

SYMPOSIUM U
MEMS and BioMEMS

April 1 – 3, 2002

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TUTORIAL

ST U: MICROSYSTEMS TECHNOLOGY - BIOLOGICAL AND MEDICAL APPLICATIONS

Monday, April 1, 2002
1:00 p.m. - 5:00 p.m.
Salon 14/15 (Marriott)

The tutorial will cover micromachining technologies—surface, bulk micromachining, LIGA, polymer-based techniques—with process and device examples. Following the technology overview, potential applications of this fast maturing technology base in biomedical sciences (such as drug delivery, separation, detection, sample manipulation, neural prosthesis, advanced research tools, etc.) will be highlighted. A survey of current research projects/prototype devices, with an eye towards their potential function and place in realizing the impending “micro/nano/biotechnology” revolution, will be presented. This tutorial is intended to provide a basic working knowledge of the micromachining technologies and their foreseeable applications in the biomedical sciences.

Instructors:

Luke P. Lee, University of California-Berkeley
Ronald P. Manginell, Sandia National Laboratories
Murat Okandan, Sandia National Laboratories

SESSION U1: MICROFLUIDICS

Chairs: Luke P. Lee and Jeffrey T. Borenstein
Tuesday Morning, April 2, 2002
Olympic (Argent)

8:00 AM *U1.1

SOFT PLUMBING FOR INTEGRATED MICROFLUIDIC DEVICES. **Stephen Quake**, Department of Applied Physics, California Institute of Technology, Pasadena, CA.

We have been using soft lithography to make microfluidic chips for ultrasensitive analysis of single DNA molecules and cells. There are numerous advantages to fabricating chips out of polymeric materials, and as a result we have been able to rapidly and inexpensively fabricate active devices with moving parts, such as pinch valves and peristaltic pumps. We have developed a series of microfluidic devices for cellular and molecular analysis, ranging from protein crystallization to a microfabricated fluorescence activated cell sorter. The novel valve and pump components for on-chip fluidic manipulation that we developed in the course of this research will be useful for fabricating more complex chip designs for a variety of biotechnological applications.

8:30 AM *U1.2

PLASTIC BIOMEMS MICROFLUIDIC DEVICE ARRAYS FOR BIOASSAY APPLICATIONS. **Antonio J. Ricco**, ACLARA BioSciences, Mountain View, CA.

Plastic microfluidic array platforms and novel multiplexed assay chemistries are being developed for a variety of applications, including protein and gene expression analysis, protein-protein interaction assays, genotyping, DNA sequencing and fragment analysis, sample preparation, and high-throughput pharmaceutical screening. The low production costs of plastic substrates make it possible to produce economical single-use devices, eliminating both the need for cleaning/reuse and the concern of sample-to-sample carryover contamination. Further, the low cost and relative ease of including many hundreds of microchannels and reservoirs on a single microtiter-plate-size substrate enable the manufacture of highly parallel fluidic array systems to speed analysis and increase throughput. Plastic microfluidic array systems are manufactured in a variety of application-specific designs. The process begins with the masking, lithography, and etching of a master template from a Si wafer or glass plate. A metal tool is produced from this template using electroforming, and the tool is then used to mold or emboss plastic LabCard® devices from a variety of polymer resins. The microchannels in the resulting devices are typically tens of micrometers in depth and width, following complex paths that can be centimeters in length. These microchannels connect to reservoirs that hold 0.2 - 10 μ of reagents. Hundreds of reservoirs and meters of channels can be densely packed into multiplexed designs that form 96- or even 384-assay patterns on a single 7.5 x 12.5 cm disposable card. Electrodes in the reservoirs provide electrical contact to manipulate fluids and analytes via electrokinetic forces. Reactions can be incubated on the devices; products are electrophoretically separated

and detected via confocal laser-induced fluorescence with limits of detection below 30 pM.

9:00 AM U1.3

FLOW OF BINARY FLUIDS OVER PATTERNED SUBSTRATES WITHIN MICROCHANNELS. **Olga Kuksenok**, Chemical Engineering Department, University of Pittsburgh, Pittsburgh, PA; **David Jasnow**, Physics Department, University of Pittsburgh, Pittsburgh, PA; **Julia Yeomans**, Oxford University, Oxford, UNITED KINGDOM; **Anna Balazs**, Chemical Engineering Department, University of Pittsburgh, Pittsburgh, PA.

Microfluidics, involving the transport of minute quantities of liquids in networked channels, lies at the heart of the “lab-on-a-chip” concept. It is important to develop predictive models that reveal the thermodynamic behavior and flow patterns of complex fluids in such channels. We investigate the flow and phase behavior of a binary fluid moving over a patterned substrate within a three-dimensional microchannel. The binary fluid consists of two immiscible components, and this liquid is subjected to a Poiseuille flow. We model patterned substrates by introducing domains with preferential wetting interactions for the components. Depending on the design of the patterns, fluids can undergo extensive mixing within the microchannels. A rich phase behavior can be obtained in the case where patterned regions are placed on the top and on the bottom of the channel. We investigate how the phase behavior and degree of mixing depends on the the velocity of the imposed flow field and the characteristics of the fluid. The results provide guidelines for the controlling the fluid flow and for the creating localized “mixing stations” within microfluidic devices.

9:15 AM U1.4

BIODEGRADABLE POLYMER MICROFLUIDICS FOR TISSUE ENGINEERING MICROVASCULATURE. **Kevin R. King**, Chiao-chun C. Wang, Jeffrey T. Borenstein, Draper Laboratory, Cambridge, MA, Joseph P. Vacanti, Massachusetts General Hospital, Boston, MA.

Tissue engineering is emerging as a therapeutic alternative to organ transplantation for the more than 75,000 patients currently on the organ donor waiting list. In one approach, cells are seeded on biodegradable porous scaffolds, cultured in vitro, then implanted in vivo, enabling tissue integration during polymer degradation. While this approach has demonstrated early successes, cell-polymer constructs are limited to millimeters without an integrated blood supply. Porous scaffolds rely solely on diffusion for mass transport while normal tissues leverage convection from blood vessels to enable oxygenation of large tissues. We have proposed fabricating biodegradable microfluidic channels to provide functional equivalents of the microvasculature and enable scale-up of tissue engineering scaffolds. Here we present for the first time, the fabrication of a fully biodegradable microfluidic device with features of micron-scale precision. This implantable MEMS device is a transition from poorly defined porous scaffolds to reproducible precision biodegradable scaffolds with built in convective conduits. First, conventional photolithography is used to create a master mold by polymerizing a photosensitive epoxy or reactive ion etching silicon. Next, Polydimethylsiloxane (PDMS) elastomer is replica molded to form a flexible inverse mold. The commonly used biodegradable polymer, Poly-lactic-co-glycolic acid (PLGA) 85:15, is then compression molded onto the PDMS to form micropatterned films of degradable polymer. Finally biodegradable films are fusion bonded to form microfluidic channels at the capillary size-scale. Film thicknesses from 100 μ m-1mm with 4 μ m micropattern resolution are demonstrated. SEM of bonded biodegradable films reveals no observable bond interface and no significant pattern deformation. Bonded microfluidic channels support 30 psi during flow studies, and we have used the processes to develop complex microfluidic networks for cell culture and implantation as well as simple channels to investigate the fluidic dynamics in degradable microchannels. The processes described here are high resolution and fully biodegradable. In addition, they are fast, inexpensive, reproducible, and scalable, making them ideal for both rapid prototyping and manufacturing of tissue engineering scaffolds.

9:30 AM U1.5

SOFT LITHOGRAPHY USING A THICK SU-8 NEGATIVE PHOTORESIST IN FABRICATION OF MICROCHANNELS FOR A CELL-BASED BIOSENSOR. **Jeb H. Flemming**, Sara S. Sokolowski, Ronald P. Manginell, Susan Brozik, Sandia National Labs; Albuquerque, NM; **Gabriel P. Lopez**, Maggie Werner-Washburne, University of New Mexico, Albuquerque, NM.

