has a Curie temperature ~ 376 K and the martensitic phase transformation occurs ~ 202 K. Above 202 K, Ni@sub 2@MnGa adopts a cubic L2@sub 1@ crystal structure with weak magnetic anisotropy. Below 202 K, it will transform to a tetragonal structure with greatly enhanced magnetic anisotropy. In this low-symmetry tetragonal phase, a twinning structure will be formed by three types of martensitic variants with different magnetic easy axes. External magnetic and/or stress fields can be employed to adjust the volume fraction of the twinned martensitic variants by the motion of twin boundaries. This will result in large reversible strain and this novel mechanism is thus called ferromagnetic shape memory effect. In bulk single crystals of Ni@sub 2@MnGa, strain as large as 6.1% has been demonstrated. This makes it a promising candidate for magnetic field driven actuator material. For micro-electro-mechanical-system (MEMS) actuators, conceptual designs based on single-crystal Ni@sub 2@MnGa films have been proposed. The first single-crystal growth of Ni@sub 2@MnGa thin film has been reported by the authors. @footnote 1@ The 300 Å-thick film grows pseudomorphically on a (001) GaAs substrate with a unique tetragonal structure ($a = b = 5.65$ Å, $c = 6.12$ Å). The Curie temperature was measured to be ~ 320 K. Moreover, martensitic phase transformation is observed in a partially released 450 Å-thick epitaxial film. In this presentation, we will report the growth, characterization, and patterning of 900 Å-thick single-crystal Ni@sub 2@MnGa films to elucidate the concept of using it in MEMS actuators. @FootnoteText@ @footnote 1@ J.W. Dong, L.C. Chen, C.J. Palmstrom, R.D. James, and S. McKernan, Appl. Phys. Lett., 75, pp. 1443-1445 (1999).

11:00am **MM-ThM9 Stability of Alkylsilane Monolayer Films in Humid Environments, T.M. Mayer, H.I. Kim, M.G. Hankins, M.P. de Boer**, Sandia National Laboratories

Alkylsilane monolayer films on SiO@sub 2@ are used to prevent adhesion in micromechanical (MEMS) devices. We have studied the stability of these films in humid environments, where degradation may lead to loss of hydrophobic character, water adsorption, and adhesion of MEMS components by capillary condensation. In this work we study silane monolayer films with both fluorocarbon and hydrocarbon side chains, deposited by both solution and chemical vapor deposition methods. In-situ ellipsometry and interfacial force microscopy measurements examine water vapor adsorption and its effect on adhesion and friction. Ex-situ atomic force microscopy and x-ray reflectivity measurements examine the morphology and density of the films before and after exposure. We find that chemical binding of the film to the surface is critical for its stability. Silanol films are not strongly bound to the surface and exhibit substantial water adsorption. This is accompanied by an irreversible increase in friction when probed with a similarly functionalized tip. In the presence of high humidity at room temperature, the silanol film restructures to form small droplets on the surface, leading to increased adhesion in cantilever beam MEMS test structures. In contrast, silanol films that have been annealed to react with surface hydroxyls are strongly bound to the surface and display negligible water adsorption, no effect on adhesion or friction, and no

surface restructuring after exposure to high humidity ($>80\%$ RH) for short periods (10 hr) at room temperature. Stability of these films after more severe exposure (longer times at higher temperature), mechanisms of degradation, and long-term effects on the performance and reliability of MEMS devices will be addressed. 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.

Microelectromechanical Systems (MEMS)

Room 130 - Session MM-ThA

Fabrication and Integration Processes for MEMS

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

2:00pm MM-ThA1 Integration of a Honeycomb Micromirror with a Surface Micromachined 2D Scanner for Improved Performance, P.R. Patterson, University of California at Los Angeles; **G.-D.J. Su, D. Hah,** University of California at Los Angeles, U.S.A.; **M.C. Wu,** University of California at Los Angeles