Living cells have a remarkable ability to respond to external factors, unrivaled by mechanical devices, by incorporating analyte detection and signal transduction in a predetermined biochemical pathway. Utilizing this detection ability, a BioMEMS based cell-based biosensor using *Brewers yeast* (*Saccharomyces cerevisiae*) packed in a microfluidic channel has been engineered. The microchannel is

developed using a mold fabricated with SU-8. SU-8 is a thick, near UV, negative epoxy based photoresist. Molds of 20-45 um thick have been developed for the cell-based biosensor. This mold creates a positive microchannel using poly(dimethylsiloxane) (PDMS). This is a fast, cheap, and simple process to integrate soft lithography into a BioMEMS device. Yeast cells are trapped inside the microchannel using packing posts allowing protection from environmental stresses and access to target analytes. These cells have been genetically engineered (by attaching the Green Fluorescent Protein (GFP) gene to the GAL7 promoter) to produce the GFP fluorophore in the presence of galactose. The GFP excites at 395 nm and fluoresces at 509 nm. We report on cell viability in the microchannels, effects of analyte concentration on cellular response, and the overall time to detection. Although this setup describes only one fluorophore in one cell-line, a more elaborate system including several fluorophores in many yeast cell-lines, each detecting a different target analyte, will be designed in the future utilizing a matrix of microfluidic channels.

9:45 AM U1.6

OBJECT MANIPULATION IN SOFT MICROFLUIDIC DEVICES. Erhan Ata, Aaron Birkbeck, Sadik Esener, University of California, San Diego, CA; Cengiz S. Ozkan, Mhrihah Ozkan, University of California, Riverside, CA; Mark Wang, Genoptix Inc., San Diego, CA.

In this paper, we report on microfluidic devices that utilize object-photon and object-electric field interactions. Microfluidic devices have been fabricated by polydimethylsiloxane (PDMS) elastomer molding of patterns lithographically defined on a thick negative photoresist. Polystyrene microspheres dispersed in water were transferred to the fluidic channels using a syringe pump. Using VCSEL radiation induced optical traps and/or photonic pressure with only a few mW of optical power, microspheres have been manipulated selectively and switched to desired outlets of microfluidic channel devices. In addition, parallel optical manipulation of the microspheres were carried out using VCSEL optical tweezer arrays. Finally, we have shown that polymer based microlenses can be aligned and manipulated electrophoretically in polymer wells. Such microfluidic devices can be utilized in a variety of biomedical and photonics applications.

10:30 AM *U1.7

MICRO- AND NANO-FABRICATION OF POLYMER BASED MICROFLUIDIC PLATFORMS FOR BIOMEMS APPLICATIONS. Siyi Lai, L. James Lee, Liyong Yu, Kurt W. Koelling, Dept of Chemical Engineering, The Ohio State University, Columbus, OH; Marc J. Madou, Nanogen, San Diego, CA.

The miniaturization of chemical and mechanical instrumentation for micro electromechanical systems (MEMS) has gained a great deal of attention in industry over the last years. Examples include ink jet nozzles, air bag triggers, on-chip flow-through-PCR, microreaction technology, and electrophoretic separation devices. In the past, these devices have been fabricated almost exclusively in silicon, glass or quartz because of the similar technology available in the micro-electronics industry. For many applications, particularly in the biochemistry and biomedical field, polymeric materials are a better choice because of their lower cost and bio-compatibility. Polymer micro-/nano-fabrication techniques, however, are still not well developed. We present in this work various ways to fabricate microfluidic devices (thin wall injection molding, hot embossing, and reactive molding) based on several optical clear thermoplastic and thermoset polymers (e.g. optical quality polycarbonate, PMMA, PDMS, and polyurethane). Micro-bonding and surface modification of such platforms will also be discussed.

11:00 AM *U1.8

DISPOSABLE SMART PLASTIC BIOCHIPS FOR CLINICAL DIAGNOSTICS. Chong H. Ahn, Jin-Woo Choi, Sanghyo Kim, Young Soo Sohn, Suresh Murugesan*, Gregory Beaucage*, and Joseph H. Nevin, Microsystems and BioMEMS Lab, University of Cincinnati, Department of Electrical and Computer Engineering and Computer Science, Department of Material Science*, Cincinnati, OH.

Diseases, including toxicity, can be diagnosed by performing various biochemical analyses and by observation of symptoms. The early, rapid, and sensitive detection of the disease state is a vital goal for the clinical diagnoses. The biochemical changes in the patient's blood can signal organ damage or dysfunction prior to observable microscopic cellular damages or other symptoms. So, there has been a large demand for the development of an easy-to-handle and inexpensive clinical diagnostic biochip using fully integrated plastic microfluidic chips, which has the sampling/identifying capability of fast and reliable measurements of metabolic parameters from a human body with minimum invasion. In this paper, the development of disposable smart plastic fluidic biochips for clinical diagnostics will be presented. The plastic fluidic chip includes fully integrated microchannels, passive valves, passive

multiplexers, mixers, dispensers, pressurized air bladders, and air/buffer reservoirs. Among several substrates available for the bio-fluidic chips, plastics have recently become one of the most popular and promising due to their low cost, ease-of-fabrication and favorable biochemical reliability and compatibility. Plastic substrates, such as polyimide, PMMA, PDMS, polyethylene or polycarbonate, offer a wide range of physical and chemical material parameters for the applications of bio-fluidic chips at generally low cost using replication approaches. The disposable smart plastic biochip is composed of integrated modules of plastic fluidic chips for fluid regulation, chemical and biological sensors, and electronic controllers. As a demonstration vehicle, the biochip has the specific goal to detect and identify three metabolic parameters such as PO₂ (partial pressure of oxygen), Lactate, and Glucose from blood. In this presentation, an overview of the speaker's recent research achievements for disposable smart plastic biochips will be presented, discussing the relevant issues to the material, design, fabrication, and characterization of the plastic biochips.

11:30 AM *U1.9

MICROMACHINING TECHNOLOGY DEVELOPMENT FOR MICROFLUIDICS AND BIOMEMS. Murat Okandan, Paul Galambos, Sita Mani, Harold Stewart, Sandia National Laboratories, Albuquerque, NM.

In the last decade, examples of devices manufactured with SUMMITT (surface micromachining) technology have demonstrated the capabilities of polysilicon surface micromachining. Currently we are working on enhancements to this technology to enable microfluidics and BioMEMS. Our goal is to ultimately provide functionality and integration that is not feasible with other microfabrication technologies. The enhancements build on the key features of surface micromachining: manufacturability and compatibility with CMOS processing, and allow us to leverage the investment already made in the microelectronics processing technology. Some of the initial demonstration devices include valves, pumps, integrated electrodes and a prototype microtransfection device. The microtransfection device is intended to demonstrate the integration of microfluidic, electrical, mechanical and optical components. This device is designed to mechanically disrupt the membrane of the cells flowing through the microfluidic channel to allow delivery of large molecules (DNA, RNA, proteins, etc.) that would normally be blocked. The ultimate 'system on a chip' for biomedical and sensing applications will require incorporation of microfluidic, mechanical, optical, electrical and chemical subsystem components into an integrated system. The novel devices that are being presented here are fabricated utilizing a process that incorporates silicon nitride layers, allowing optical access into the structures as well as the integration of electrical subsystems for electrochemistry, electrokinetics and mechanical actuation. In a system view, integration of MEMS components similar to the ones shown here will allow multiple complex functions to be performed on a chip. With the availability of these components in almost a mix-and-match fashion, this technology development is expected to enable a high level of system integration at a smaller (chip) scale and enhance the manufacturability and applicability of BioMEMS.

SESSION U2: MATERIALS AND PROCESSES
Chairs: Ronald P. Manginell and Peter J. Hesketh
Tuesday Afternoon, April 2, 2002
Olympic (Argent)

1:30 PM U2.1

SURFACE ENGINEERING OF POLYSILICON MEMS FOR FATIGUE RESISTANCE. C.L. Muhlstein, Materials Sciences Division, Lawrence Berkeley National Laboratory, and Dept of Materials Science and Engineering, University of California, Berkeley, CA; W.R. Ashurst, Department of Chemistry, University of California, Berkeley, CA; E.A. Stach, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA; R. Maboudian, Department of Chemistry, University of California, Berkeley, CA; and R.O. Ritchie, Materials Sciences Division, Lawrence Berkeley National Laboratory, and Dept of Materials Science and Engineering, University of California, Berkeley, CA.

Recent research has established that the susceptibility of silicon structural films used in MEMS to premature failure under cyclic fatigue loading originates from a degradation process that is confined to the surface oxide. In ambient air environments, a sequential, stress-assisted oxidation and stress-corrosion cracking process can occur within the native oxide on polycrystalline silicon (referred to as reaction-layer fatigue); for the structural films of micron-scale dimensions, such incipient cracking in the oxide can lead to catastrophic failure of the entire silicon component. The basis for this understanding is direct observation of the fatigue damage using high-voltage transmission electron microscopy and the measurement

of the evolution of crack size and growth rates during testing. Since the degradation process is intimately linked to the thin reaction layer on the silicon, modification of this surface and the access of the environment to it can dramatically alter the fatigue resistance of the material. The purpose of this paper is to evaluate the efficacy of modifying the fatigue behavior of polycrystalline silicon with alkene-based self assembled monolayers. Specifically, 2- μm thick polysilicon fatigue structures were coated with a self-assembled monolayer based on 1-octadecene and cyclically tested to failure in laboratory air. By applying the coating the formation of the native oxide was prevented. Compared to the fatigue behavior of untreated polysilicon, the lives of the coated samples ranged from 10^5 to $>10^{10}$ cycles at stress amplitudes greater than $\sim 90\%$ of the ultimate strength of the film. The dramatic improvement in fatigue resistance was attributed to the monolayer inhibiting the formation of the native oxide and stress corrosion of the surface. It is concluded that the surprising susceptibility of thin structural silicon films to premature fatigue failure can be inhibited through the use of such self assembled monolayer coatings.

1:45 PM U2.2

FABRICATION OF MEMS BY SUPERCONFORMAL ELECTRODEPOSITION OF METALS: MODEL AND EXPERIMENT. Daniel Josell, Thomas Moffat and Daniel Wheeler, NIST, Gaithersburg, MD.

We explain the mechanism responsible for superconformal electrodeposition of metals in small, high-aspect ratio features and its application for the design and fabrication of metallic MEMS and NEMS structures. Simulations from a model based on this mechanism are compared to filling experiments in features as small as 100 nm wide and approximately 500 nm deep. We first identify additive-containing electrolytes that yield superconformal electrodeposition of metals [1]. All kinetic parameters describing the rate of additive accumulation on metal surfaces as well as the impact of the accumulation on metal deposition rate are obtained from deposition studies with flat metal specimens in electrolytes containing a range of additive concentrations. Accumulation of additive that accelerates local deposition rate is described by diffusion across a boundary layer and deposition voltage dependent interface kinetics. Kinetic parameters are found to be linear in the fractional coverage of the accelerating additive, which displaces inhibiting additive on the metal surface and saturates at one monolayer coverage [2,3]. For non-flat specimens (e.g., features in patterned substrates in which MEMS are being electrodeposited), area change during deposition causes additional change of local accelerator coverage through mass conservation. Accumulation of accelerator, and thus the deposition rate, at the bottoms of superfilling features is found to be dominated by this area change effect. The model, with no free parameters, is used to predict filling behavior of lithographically patterned features. Predictions are in excellent agreement with experimental results [2-4]; initial period of conformal growth, subsequent superfill, and development of overfill bump are all observed and explained. Predictions of fill versus void formation in particular features for different deposition voltages, electrolyte compositions and feature dimensions agree with experimental results. While the authors view this work as a means of fabricating small, high-aspect ratio metallic structures that are fully dense, it can also be viewed as a means of intentionally designing features with continuous internal cavities (i.e., pipes).

1. T.P. Moffat, J.E. Bonevich, W.H. Huber, A. Stanishevsky, D.R. Kelly, G.R. Stafford and D. Josell, *J. Electrochem. Soc.* 147, 4524 (2000).
2. T.P. Moffat, D. Wheeler, W.H. Huber, and D. Josell, *Electrochem. Solid-State Lett.* 4, C26 (2001).
3. D. Josell, T.P. Moffat, D. Wheeler and W.H. Huber, *Phys. Rev. Lett.* 71, 016102 (2001).
4. D. Josell, D. Wheeler, W.H. Huber, J.E. Bonevich and T.P. Moffat, A Simple Equation for Predicting Superconformal Electrodeposition in Submicrometer Trenches, *J. Electrochem. Soc.* (in press).

2:00 PM U2.3

LIFT-OFF METHODS FOR MEMS DEVICES. Shih-Chia Chang and Jeffrey Kempisty, Delphi Research Laboratories, Warren, MI.

In conventional integrated circuits, the commonly used thin film materials such as aluminum, silicon, silicon nitride or silicon dioxide are readily delineated by wet chemical or plasma etching methods. However, for MEMS devices, because of processing consideration and functional requirement, thin films such as platinum, tantalum, nickel or iron which are difficult to be etched by conventional methods, may have to be used either for the formation of transducing elements or electrical interconnects. In those cases, lift-off will be the method of choice for patterning the device features. The key requirement for the lift-off method is that the mask has an etched wall with a negative slope. In this work, several different lift-off methods were explored. In the first method, a tri-level resist scheme was used to fabricate the

lift-off mask. Good line width resolution was achieved; however, process complexity and longer processing time were the drawbacks. In our second method, an aluminum/photoresist double layer was used for the lift-off mask. An overhang structure was obtained by undercutting the underlying aluminum layer. While this process is simple, the thin film device materials are limited to that are not attacked by the aluminum etch solution. In the third method, a composite layer of two different photoresists (AZ1811 and LOR 10A) was used for the lift-off mask. The fast dissolution of the underlying LOR 10A photoresist in the photoresist developer (LDD-26W) resulted in an overhang mask structure. In the lift-off methods mentioned above, regular positive photoresist was used to form the lift-off mask, they are suitable for thin film materials with thickness of $\sim 2\mu\text{m}$ and processing temperature $< 100\text{C}$. For thicker film materials and higher processing temperature, two negative resists, Futurrex ($\sim 40\mu\text{m}$) and SU-8 ($\sim 100\mu\text{m}$) were used as the lift-off mask. The mask formation process is relatively simple and very high aspect ratio (SU-8) was readily obtained using regular UV lithographic method. The removal of the resist was somewhat complicated. All these aspects will be discussed in the presentation.

2:15 PM U2.4

OPTIMIZATION OF PZT BASED MEMS. Firas Akashch, Todd Myers, Susmita Bose and Amit Bandyopadhyay, Mechanical and Materials Engineering, Washington State University, Pullman, WA.

Due to their excellent piezoelectric properties, PZT ceramics are attractive materials for many MEMS applications including sensors, actuators, and transducers. By depositing a PZT film on a micro-machined platinumized silicon substrate, membranes can be actuated in the flexural mode, which has been the most popular mode of operation for various MEMS devices. In this work, 2D arrays of PZT actuated membranes have been designed, modeled, and fabricated employing the K31 coupling effect, which can be used for numerous applications. Process compatibility issues were studied for the fabrication of these devices. PZT-driven membranes were characterized for their ferroelectric response. To develop a better understanding of the device performance as a function of materials and geometries, a single representative element of such structures was also modeled by using time-domain finite element analysis using commercial FE package, PZFlex, which allows simulating the temporal evolution of the system. The resulting data was used to evaluate the device based on some measures of performance including the device effective coupling factor to the acoustic radiation, its acoustic impedance, and its resonance frequency. We report results on the influence of various design parameters such as the individual layers thickness, layering materials and membrane dimensions, have on the overall performance of the device. Results show that the device-coupling factor is significantly affected by the PZT and silicon layers thickness and the top electrode configuration. While it exhibits an optimum with respect to the PZT and silicon layer thickness, any increase in the gold top electrode thickness adversely impairs the coupling factor. This can be attributed to the accompanying increase in input energy going into kinetic energy rather than stored elastic energy available as a mutual energy. Results also indicate that considerable flexibility in controlling the levels of the various performance critical parameters without sacrificing the rest is possible.

2:30 PM U2.5

PROCESSING INFLUENCE ON THE RELIABILITY OF PLATINUM THIN FILMS FOR MEMS APPLICATIONS. Danick Briand, Stephan Heimgartner, Mireille Leboeuf, Massoud Dadras, Nico F. de Rooij, Institute of Microtechnology, University of Neuchatel, Neuchatel, SWITZERLAND.

Platinum is a material used in microsystem technology due, among others, to its capability to withstand high temperature post-processing and operation. We have investigated the influence of high temperature post-processes on the microstructural, electrical, and mechanical properties of evaporated platinum thin films with a tantalum adhesion layer. The aim of this work was to define the post-processing parameters influencing the stability and reliability of these films for high temperature operation. Post-processes such as the deposition of silicon nitride passivation layers by PECVD or LPCVD and the annealing at high temperature (up to 800°C) in an inert or reactive atmosphere were performed separately or successively on platinum films having different thickness. The film properties were characterized by using SEM, XRD, RBS, TEM analysis, and electrical, stress and adherence measurements. Depending on the post-processing conditions, platinum films having different electrical and mechanical properties were obtained. The post-processing temperature and the proportion of Pt/Ta in the films influenced their electrical characteristics (resistivity, TCR). RBS and TEM showed that the tantalum adhesion layer is mainly responsible for these changes due to its diffusion in platinum at high temperature. It was observed by SEM and XRD that grains growth and reorientation occurred in the platinum films during the heat treatments, which were

stabilised in a short time. A passivation made of LPCVD nitride film can therefore electrically stabilised the film if remained during operation at high temperature. However, if removed, post-processing at higher temperatures than 500°C of platinum films in an atmosphere containing oxygen induces an oxidation of tantalum which yield to more less severe hillocks formation and adhesion problems. The Pt/Ta films properties depend on the annealing conditions and on the history of the film, if the annealing was performed just after the deposition or after successive thermal processes.