We have developed a novel fabrication process to integrate lightweight single crystal silicon honeycomb micromirrors with surface micromachined 2D scanners for improved optical flatness, compared to polysilicon alone, and improved response, compared to solid (higher mass), micromirrors. The honeycomb micromirrors are formed by silicon fusion bonding of two, silicon on insulator, SOI wafers allowing for precise control of the core and facesheet thickness, here we used 25 μm and 10 μm , respectively. The core SOI wafer is patterned with hexagonal cells of 100 μm long sides and 10 μm thick walls. Design flexibility is an inherent feature of the integrated process, core and facesheet for the honeycomb may be chosen from a wide range of commercial SOI, and the actuator and mirror are developed independently and subsequently bonded with a polymer. The electrostatically actuated 2D scanner with the bonded honeycomb micromirror has a mirror area of 950 μm x 950 μm and an optical scan angle of $\pm 6^\circ$. The reduced mass bonded honeycomb micromirror shows an increase in resonant frequency, 158Hz, over an otherwise equivalent solid bonded micromirror measured at 108Hz.

2:20pm MM-ThA2 Freestanding Microheater in Si with High Aspect Ratio Microstructures, W.-C. Tian, S.W. Pang, The University of Michigan

A micromachined gas chromatography system on a chip can be used for environmental monitoring with the advantages of high sensitivity, low power, and portability. To increase sensitivity, a preconcentrator with heating elements and adsorbents is used to adsorb gases and release them at higher concentration to the separation columns. Freestanding, high aspect ratio microstructures in Si are micromachined as preconcentrators. Heat loss to the substrate is minimized by using freestanding heaters to reduce power consumption. A high aspect ratio microheater provides large volume for high adsorbent capacity and hence high sensitivity. Dry etching of Si using etch and passivation cycles has been developed to produce 240 μm thick Si microheaters with 3 μm wide wires, achieving a high aspect ratio of 80:1. This optimized dry etching technology results in high etch rate with vertical profile for thick Si microheaters up to 535 μm . A 400 μm thick Si microheater with 100 μm wide wires, 100 μm gaps, and an area of 9 mm² has been fabricated. With the heater on 125 μm thick Si membrane, it takes 850 mW to increase the temperature by 285 $^\circ\text{C}$. The power consumption is reduced to 475 mW for the same temperature raise with freestanding Si microheater. In addition, Si microheaters consist of wires and posts with different conductivity are tested for their heating efficiency. These high aspect ratio, freestanding Si microheaters can provide high power efficiency, large adsorbent capacity, and high mechanical strength as preconcentrators.

2:40pm MM-ThA3 MEMS and NEMS Physical and Chemical Sensors: Fabrication and Integration, P.G. Datkos, Oak Ridge National Laboratory; **T.G. Thundat,** Oak Ridge National Laboratory; **M.S. Sepaniak,** University of Tennessee

INVITED

3:40pm MM-ThA6 Fabrication of Novel Si@sub 3@N@sub 4@ Micromesh Spider Web Bolometer Using Deep Trench Etching on SOI Wafer, M.H. Yun, Jet Propulsion Laboratory, Caltech-NASA; **A.M.P. Turner, J.J. Bock, J.A. Podosek,** Jet Propulsion Laboratory

Bolometers are used for sensitive detection of radiation throughout the electromagnetic spectrum, from X-ray to millimeter-wave. The sensitivity of a bolometer can be improved by reducing its base temperature, and reducing its thermal conductivity. Sub-millimeter wave bolometers have achieved a steady increase in sensitivity over the past decade. In this research, we have fabricated and developed extremely sensitive Si@sub 3@N@sub 4@ micromesh spider web bolometers for sub-millimeter astrophysics using microelectromechanical system (MEMS) techniques. The spider-web architecture provides high infrared absorption with minimal heat capacity and volume. We use silicon-on-insulator (SOI) bonded wafers,

with a 2 μm of top silicon layer, a 1 μm SiO₂@sub 2@ insulating layer, and a 350 μm of bottom silicon layer, to fabricate the devices. Using a deep trench reactive ion etching (RIE) from the bottom silicon to the insulating layer, followed by wet etching to remove SiO₂@sub 2@, a 151-element polygonal spider web array was formed on the 4" SOI wafer. We also observed that the deep trench etching may result in less surface roughness and higher conductivity in the silicon nitride supports. To achieve the best accuracy performance, e-beam lithography is also employed to form contact pad layer. Several Au depositions using photolithography processes form the absorber for optimal infrared absorption, the electrical leads which define the thermal conductance, and the wiring layer for electrical readout. Another silicon wafer is patterned and etched to rest behind the array wafer, forming @lambda@/4 backshorts for maximum optical efficiency. The use of MEMS techniques in this research has improved the sensitivity and format of bolometer arrays. The fabrications of various sub-millimeter device arrays are under development at JPL/Caltech-NASA.