2:45 PM U2.6

FABRICATION OF THICK MOLDED TUNGSTEN MEMS DEVICES. J.G. Flemming, Sandia National Laboratories, Albuquerque, NM.

This paper describes the fabrication of thick, molded, tungsten and silicon nitride parts. The process has three key elements: the use of deep trench etch processes to create molds and release layers in silicon; highly conformal tungsten, silicon and silicon nitride chemical deposition processes; and the use of 111 orientated silicon substrates as a sacrificial layer. Relatively thick structures are of interest for a variety of reasons, increased Z dimension stiffness, increased capacitance and therefore force, and increased mass for inertial applications. Typical surface micromachining works with silicon layers roughly 2 microns thick. By stacking together multiple silicon layers along with their intermediate sacrificial layers, it is possible to create structures that are roughly ten microns thick. However, this is a relatively complex process requiring multiple mask levels and depositions. It is therefore desirable to investigate other ways to fabricate thick structures. Recent advances in deep reactive ion etch processes enable the creation of deep, high aspect ratio trenches and holes. These molds can be filled with either silicon nitride for insulation and anchors to the substrate, or tungsten for conductive lines. The parts are then released by etching into the substrate and removing excess silicon with a KOH etch, which does not attack either silicon nitride or tungsten. By using a silicon 111 substrate it is possible to control the amount of the gap between the active tungsten lines and the silicon substrate. Since this is determined by the depth of an etch process this separation can be quite large, up to 15 microns in one application. The various steps in the process will be outlined using the example of a lateral, contacting, radio frequency switch. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy.

3:30 PM U2.7

DRY ETCHING TECHNIQUES OF AMORPHOUS SILICON FOR SUSPENDED METAL MEMBRANE RF MEMS CAPACITORS. R. Fritschi, C. Hibert, Ph. Flücker, Swiss Federal Inst of Tech, EPFL Ctr of Microtechnology (CMI), Lausanne, SWITZERLAND; A.M. Ionescu, Swiss Federal Inst of Tech, Electronics Lab (LEG), Lausanne, SWITZERLAND.

Nowadays, passive components, such as tunable capacitors, integrated on silicon or SOI substrates are subject of increased interest for programmable RF ICs. Conventional on-chip tunable capacitors are solid-state devices that suffer from excessive series loss, caused by large series resistance, and a low quality factor. MEMS technologies seem very promising to alleviate these problems. The realization of MEMS capacitors needs the releasing of suspended membranes, which act as mobile electrodes. The usual technique for releasing structures is a standard polysilicon surface micromachining process, which involves a silicon dioxide sacrificial layer wet etch and a supercritical carbon dioxide drying process. Other methods use organic sacrificial layers released in an oxygen plasma. This work reports on a full-dry process sequence for patterning and releasing aluminum (Al/1%Si) membranes. Base electrodes and membranes are patterned by dry etching in a Cl₂/BCl₃ gas mixture. The sacrificial layer is an amorphous silicon layer deposited either by sputtering or by PECVD. It is patterned by dry etching in a fluorine chemistry to give the three-dimensional membrane shape. A sputtered or PECVD silicon dioxide barrier layer is deposited to prevent Al diffusion into silicon. The membrane releasing is done in a SF₆ plasma with a high selectivity to SiO₂ and aluminum. The proposed process is fully compatible with CMOS post-processing. Various tunable capacitor architectures are designed, realized and measured in order to capture the main advantages and drawbacks of the new proposed full-dry process. The Design-Of-Experiment includes variations of parameters such as: (i) membrane shape, (ii) membrane size, (iii) number of etch holes, (iv) suspension beams novel architecture. The proposed design of tunable capacitors aims at CMOS-compatible devices with voltage operation below 5 V, high quality factor over wide-band (1-10 GHz) and capacitance tuning range in excess of 70%. Fabrication of quasi-identical MEMS capacitor structures on both silicon and Silicon-On-Sapphire (SOS) substrates also allows to quantitatively evaluate the RF performance gain in case of highly resistive substrates.

3:45 PM U2.8

DEVELOPMENT OF 1TO 5 μ THICK MONOLITHIC SAPPHIRE MEMBRANES AS SUBSTRATES FOR FAR IR DETECTION APPLICATIONS. Brook Lakew, Goddard Space Flight Center, Greenbelt, MD; Shahid Aslam, Raytheon ITSS Corp, Lanham, MD; John Brasunas, Goddard Space Flight Center, Greenbelt, MD; Rainer Fettig, Raytheon ITSS Corp, Lanham, MD.

Ultrathin (1 to 5 μ thick), monolithic, single crystal, R-plane sapphire membranes have been created. A new technique has been developed to pattern the etch mask. Single element as well as 1 dimensional monolithic sapphire arrays have been obtained. A single monolithic membrane has been coated with a GdBCO high temperature superconducting thin film. The paper will discuss the performance (Responsivity, NEP and D*) of the transition edge, superconducting, Far IR thermal detector thus developed. Etching, etch mask patterning of R-plane sapphire will also be discussed. Other possible applications of monolithic sapphire membranes, including 2-D arrays, will also be addressed.

4:00 PM U2.9

PATTERNING TECHNOLOGY FOR MEMS WITH ANISOTROPICALLY ETCHED GROOVES. Fred P. Gnadinger, Bradley J. Luttrell, COVA Technologies Inc., Colorado Springs CO; Meredith Metzler, Metzler Engineering, Freeville, NY; Jill Steeper, Shipley Ronal, Marlborough, MA.

MEMS processing quite often requires features to be patterned on surfaces other than those parallel to the device surface, for example on sloped surfaces such as anisotropically etched through- holes and grooves in silicon. Standard spin- on photoresist processing is not feasible in these instances, since the photoresist would simply fill in the grooves or through- holes. A new photoresist and patterning technology for MEMS processing is presented that relies on an electroplated photoresist that maintains its thickness uniformity even on a sloped surface. Patterning results are presented that were achieved in processing integrated silicon chip antenna coils used in RFID. Special emphasis is given to the characterization of minimum achievable feature sizes and spacings as a function of the size and shape of the grooves or through- holes.

SESSION U3: POSTER SESSION MEMS AND BIOMEMS

Chairs: Ronald P. Manginell, Peter J. Hesketh,
Luke P. Lee and Jeffrey T. Borenstein
Tuesday Evening, April 2, 2002
8:00 PM
Metropolitan Ballroom (Argent)

U3.1

DEEP ANISOTROPIC ETCHING OF GaAs WITH CHLORINE-BASED CHEMISTRIES AND SU-8 MASK USING RIE AND HIGH DENSITY ICP ETCHING METHODS. Yuh-Min (Johnson) Chiang, Joy Lau, Mark Bachman, G.P. Li, University of California at Irvine, Department of Electrical and Computer Engineering, Irvine, CA; H.K. Kim, Yunju Ra, Bethel Material Research, Placentia, CA; Kurt Ketola, Raytheon, El Segundo, CA.

GaAs-based microelectromechanical systems (MEMS) are emerging technologies, which lead to integrate new sensing and actuating devices with microwave and millimeter-wave electronics. However, for the fabrication of MEMS devices in this application, the capability of selective deep etching of GaAs substrate is a critical issue to be solved. This paper presents a study of a highly anisotropic deep GaAs etching. Two different plasma etching methods were used for the GaAs etching with chlorine-based chemistries: reactive ion etching (RIE) with Cl₂/BCl₃/SiCl₄ mixtures and high-density inductive coupled plasma (HDICP) etching with Cl₂/BCl₃ mixtures. An UV-LIGA photoresist, SU-8, which provides a high aspect ratio at a high resistance to etchants, is selected as the etching mask for this work. Process parameters such as source power, bias power, chamber pressure, total flow rate, and composition of gas mixtures were varied for best results. The effect of the process parameters on the sidewall profile, uniformity, surface morphology, and etching rate will be presented. Issues, such as selectivity of SU-8 and GaAs substrate, reflow of SU-8, and loading effect were also investigated. An optimized etching process is developed to achieve a deep etch in excess of 300 μm of GaAs with a single layer of SU-8 photoresist.

U3.2

ELECTRICAL PROPERTIES AND NOISE OF POLY SiGe DEPOSITED AT TEMPERATURES COMPATIBLE WITH MEMS INTEGRATION ON TOP OF STANDARD CMOS. Sherif Sedky, IMEC, Leuven, BELGIUM.

The main objective of this work is to investigate the electrical conductivity, temperature coefficient of resistance and noise of poly $\text{Si}_x\text{Ge}_{1-x}$ deposited at a temperature suitable for post-processing MEMS on top of standard CMOS wafers with Al interconnects. For this study, boron in situ doped poly $\text{Si}_x\text{Ge}_{1-x}$ has been deposited at 400°C and 2 Torr. The Ge content has been varied from 69% to 100%. It is demonstrated that boron in situ doping can be activated at temperatures as low as 400°C with a minimum resistivity of 1 m Ω -cm. On the other hand, for a boron ion implantation dose of 10¹⁶ cm⁻², the resistivity of poly $\text{Si}_{31}\text{Ge}_{69}$ is 20 m Ω -cm (measured after annealing at 450°C for 30 minutes). Furthermore, the mechanical properties of this material is suitable for realizing flat suspended structures suitable for MEMS. The effect of decreasing the Ge content, below 50%, on activating boron or phosphorus in situ doping or ion implantation has been investigated by depositing poly $\text{Si}_{57}\text{Ge}_{43}$ at 40 Torr and 520°C (This temperature has been proven to be compatible with CMOS backend [1]). The boron concentration has been varied from 7.85x10¹⁵ B/cm³ to 8.76x10¹⁸ B/cm³ and the corresponding resistivity has been found to vary, respectively, from 100 Ω -cm to 0.2 Ω -cm. It is demonstrated that for the same doping concentration, the resistivity of phosphorus doped samples is one order of magnitude higher than that of boron doped samples. The measured resistivity of boron doped samples is almost unaffected by increasing the annealing temperature from 520°C to 650°C whereas that of phosphorus doped samples changed noticeably. The dependence of noise on resistivity and lateral dimensions has been characterized and it is shown that there is a significant low frequency (1/f) noise. For the same resistivity, the measured 1/f noise in boron doped poly SiGe samples is one order of magnitude lower than that measured in phosphorus doped samples. In conclusion, the electrical and mechanical properties of boron in situ doped poly SiGe is suitable for integrating MEMS on top of standard CMOS wafers with Al interconnects as it can be completely processed at temperatures as low as 400°C.

References:

[1] S. Sedky, A. Witvrouw, H. Bender and K. Baert, 'Experimental determination of the maximum post processing temperature of MEMS on top of standard CMOS wafers', IEEE Transactions on Electron Devices, Vol. 48 (2), p. 1-9, February, 2001.

U3.3

SPUTTERED TANTALUM AS A STRUCTURAL MATERIAL FOR SURFACE MICROMACHINING APPLICATIONS. Sherif Sedky, Paolo Fiorini, Ann Witvrouw and Kris Baert IMEC, Leuven, BELGIUM.

Aluminum (Al) is a widely used metal in surface micromachining applications, specifically for RF switches, which take advantage of its low resistivity. Unfortunately stress relaxation (creep) might seriously affect device reliability. The objective of this work is to improve MEMS reliability by using Tantalum (Ta) as a structural material, as it is known for its low creep rate. We report, for the first time, on the fabrication of surface micromachined bridges using Ta either as a single layer or in combination with Al. Stress in Ta has been tuned by adjusting the sputtering power, pressure and target-to-substrate distance. It has been found that for a sputtering power of 600 W, stress changes from compressive to tensile at a pressure around 1.2 Pa. This pressure corresponds to a phase transition of the film structure from cubic alpha to tetragonal beta. The corresponding resistivity is 170 μ Ω -cm, in agreement with values reported for the tetragonal beta phase. On the other hand, it has been found that the substrate influences the stress values significantly. Sputtering Ta at 1.4 Pa yields a mean tensile stress of +40 MPa if the substrate is coated by a hard baked photoresist, or of +350 MPa if the substrate is silicon oxide or AlSi. Stress gradient has been determined by analyzing the profile of surface micromachined cantilevers. The only drawback of using Ta in micromachining applications is its high resistivity, which is, for example, not suitable for RF switches. To overcome this problem, a stack of 1 μ m AlSi/ 1 μ m Ta/ 1 μ m AlSi has been realized. The sheet resistance of this stack is 27 m Ω /sq., whereas its mean stress, is +350 MPa tensile. In conclusion it has been demonstrated that the low tensile stress (+ 40 MPa) and the low creep rate of sputtered Ta, makes this material superior to Al, specifically for applications where reliability is a critical issue. Furthermore, it has been shown that sandwiching Ta between two AlSi layers extends the use of this material to low resistivity applications.

U3.4

A MICROMACHINED MONOLITHIC INKJET PRINT HEAD USING PIEZOELECTRIC $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ ACTUATORS. Kyoung-Won Na, Seung-Mo Lim, Won-Youl Choi, Jae-Woo Chung, and Seung-Jin Kim, MEMS Laboratory, Samsung Advanced Institute of Technology (SAIT), Suwon, KOREA.

We fabricate a piezoelectrically driven monolithic inkjet print head that consists of silicon membranes, ink chambers, nozzle guides and nozzles, and piezoelectric $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ (PZT) thin film

actuators. The 10 μ m thick silicon membrane and ink chamber were fabricated in a SOI (Silicon On Insulator) wafer by anisotropic dry etching using ICP-RIE. The volume of ink chamber is 2500 \times 260 \times 100 μm^3 . Nozzle guides and nozzles were successively fabricated in a silicon wafer by wet and dry deep etching. The diameter and length of nozzles are 24 μm and 24 μm , respectively. The piezoelectric actuating PZT layer was integrated on the silicon membrane. The SOI wafer of ink chambers and silicon wafer of nozzles were bonded by SDB (Silicon Direct Bonding) process. The nozzles are embedded at four columns on each chip where their pitch is 1/90 inch at each column. A pulse width of 5 μs and voltage of 30V have been used in printing tests. The drop volume and velocity were measured to be 3pl and 4m/s, respectively.

U3.5

SIZE EFFECTS ON THE MECHANICAL PROPERTIES OF MICRO-DEVICE MATERIALS. Horacio D. Espinosa and Barton C. Prorok, Northwestern University, Dept. of Mechanical Engineering Evanston, IL.

Over the past decade, there has been great motivation to reduce the size of many mechanical systems to the micron and sub-micron scale by fabricating devices out of thin film materials. Whatever the application, successful development of these thin film material requires a thorough understanding of their mechanical properties. The Membrane Deflection Experiment (MDE) was used to achieve direct tensile stressing of free-standing thin film gold specimens [1,2]. The procedure involves applying load with a nanoindenter to the center of the spanning membrane. Simultaneously, an interferometer focused on the bottom side of the membrane, through a specially micro-machined window, records the deflection. The result is direct tension in the gauged regions of the membrane with load and deflection measured independently. We present stress-strain signatures obtained on gold films 300, 500 and 1000 nm thick. Young's modulus of thin film gold was consistently measured in the regime of 53-55 GPa, significantly lower than the value of 78 GPa for bulk Au, however, values reported for thin film Au have varied from 30-78 [3]. A number of size effects on the mechanical properties were observed including yield stress changing with membrane width and film thickness that significantly altered plastic deformation and failure behavior. A clear transition in plastic deformation behavior was observed when the film thickness decreased from 1000 to 500 nm. We will also present results obtained on thin Ultra-Nano-Crystalline Diamond (UNCD) films and gold films coated with 30 \approx SiO₂ layers simulating surface passivation.

1. H.D. Espinosa, B.C. Prorok, and M. Fischer, Proceedings of the SEM Annual Conference on Experimental and Applied Mechanics, June 4-6, 2001, Portland, Oregon, 446-449 (2001).
2. H.D. Espinosa and B.C. Prorok, submitted to J. Mech. Phys. Solids. (2001).
3. W. Nix, Met. Trans. A, Vol. 20A (1989), p. 2217.

U3.6

A MICRO-FLUXGATE MAGNETIC SENSOR USING CLOSELY-COUPLED EXCITATION AND PICK-UP COILS. Won-Youl Choi, Kyoung-Won Na, Sung-Jin Ahn, and Sang-On Choi, MEMS Laboratory, Samsung Advanced Institute of Technology (SAIT), Suwon, KOREA.