4:00pm MM-ThA7 Analog Beam Steering Vertical Comb Drive MEMS Actuator, J.J. Fijol, Standard MEMS, Inc., US; **J. Prohaska, M. Smith,** Standard MEMS, Inc.; **T. Wester,** ProcessTek, US; **G.W. Tasker,** Standard MEMS, Inc.

The design, modeling, fabrication and characterization of a vertical comb drive actuator are presented. This micro-electro-mechanical device includes a rotating platform supported by two torsion springs and an integrated vertical comb drive actuator. The comb structure was etched into the underside of the rotating platform yielding a compact three-dimensional device. An Au mirror was deposited on the rotating platform and the actuator was used for single axis analog beam steering. The vertical comb design eliminates pull-in, generates large actuation forces (>500 μN) and minimizes the footprint to dimensions approximately equivalent to the size of the mirror (1750 x 2000 μm). Device fabrication required fusion bonding of two wafers; a thick (1000 μm) Si bottom wafer and a top SOI wafer. Deep reactive ion etching (DRIE) was used to etch one half of the comb structure into the substrate wafer and the other half into the handle of the SOI wafer. The mirror platform and torsion hinges were formed in the SOI wafer's device layer using a novel dry release process that eliminated stiction. A matrix of devices were fabricated with varying comb lengths, number of comb fingers and gap spacing (between the upper and lower comb fingers). Devices were operated with a single sided displacement and rotational angles of ~ 13 degrees were measured for an applied voltage of 200V. Resonant frequency measurements were also performed and the primary resonances were observed between 30 to 200 Hz. Characterization of the mirror surfaces using interferometric microscopy shows the mirror flatness to be better than @lambda@/30 (at @lambda@ = 1550 nm), yielding diffraction limited beam steering.

4:20pm MM-ThA8 An Integrated MEMS Fabrication Technology Using SU-8 Negative Resists and Conducting Polymers, S. Li, E. Smela, R. Ghodssi, University of Maryland at College Park

Tall, narrow channels are necessary for many microfluidics applications, and a simple way to fabricate such channels is desirable. In addition, electrodes are frequently required for fluid pumping. Since polymers can be inexpensive, easy to pattern, and modified to be biocompatible, our goal is to make all-polymer microfluidic systems. EPON SU-8 is a light-sensitive epoxy polymer that can be patterned using conventional UV photolithography. We have previously used it to fabricate high aspect ratio microstructures, and in this work we made channels 15-micron wide and 250-micron deep on top of a patterned gold film on a silicon wafer. Surface micromachining was used (rather than, for example, deep reactive ion etching) to achieve deep channels with straight sidewalls in one simple step. For electrodes, we are investigating the use of conducting polymers such as polypyrrole (PPy). Polypyrrole, which has good biocompatibility, was electrochemically deposited onto the patterned gold electrodes in the bottom of the channel. Thus, the microfluidic channels in the SU-8 had patterned PPy electrodes embedded at the bottom. The two plastic-based MEMS technologies were successfully integrated, demonstrating materials and process compatibility. Preliminary results and a detailed fabrication process for combined SU8 and PPy will be presented.

4:40pm MM-ThA9 Elimination of Defects on Quartz Plate Surface Induced by Deep Drying Etching and Subsequent Quartz Plate Bonding, T. Fukasawa, H. Ogawa, Y. Horiike, The University of Tokyo, Japan

Two issues were studied for fabrication of a microcapillary on a quartz plate in the Bio-MEMS chip. One is generation of the cone-like defects on the quartz bottom surface which were etched deeply with fluorocarbon plasmas. The other is less tight bonding of a pair of quartz plate which is

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performed at 1.3 MPa, RT and 24 hours following dipping them in a 1% HF solution. In the former issue, we noticed that defects were distributed randomly, nevertheless these diameters were almost same. Increasing RF-self bias voltage (Vdc) decreased the number of defects. No defect was observed on the quartz surface at high Vdc of 940V, while the masking Cr film was eroded considerably. The results imply that certain masking materials remain on the quartz surface after its finishing, while AFM and XPS measurement of the surface do not reveal the origin of materials at present. Hence, at first high Vdc of 900V added to the sample during 2 min. to remove the mask materials by sputtering, and then Vdc was decreased down to 500V to etch the quartz plate. Eventually, no defects and high etch selectivity to the Cr mask was achieved. For the latter issue, the quartz bonding mechanism was studied employing an in-situ IR-ATR spectroscopy. 10wt % HF solution was introduced between a quartz plate and a trapezoidal Ge prism whose surface was covered by a sputtered grown SiO₂ film with 70nm thickness. Elapse time dependence of IR absorption spectra was measured under 5 MPa. H₂SiF₆ was observed at the interface and this intensity decreased with increasing the pressing time. Thus the bonding mechanism was considered as follows: At first, H₂SiF₆ is produced by etching of SiO₂ in a HF solution, then it changes to SiO₂ at presence of H₂O, thereby acting as a paste layer to bind both surfaces. Based on the mechanism, high strength bonding of quartz plates was performed successfully using a H₂SiF₆ solution instead of a 1% HF one.

5:00pm **MM-ThA10 Production Plasma Etching of PZT Structures for Piezoelectric Actuators**, *L.G. Jerde, J.P. Almerico, S. Marks, P.F. Werbaneth*, Tegal Corporation

Lead Zirconium Titanate (PZT) is a Perovskite structure dielectric material that is very well suited for piezoelectric actuator applications. The film growth characteristics of PZT and its need for an oxidation barrier effectively limit the choice of electrode materials for the piezoelectric actuator structure to platinum. Neither PZT nor platinum readily form soluble or volatile reaction products. This makes wet etch patterning processes impractical and dry etch patterning processes difficult for PZT based piezoelectric actuator structures. The conventional approach used to pattern these structures utilizes ion milling. The inert ion beam in an ion milling system results in defining the structure by sputter etching material from the exposed surface. The major problem with sputter etching these structures is that the edge of the previously defined PZT layer becomes coated with sputtered Pt atoms during the patterning of the bottom platinum electrode. This results in high leakage currents and limited device performance. We have developed a plasma etch process for photoresist etch masks to eliminate the limitations of ion milling and meet all the production requirements for defining PZT based piezoelectric actuator structures. This process utilizes a patented dual frequency reactor technology, magnetically confined plasma and a combination of feed gases. This technology results in synergistically combining both chemical and sputter etching to successfully meet the requirements for defining PZT based piezoelectric actuators. We shall describe this process and its performance in this paper.

Microelectromechanical Systems (MEMS)

Room 134/135 - Session MM-ThP

Poster Session

MM-ThP1 Determination of the Young's Modulus and Residual Stress in 3C-SiC Films Using the Load-Deflection Technique, J. Mitchell, C.A. Zorman, M. Mehregany, Case Western Reserve University

Silicon Carbide is an attractive mechanical material for MEMS due to its high Young's modulus coupled with its high temperature stability and chemical inertness. SiC is also receiving attention as a material for NEMS for these same reasons. The cubic polytype (3C-SiC) can be epitaxially grown on (100)Si substrates, thus providing an excellent opportunity for measuring the mechanical properties of SiC, since test structures can be fabricated using conventional Si bulk micromachining techniques. Using an interferometric load-deflection technique applied to bulk micromachined diaphragms, we have measured the spatial distribution of the Young's modulus and residual stress of 3C-SiC films grown on large-area (100 mm-dia.)(100) Si wafers. In addition to the spatial distribution, the run-to-run variation and the variation as a function of film thickness were also characterized. In general, we found that the Young's modulus, which averaged about 360 GPa, is insensitive to location on the wafer as well as film thickness. This is in stark contrast to the residual stress, which varied by as much as 200 MPa across a 100 mm-diameter wafer, and is, in general, higher for thinner films. This paper will detail the film growth process, and test sample preparation procedure, the testing technique, and the test results for 3C-SiC film thicknesses ranging from 0.125 to 2 microns. Issues pertaining to making measurements of high modulus films such as SiC using the load-deflection technique will also be discussed.