This paper presents a micro-fluxgate magnetic sensor composed of a rectangular-ring shaped magnetic core and solenoid excitation and pick-up coils. In order to improve the sensitivity of sensing element, the excitation and pick-up Cu coils are formed as a closely-coupled structure. This unique coil structure allows to excite the magnetic core in an optimal condition with reduced excitation current. The 10mm thick excitation and pick-up Cu coils were electroplated using Cr (300 \AA) / Au (1500 \AA) seed layer. The 2.5 μm thick Ni_{0.8}Fe_{0.2} (permalloy) magnetic core layer was also electroplated with photoresist frame using sputtered Ni_{0.8}Fe_{0.2} seed layer. The core layer was patterned by photolithography and wet etching using dilute sulfuric acid. The magnetic core has a DC permeability of \sim 1100 and coercive field of 0.10e. The magnetic core is easily saturated due to the low coercive field and closed magnetic path for the excitation field. The chip size of the fabricated sensing element is 2.4 \times 2.6mm². Excellent linear size response over the range of -500 μT to +500 μT is obtained with 30V/T sensitivity at 1MHz excitation pulse. The very low power consumption of \sim 50mW was measured. This magnetic sensing element to measure a weak magnetic field is very useful for various applications such as: portable navigation systems including emergency call, military research, medical research, and space research.

U3.7

INTEGRATED MAGNETIC SENSING OF ELECTROSTATICALLY ACTUATED THIN FILM MICROBRIDGES. J. Gaspar^{1,2}, H. Li^{1,3}, P.P. Freitas^{1,3}, V. Chu¹ and J.P. Conde² ¹INESC Microsystems and Nanotechnologies, Lisbon, PORTUGAL. ²Department of Materials Engineering, Instituto Superior Técnico, Lisbon, PORTUGAL.

This work reports on the fabrication and characterization of thin film micromachined bridges that are electrostatically actuated and that have the capability of on-chip movement monitoring by using an integrated magnet-sensor configuration. The integrated magnetic sensing along with thin film technology and surface micromachining show that it is possible to combine MEMS with control electronics on large area substrates.

Bilayer bridges of aluminum (Al) and hydrogenated amorphous silicon (a-Si:H) are processed at low temperature ($\leq 110^\circ\text{C}$) on a glass substrate. An Al electrode (gate) is placed beneath the structures to enable electrostatic actuation. In order to monitor the movement of the structures, a cobalt-platinum (CoPt) permanent magnet is deposited and patterned on the top of the microbridges and a spin-valve (SV) magnetic sensor is positioned on the substrate close to the bridge.

The structure bends towards the substrate when a voltage is applied between the gate counter electrode and the bridge. The deflection is monitored with two different techniques: (1) magnetic detection: the deflection of the microstructures will change the magnet position with respect to the SV sensor, varying its magnetoresistance (MR) signal; (2) optical detection: an He-Ne laser is focused on top of the structures and the reflected light is read with a quadrant detector, whose signal is proportional to the deflection of the beam. From the measured deflection vs. voltage curves, it is possible to estimate mechanical and structural parameters such as the flexural rigidity (EI). An electromagnetomechanical model will be described in detail and used to analyze the experimental results. Devices with varying dimensions are characterized under different deflection regimes. The response of the structures to quasi-DC, AC and step voltages is measured. For low levels of excitation, sub-nanometric precision is achieved. The AC measurements show that the structures have resonance frequencies in the MHz range.

U3.8
ENHANCED HYDROSTABILITY OF MESOPOROUS SILICATE FILMS THROUGH INCORPORATION OF ALUMINA OR ORGANIC FUNCTIONALITY. Sarany Singer, Darren Dunphy, Sandia National Laboratories, Materials Chemistry Department, Albuquerque, NM; C. Jeffrey Brinker, Sandia National Laboratories and University of New Mexico, Department of Chemical and Nuclear Engineering, Albuquerque, NM.

Highly porous silicate films (i.e. those obtained via a surfactant-templated route) are attractive as fluidic platforms, ion-permeable membranes, and as potential encapsulation media for biological or chemical sensors. For example, entrapment of biological components such as enzymes or whole cells in robust porous silica films may provide protection from environmental stress, and allow the rapid transport of analyte/nutrient/waste etc. throughout the silicate matrix. However, templated silicate thin films are unstable in aqueous media, as a result of their high surface areas and low thicknesses, coupled with moderate solubility of silica in water (ca. 100 ppm at pH 7). To circumvent this serious issue, two general methods of increasing film hydrolytic stability will be discussed: addition of alumina to templated silica films, through addition of aluminum compounds to the silica sol or post-synthesis grafting of aluminates to the as-deposited film. Addition of even 0.02 Al/Si significantly increases film stability at neutral pH, as determined with spectroscopic ellipsometry. The relationship of film stability to synthesis conditions (% Al, identity of Al precursor, type and concentration of surfactant template) and solution composition (pH, ionic strength) will be compared for films prepared by direct synthesis versus post-synthesis alumina grafting. The effect of Al concentration on film structure, as determined by x-ray diffraction and nitrogen adsorption, will also be discussed. Addition of alumina to porous silica only increases the hydrolytic stability of films that have been thermally treated under conditions not suitable for biomolecular or cellular entrapment, however. For this reason, we have also investigated the use of organosilane chemistry to increase aqueous stability in as-deposited films. Examples of organic functionality that we have evaluated for increased hydrolytic stability include alkyl and amine groups, incorporated into our films using either pendant or bridged organosilane precursors.

U3.9
DEVELOPMENT OF TiAl ALLOY FILMS FOR APPLICATION IN MEMS BIMORPH ACTUATORS. Xuexuan Qu, Qingxin Zhang, Quanbo Zou, N. Balasubramanian, Institute of Microelectronics, Singapore, SINGAPORE; Ping Yang, Singapore Synchrotron Light Source (SSLS), National University of Singapore, SINGAPORE; Kaiyang Zeng, Institute of Materials Research & Engineering, Singapore, SINGAPORE.

In this letter, we demonstrate the feasibility of applying TiAl alloy

films for the fabrication of bimorph actuators, which is aimed at improving the MEMS performance. The TiAl alloy films were prepared by thermal annealing at 400C of Ti/Al multilayers, which were deposited by DC magnetron sputtering from Ti and Al targets. The microstructure and surface morphology of TiAl alloy films were analyzed by XRD and SEM, which showed that TiAl alloy film is formed in the mixed phases of TiAl₃ and Ti₃₆Al₆₄, depending on the deposition conditions. The surface of TiAl films was very smooth and no hillocks were formed. The resistivity of TiAl film is about 9 $\mu\Omega\text{cm}$, and the stress is about 200MPa. Our nano-indenter measurements showed that the Young's modulus and hardness of TiAl alloy films are 175GPa and 6.5 GPa respectively, which are larger than that of Al and compatible to Si. We have successfully fabricated the bimorph actuators based on the TiAl alloy films and our test cantilevers up to 500 μm long showed straight with tip bending as small as $\pm 5\mu\text{m}$, indicating negligible stress gradient in TiAl film. The TiAl-based bimorph actuators were fabricated according to standard process steps, which included the release of the actuator by dry etch of poly Si or Si with SF₆ or XeF₂. Our preliminary testing results indicated that TiAl alloy film has potential application for bimorph actuators.

U3.10
A METHOD FOR QUANTIFYING FORCE-DEPENDENT PROPERTIES OF BEAM STRUCTURES IN MESO-MEMS SYSTEMS. Anupama B. Kaul, Tomasz Klosiowski, and Joshua Liu, Advanced Technology Center, Motorola Labs, Schaumburg, IL.

An approach for measuring force-dependent properties of microscopic structures commonly found in MEMS has been developed. The system has the capability of measuring forces and deflections of the order of micro-newtons and micro-meters, respectively. By implementing a visual inspection system, force can be selectively applied to localized areas on a beam, and the resulting force-deflection characteristic obtained at selected locations. From this data the beam stiffness can be calculated coupled with other parameters such as the effective elastic modulus. In addition, by applying a known mechanical force and deflection, correlation to electrostatic forces can be obtained, from which the magnitude of the electrostatic actuation and feedback effect can be extracted. The stiffness of several beam geometries that have inherently different spring constants, which influence actuation voltages, was obtained experimentally and the results were compared and correlated to simulation. Simulations were performed using the finite element method and ANSYS code. The technique also allows the determination of time and environment related property changes such as seen in stiffness, which will be discussed. In addition, characterization of other force-dependent parameters such as contact resistance and stiction is also possible using this set-up.

U3.11
MICROSTRUCTURE AND MORPHOLOGY OF HIGH REFLECTIVITY THIN FILMS FOR MEMS MICROMIRRORS. Yoosuf Picard, Steven Yalisove, University of Michigan, Dept of Materials Science and Engineering, Ann Arbor, MI; David Adams, Sandia National Laboratories, Advanced Manufacturing Processes Lab, Albuquerque, NM; Olga Spahn, Sandia National Laboratories, Compound Semiconductor Research Lab, Albuquerque, NM.