MM-ThP2 A Trench Etching Technique Using MERIE to Fabricate MEMS Accelerometers, K.-W. Kok, National University of Singapore, TEMIC Automotive (Singapore) Pte Limited, Singapore; W.J. Yoo, National University of Singapore

Plasma etching is an important process to form deep high aspect ratio beams in fabrication of MEMS devices. Properties pertaining to the anisotropic trench etching process have been studied using SiF₄/HBr/NF₃/HeO₂ gas mixtures by a magnetically enhanced reactive ion etcher (MERIE). We investigated the taper angle and etching rate in the trenches, the dependency of the etching rates on pattern-size and open area ratio, and roughness on the sidewall. The etching masks of SiO₂ and Si₃N₄ were used. In these conditions, etching selectivities of the silicon substrate with respect to the etching mask were in the range of 60 to 120 for the SiO₂ mask and these were about three times higher than for the Si₃N₄ mask. The high etching selectivities from the SiO₂ mask resulted in the steep trench profile and this made possible to form the deep trench structures of the aspect ratio of 25. Furthermore, the open area ratio on the wafer was varied in the range of 10% to 50% to determine loading effects which are problematic in inductively coupled plasma (ICP) etching. Etching rates and their uniformity across the wafer in the ICP were known to be strongly affected by the open area ratio. We found that, in the MERIE, the etching rates remained constant and their uniformity was less than 2% regardless of the open area ratio for the all pattern sizes investigated. The surface roughness on the sidewall was maintained within 5nm after deep trench etching up to 20mm, and the electrical test proved that this was acceptable to control capacitance of the MEMS accelerometers accurately.

MM-ThP3 Micro-fabrication of a Novel n-channel Field Effect Transistor Cantilever to Sense Charge Traps, M.S. Suh, G.H. Yon, Seoul National University, Korea; Y. Kuk, Seoul National University, Korea, South Korea

We have micro-fabricated a novel n-channel field effect transistor (FET) cantilever that is proposed to sense surface potential profile in nanometer scale. Conventional techniques used in surface and bulk micro-electromechanical system (MEMS) and combined complementary metal oxide semiconductor (CMOS) process have been employed to make a novel n-channel FET cantilever made of silicon on insulator (SOI) wafers. The cantilevers with various beam lengths, width, and thickness have been fabricated and their resonance frequencies were measured. Thermal annealing after high-dose ion implantation controlled the channel length between the source and the drain. This cantilever resembles nchannel metal oxide semiconductor FET (n-MOSFET) without a gate electrode. If a biased or charged sample is positioned closed to the cantilever, it works as

Microelectromechanical Systems (MEMS)

Room 130 - Session MM+BI+NS+EL+SS-FrM

New Frontiers in MEMS: NEMS and BioMEMS

Moderator: N.E. McGruer, Northeastern University

8:20am **MM+BI+NS+EL+SS-FrM1 Optomechanical Effects in and Properties of Nanomechanical Resonant Structures**, **L. Sekaric**, **M. Zalalutdinov**, **S.W.P. Turner**, **A.T. Zehnder**, **J.M. Parpia**, **H.G. Craighead**, Cornell University
Recently we reported optical excitation and parametric amplification¹ of single-crystal silicon MEMS oscillators with resonant frequencies up to 1MHz. Utilizing the interferometric pattern of a laser beam in a Fabry-Pérot cavity formed by the oscillator, we demonstrated a mechanism which can be used both as a driving/amplification scheme and a detection scheme. Here we report observation of this phenomena in single-crystal silicon nanomechanical oscillators with frequencies up to 25MHz and with dimensions up to 2µm. High mechanical quality factors (Qs) of these structures were instrumental in enabling us to observe these phenomena. Qs of micron-scale and sub micron structures have been observed to have been relatively low (~ 10@super 3@) as measured in vacuum and at room temperature. We succeeded in improving the Qs of these devices (~ 10@super 4@) at room temperature and high vacuum. We will describe the bulk and surface treatments used to achieve high Q. In addition, these structures act as passive modulators of the laser light at their resonant frequencies. The sensitivity of the measurement technique and the inherent amplification of the motion via the optical drive presents us with a very efficient interferometer/modulator easily integrable on chip. Our initial modeling shows that the laser power needed to set these devices into oscillation is only up to few tens of microwatts. Our long-standing interest in nanomechanical structures fabricated in different materials, presents us with a natural extension for our current and future work - clearly being at an advantage of using this driving scheme even with electrically insulating device layers and with no theoretical frequency limit. @FootnoteText@
¹ M. Zalalutdinov, A. Olkhovets, A. Zehnder, B. Ilic, D. Czaplewski, H. G. Craighead, and J. M. Parpia, "Optically pumped paramagnetic amplification for micromechanical oscillators", Appl. Phys. Lett., Vol. 17 (16) 181 (2001).

8:40am **MM+BI+NS+EL+SS-FrM2 Micromechanical Cantilever Magnetometers with Integrated Quantum Dots**, **M. Wilde**, **M. Schwarz**, **D. Grundler**, **C. Heyn**, **D. Heitmann**, University of Hamburg, Germany
We have prepared highly sensitive micromechanical cantilever magnetometers with integrated semiconductor quantum dots. They allow us to study, for the first time, the very tiny magnetic moment of the quantum dots which contain only a few 100 electrons. We have used GaAs-AlAs-molecular beam epitaxy with its inherent atomic precision, both, for the optimization of the mechanical properties of the cantilever and for the monolithic integration of the investigated electronic nanostructures.¹ Using laser-interference lithography, tailored periodic arrays of quantum dots have been prepared on the beam. Experiments have been performed down to a temperature of 250 mK in a magnetic field up to 16 T. Field-induced magnetic oscillations have been observed on the quantum dots. The magnetization is significantly different from that of the two-dimensional reference sample and exhibits several new features. Our results suggest that, both, the quantum confinement and the effect of electron-electron interaction have an important effect on the magnetic moment of the quantum dots. Support by the Deutsche Forschungsgemeinschaft Gemeinschaft via Sonderforschungsbereich SFB 508 is gratefully acknowledged. @FootnoteText@
¹ M. Schwarz, D. Grundler, I. Meinel, Ch. Heyn, and D. Heitmann, Appl. Phys. Lett. 76, 3564 (2000).

9:00am **MM+BI+NS+EL+SS-FrM3 Nano-Electromechanical Systems: Physics and Applications**, **R.H. Blick**, Ludwig Maximilians University, Munich, Germany
INVITED
Mechanical devices in combination with modern semiconductor electronics offer great advantages as for example their robustness against electrical shocks and ionization due to radiation. The main disadvantage of mechanical devices so far is the low speed of operation. This has been overcome with the realization of nanomechanical systems (NEMS), which allow operation at frequencies up to 500 MHz. I will discuss recent work on such nanomechanical resonators focussing on nonlinear dynamics, mechanical mixing, parametric resonance, and possible uses in quantum

squeezing experiments. Furthermore, I will present record mechanical quality factors of $Q > 10000$. Finally, I will outline an approach to using NEMS for applications in biology (Bio-NEMS).

9:40am **MM+BI+NS+EL+SS-FrM5 Zeptonewton Force Detection at Millikelvin Temperatures**, **H.J. Mamin**, **D. Rugar**, IBM Almaden Research Center

Scanning force microscopes routinely operate with forces in the piconewton range, but new applications such as cantilever-based magnetometry and magnetic resonance force microscopy demand force resolutions that can be a million times smaller. The minimum detectable force is ultimately limited by the dissipation in the cantilever and its temperature. We have pushed this limit by cooling a single-crystal silicon cantilever in vacuum to a temperature below 100 mK. To sense the sub-angstrom thermal-mechanical motion with minimal heating of the cantilever, an improved optical fiber interferometer was developed that could be operated at optical powers as low as 2 nW. The cantilever mean square amplitude of vibration showed the expected linear dependence on temperature down to 400 mK, at which point other noise sources became significant. At the lowest temperature, the cantilever achieved a noise temperature of 220 mK, with a corresponding force noise of 820 zeptonewtons in a 1 Hz bandwidth.