The development of micromirrors intended to handle optical power levels approaching 1 W have led to close examination of laser-induced damage attributed to thermal absorption. Common to virtually all actuated micromirrors is extreme thermal isolation. Therefore, minimizing thermal absorption by maximizing reflectivity has led to research of high-bandwidth, reflective thin films and Bragg reflectors for target wavelengths. However, depositing multilayer films on plane wave micromirrors can lead to deviation from a desired curvature due to residual intrinsic stress. Maintaining flatness of a micromirror (2 μm thick) to within $\lambda/10$ requires keeping the film stress below ± 10 MPa. In this work, multilayer thin films consisting of Cr, Au, Si, and Si₃N₄ are deposited on Si (100) substrates using DC planar magnetron sputtering. Au is reflective (90% and above) over a range of infrared wavelengths. Cr is a typical adhesion-promoting layer between Au and Si. Alternating successive layers of low- and high-refractive index thin films grown at thicknesses that satisfy the Bragg condition optimize reflectivity at a particular wavelength of incident radiation. Si₃N₄ (low n) and Si (high n) constitute the proposed dielectric bi-layers. The influence of Ar pressure (for Cr, Au, and Si films) and Ar/N₂ partial pressures (for reactive deposition of Si₃N₄) will be correlated to both the optical properties and stress of the thin films. The stress of multilayers deposited onto blank wafers is determined through curvature measurements by a laser-scanning device and applying Stoneys equation. The curvature changes introduced by multilayer coatings deposited onto individual MEMS micromirrors are analyzed by white light interferometry. Close examination of the roughness at each thin film interface is conducted by cross-sectional transmission electron microscopy (TEM). X-ray diffraction (XRD) shows crystal structure of each film. Reflectivity of each mirror coating is measured by spectroscopy.

U3.12

MECHANICAL PROPERTIES OF BORON DOPED Si MEMBRANES. Gabe Kuhn, Todd Myers, Susmita Bose and Amit Bandyopadhyay, School of Mechanical and Materials Engineering, Pullman, WA.

Mechanical properties of materials used in microelectromechanical systems (MEMS) are highly influenced by their processing steps. In our research, PZT film actuated micro-machined Si substrates are being developed for numerous applications in which membranes are actuated in flexural mode, which has been the most popular mode of operation for various MEMS devices. In these devices, boron doped Si/SiO₂ membranes are fabricated first, and then Ti/Pt layers are deposited. Sol-gel PZT layers are spin coated on top of the membrane. Boron doping is used in Si as an etch stop to process membranes with predetermined thickness using standard wet etching techniques. To understand the influence of boron doping and oxide layer on residual stress development, boron doped Si/SiO₂ membranes were fabricated into fixed-end beams, which were studied via nano-mechanical testing using a Berkovich indentation tip. Initial results from boron doped 2 micron thick fixed end beams show an increase in overall beam stiffness roughly two orders of magnitude higher than the expected stiffness value of silicon beams without residual stresses. The influence of the residual stresses due to the presence of the SiO₂ layer was observed by its removal and subsequent beam testing. It was found that the stiffness of the beam dropped by approximately 37%. Regrowth of oxide and subsequent removal lowers the boron concentration in the Si due to diffusion. Testing of those beams with lower boron concentration show the influence of boron on the residual stress development of these structures. The presentation will include processing of these beams, and their nano-mechanical testing results.

U3.13

FABRICATION OF CONDUCTOMETRIC SENSOR ARRAY FOR CREVICE CORROSION STUDY. Xiaoyan Wang, University of Virginia, Dept of Electrical and Computer Engineering, Charlottesville, VA; Robert G. Kelly, University of Virginia, Dept of Materials Science and Engineering, Charlottesville, VA; Michael L. Reed, University of Virginia, Dept of Electrical and Computer Engineering, Charlottesville, VA.

Microfabricated crevice corrosion samples have been employed in experiments that provide important information desired for developing an accurate, comprehensive, and reliable crevice corrosion model. Acquiring real-time spatial information of crevice corrosion is essential in studying the corrosion process. Integration of arrays of solid-state microsensors, such as conductometric sensors, and ion concentration potentiometric sensors, into the crevice corrosion samples will allow for in-situ real-time data acquisition. In the present work, conductometric sensor arrays and crevice corrosion samples are made via the techniques developed for thin film semiconductor processing and microelectromechanical systems (MEMS) fabrication. The crevice corrosion testing sample is constructed by coupling a crevice former to a crevice substrate and has a uniform crevice gap. A conductometric sensor array is incorporated into the crevice former built on a silicon wafer. Each of the sensors is composed of a pair of thin film gold electrodes, which enables in-situ spatial conductivity analysis of crevice corrosion. In combination with built-in circuits, information about metal ion concentration and active chemistry inside the crevice is achieved.

U3.14

A NANOFUIDIC DEVICE FABRICATED BY E-BEAM LITHOGRAPHY FOR DNA ANALYSIS. Han-Jun Kim, Michael Pio, Luke P. Lee, University of California, Berkeley, CA; Berkeley Sensor & Actuator Center, Department of Bioengineering; Eric Anderson, Lawrence Berkeley National Laboratory, Berkeley, CA.

By incorporating with electron beam lithography and micro-fabrication technology, a nanofluidic device is designed and fabricated to embed with sub-micron width channels for the analysis on DNA molecules. By different nature of ionic current reduction and/or translocation mobility of each homopolynucleotide acid, the single channel method can be considered as a promising tool for direct reading of single molecule DNA. Even with the potentials, however, limitations in analysis have been imposed because of the limited understanding on the mechanisms of unfolding and transporting polynucleotides as well as the difficulty of relating flow characteristics in the nano-scales to the detection capability governed by the molecular transport. A nanofabricated fluidic channel device embedded with various widths in sub-micron range (100~500 nm) are generated by electron beam lithography, and subsequently transferred to silicon and glass substrates with CF₄ plasma. The substrate side with the fluidic channels is bonded to a mobile ion-containing glass wafer by performing anodic bonding across an inserted thin layer of amorphous silicon in between. Hydrodynamic parameters for the

contracted parts of the channels with sub-micron widths in relation to the wider parts are best described with an analogy of flow resistance circuitry. Flow mobility is a measure of the flow rate generated by the applied field of pressure gradient or driving potential. Migration at the entry and diffusion at the translocated region are suitably expressed with deformity and diffusivity of the molecules depending on the size effect of the channel and on the solution-specific measures such as concentration, temperature and pH. Combined with electrical sensing and surface modification treatment, this work will help enable direct reading of unmodified homopolymer nucleotide fragments at a single molecule level, potentially innovating functional genomics and single nucleotide polymorphism (SNP) study.

U3.15

SURFACE MODIFICATION OF CYCLIC OLEFINIC COPOLYMERS FOR BIO-MEMS MICROFLUIDIC DEVICES. Chong H. Ahn, Sanghyo Kim, Hyoung J. Cho, Center for Microelectronics Sensors and MEMS, University of Cincinnati, Cincinnati, OH; Suresh Murugesan, Gregory Beaucage, Polymer Research Center, University of Cincinnati, OH.

Cyclic Olefin copolymers (COC) are a new class of polymers that may prove to be extremely useful in injection molding of micron scale fluidic devices. In microfluidic devices it is desirable to have a hydrophilic surface such as in flow driven by capillary action. However, such hydrophilic surfaces tend to display protein deposition when contacted with blood unlike hydrophobic surfaces. Alternatives to capillary action are then needed to control fluid flow for hydrophobic surfaces. Our goal in this research is to tune the slightly hydrophobic COC surfaces through simple surface modifications that are amenable to injection molding and other processing methods. In this study, the surface of an injection molded microfluidic component made from COC was modified in order to change the surface properties important to bio-fluidic devices. Some of the techniques used in this study were plasma treatments and ASG (aerosol gel) coating. Plasma treatments were conducted by using O₂, CF₄ and their combination gas. O₂ treated surfaces became hydrophilic with increasing time of treatment. Combining O₂ and CF₄ made the surfaces more hydrophobic compared to CF₄ only. The structural changes after the plasma treatments were examined by ATR (Attenuated Total Reflectance) spectroscopy. Titania and silica particles from the ASG process were synthesized from titanium iso-propoxide and tetraethoxysilane, respectively. Titania coated surfaces became more hydrophilic and the silica coated surfaces did not have much change in their surface characteristics. The hydrophobicity of the plastic surfaces was measured by their contact angle with water. The implication of these treatments on bio-fluidic devices and their adaptation to the injection molding process will also be discussed.