10:00am **MM+BI+NS+EL+SS-FrM6 Chemical Detection Based on Nanostructured MEMS Sensors**, **P.G. Datkos**, Oak Ridge National Laboratory; **M.S. Sepaniak**, **N. Lavrik**, **C.A. Tipple**, University of Tennessee
The recent advent of MEMS devices has opened-up new possibilities for chemical detection. Microcantilevers respond to chemical stimuli by undergoing changes in their bending when molecules adsorb on their surface. Increased effective surface area is important in such systems because it results in increased total energy of interfacial interactions. In fact, in nanostructured surfaces (quasi 3-D interfaces) the effective surface stresses can significantly exceed true surface stresses. We used electron beam lithography to fabricate ordered nanofeatures on the surfaces of a microcantilever. We then functionalized the nanostructured surface with a beta-cyclodextrine coating (to impart chemical selectivity) using self-assembled monolayer techniques. We found an increase of two orders of magnitude when nanostructured coatings have been used. We present and discuss our findings on the interactions of functionalized microcantilevers with tetrachloroethylene molecules.

10:20am **MM+BI+NS+EL+SS-FrM7 Biomedical Microsystems for Minimally Invasive Medical Procedures**, **S. Roy**, The Cleveland Clinic Foundation
INVITED

Traditional surgery for many medical problems, including gallstones, endometriosis, and various cancers, usually requires long, deep incisions and a lengthy recovery period. Minimally invasive surgery, also known as "keyhole" or "band-aid" surgery, has been used for several years as an alternative to traditional "open" surgery. Minimally invasive procedures for both diagnostics and therapeutics have generated much attention from clinicians, patients, and healthcare administrators for their ability to reduce patient pain, scarring, and hospital stays. Current tools for minimally invasive procedures typically tend to operate as mechanical appendages of the clinician, but with absence of touch-and-feel sensations and only limited vision. The ability of MEMS technology to develop miniature, low-cost, and sophisticated transducers is particularly attractive for the development of smart surgical tools that enhance clinical efficacy. The talk will present an overview of current and upcoming applications of MEMS technology in cardiology, neurology, and orthopedics that are under development at The Cleveland Clinic Foundation and other institutions. Device examples will include pressure sensors, accelerometers, strain gauges, flow meters, valves, pumps, imaging transducers, drug delivery systems, and cutting tools.

11:00am **MM+BI+NS+EL+SS-FrM9 Fabrication Process and Resulting Structures for a Micron-Scale Force-Detected Nuclear Magnetic Resonance (NMR) Spectrometer**, **L.A. Madsen**, **G.M. Leskowitz**, **D.P. Weitekamp**, California Institute of Technology; **W. Tang**, **T. George**, **K. Son**, NASA Jet Propulsion Laboratory
NMR is the most widely used method of analysis of chemical structure and dynamics at the millimeter length scale. In order to overcome the inherent poor sensitivity of traditional inductively detected NMR for small samples, we are developing the novel BOOMERANG¹ method of force-detected NMR in a homogeneous magnetic field. Our experimental NMR results on liquid and solid 3 mm samples with a prototype spectrometer motivate the scaling of our detectors to observe samples < 100 microns in

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diameter. Achieving micron-scale detectors will bring about inexpensive NMR spectrometers with superior sensitivity for in-situ analysis, sub-monolayer surface NMR, and massively parallel studies on sample libraries. Ultimately, scaling of these detectors to the nano-scale may allow single-molecule NMR spectroscopy and imaging. We present a microfabrication process for BOOMERANG NMR detectors. This double-sided process utilizes deep RIE to define a Si beam fixed at both ends with a stress buttress at its center. High-aspect ratio NiFe or CoNiFe magnet structures are electrodeposited onto the backside of this beam. A combination of photoresist and oxide sacrificial layers allows ~1 micron spacing between a field compensation magnet and the moving detector magnet, and between the compensation magnet and the Si beam. Initial results of the 6-mask process are promising. We present patterned, electrodeposited magnets on the micro-oscillator substrate, as well as our efforts to characterize the micro-detectors and improve device yield. @FootnoteText@ @footnote 1@ Sol. St. Nucl. Magn. Reson. 11, 73 (1998).

Bold page numbers indicate presenter

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