SESSION U4: BIOMEMS

Chairs: Luke P. Lee and Jeffrey T. Borenstein
Wednesday Morning, April 3, 2002
Olympic (Argent)

8:00 AM *U4.1

HIGH PERFORMANCE MICROMACHINED PLANAR FIELD-ASYMMETRIC ION MOBILITY SPECTROMETERS FOR CHEMICAL AND BIOLOGICAL COMPOUND DETECTION. Raanan A. Miller, Sionex Corporation, Wellesley Hills, MA; Angela Zapata, Draper Laboratory, Cambridge, MA; Erkinjon G. Nazarov, Gary A. Eiceman, Department of Chemistry and Biochemistry, New Mexico State University, Las Cruces, NM.

Ion mobility spectrometry (IMS) is a highly sensitive, quantitative method for organic compound detection with detection limits as low as parts-per-trillion. Unlike mass spectrometry, IMS operates at atmospheric pressure and therefore is easily configured as a field portable, handheld device. IMS is presently the choice technology for chemical warfare agent, illicit drug, and explosives detection. Many other applications could also be addressed by IMS, however, the present devices, which measure the time-of-flight of an ion are too expensive and can suffer from poor compound resolution. Many new chemical/biological applications could be addressed by a low cost IMS device with improved resolution. One such device is the micro-machined Planar Field-Asymmetric Ion Mobility Spectrometer (PFAIMS). Rather than measuring time-of-flight, it operates as an electronically tunable ion filter, where only the compound (ion) of choice passes through the filter to a detector electrode, other ions are neutralized and not measured. Unlike the conventional IMS which must operate in a low electric field regime where the mobility is constant with field, the PFAIMS operates in a high field regime (electric field > 10,000 volts/cm) where the mobility of an ion depends on the applied field strength. This high field regime provides PFAIMS with an additional knob to adjust/improve IMS resolution. The PFAIMS has been shown to resolve, in under a second, compounds such as xylene isomers which can not be resolved by

conventional IMS, mass spectrometers, and only over relatively long times by gas chromatographs (GC.). The PFAIMS has been used as a detector for a GC, providing a second dimension of information to significantly improve the identification of the individual chemical constituents of a complex mixture. The PFAIMS has also been shown to detect low concentrations of insect pheromones and has been applied to non-invasive medical diagnosis through breath analysis. This spectrometer meets a market need of low cost, quantitative, highly sensitive chemical detection, and offers promise in many biomedical applications.

8:30 AM *U4.2

MICROCANTILEVER BIOSENSORS. Thomas Thundat, Life Science Division, Oak Ridge National Laboratory, Oak Ridge, TN.

In recent years there has been a great interest in the development of biological sensors based on microcantilever platform. The microcantilever resonance frequency varies sensitively as a function of molecular adsorption. In addition, when the adsorption is confined to one side of the cantilever, the cantilever undergoes deflection due to adsorption-induced stress. Bio-specificity is accomplished by coating the cantilevers with immobilized selective layers on one side. Since cantilever motion originates from the free energy change induced by specific biomolecular binding, this technique offers a common platform for high-throughput label-free analysis of protein-protein binding, DNA hybridization and DNA-protein interactions, as well as drug discovery. The total mass adsorbed can be measured using changes in resonance frequency. By simultaneous measurement of cantilever bending and resonance frequency shift it is possible to separate specific and nonspecific adsorption in most biosensor applications. We will present recent results of detection of single-base pair mismatches in DNA, detection cancer-related proteins such as PSA, and cardiac markers.

9:00 AM *U4.3

A PROTEIN PRECONCENTRATOR BASED ON A THERMALLY-SWITCHING POLYMER MONOLAYER. Dale L. Huber, Michael A. Samara, Bruce C. Bunker, Ronald P. Manginell, Sara S. Sokolowski, Sandia National Laboratories, Albuquerque, NM.

A protein pre-concentrator based on a microhotplate architecture, utilizing a thermally-switching polymer monolayer as an active surface has been developed. A low heat capacity, thermally-isolated membrane, heated with a thin-film gold resistive heater, has been developed to drive a phase transition in poly(*n*-isopropyl acrylamide) (PNIPAM). PNIPAM has a switchable affinity for proteins, resisting absorption in its low-temperature hydrophilic state and adsorbing them in its high-temperature, more hydrophobic state. It has also been shown to allow rapid desorption of a protein layer when its temperature is decreased. Incorporation of this polymer into a microhotplate allows rapid adsorption and desorption cycles, and may have applications as a pre- or re-concentrator on chip-based proteomic devices. Prototypes of this device have been fabricated and its temperature response modeled, then calibrated through monitoring of the resistance of the gold heater lines, as well as through the use of thermo-chromic aqueous solutions. The devices interactions with myoglobin as a model protein have also been studied, and the behavior is as predicted. The adsorptions of some mixtures of proteins have been studied and these results will be discussed. Computer control of the device has been achieved, allowing for the programming of routines to capture and release biologically relevant samples.

9:30 AM U4.4

NOVEL INTERFACE TO BIOLOGICAL SYSTEMS FOR RETINAL PROSTHESES. Mark C. Peterman, Department of Applied Physics, Stanford University, Stanford, CA; Harvey A. Fishman, Department of Ophthalmology, Stanford University, Stanford, CA.

The development of retinal prostheses requires a method for interconnecting an imaging system to the retina. Such a system must be able to individually address and stimulate retinal neurons, a significant advance from current technology. As a step toward this goal, we present a novel electronic-to-biologic interface using microfabricated apertures in a silicon substrate. Apertures are created in a thin silicon nitride membrane, after which the surface is appropriately modified to support cell growth. Excitable cells are seeded on the device and imaged using Ca^{2+} -sensitive dyes in either an inverted or confocal microscope. Using rat pheochromocytoma (PC12) cells and rabbit retinal pigment epithelial cells, we show the ability to stimulate through the apertures, from many cells in a localized area down to a single cell over the aperture. The device allows for the stimulation of individual cells at precise locations, a necessary requirement for future high-resolution retinal prostheses.

9:45 AM U4.5

BIOMIMETIC CELL SORTING WITH SELECTIN PROTEINS.

Wesley C. Chang, Luke P. Lee, Dorian Liepmann, Berkeley Sensor and Actuator Center, University of California, Berkeley, CA.

New approaches for separating and sorting cells in miniature biological assay systems must function with microliter sample sizes and minimize preparation steps while still generating high sample throughput. These requirements can be fulfilled by harnessing the selectivity of adhesion proteins for specific cell types. One such paradigm uses the affinity of selectin proteins for leukocytes, leukocyte progenitors, and even some blood-borne cancer cells. Physiologically, selectins mediate recruitment of leukocytes along the lumen of blood vessels near sites of injury. Cells adhere reversibly to selectin-presenting surfaces and can roll slowly along these surfaces under the influence of fluid shear from passing blood. The use of this rolling mechanism *in vitro* to capture leukocytes from fluid samples has been demonstrated using micro-channels containing a lattice of posts to maximize surface area exposed to flowing sample. The structured flow channels, which provide surface area to volume ratio of $0.15\mu\text{m}^{-1}$, were fabricated in silicon with conventional micromachining, and all surfaces were coated with silicon dioxide. Chimera proteins consisting of the cell-binding segment of E-selectin and a water-soluble IgG tail were immobilized at various area densities to the silicon dioxide surfaces via adsorption from buffered solution. Suspensions of HL-60 cells, bearing ligands for E-selectin, were flowed through the channels and adhered to the flow surfaces even under high free stream flow rates (highest: 16,000 microns/sec). At saturation density of >2000 molecules/ μm^2 , the adsorbed E-selectin chimera restricted the cell rolling speed to an average of 0.14% of the speed of the free stream flow, while in the lattice of posts average transit speed of cells was just 0.04% of that of the free stream. Local concentration of cells in this lattice rose quickly from the 300,000 cells/mL of the original sample to 15.8 million/mL in 3 min, a 52-fold increase.

10:15 AM U4.6

MICROFABRICATED DEVICE FOR IMPEDANCE-BASED DETECTION OF BACTERIAL METABOLISM. Rafael Gómez, Rashid Bashir, School of Electrical and Computer Eng; Arun K. Bhunia, Dept of Food Science; Michael R. Ladisch, Dept of Agricultural and Biological Eng, Purdue Univ, West Lafayette, IN.

We present the fabrication and characterization of a microfabricated device for impedance-based detection of a few live bacterial cells. Impedance-based detection of bacteria is a well established technique in microbiology, but, to the best of our knowledge, it had never before been attempted at the microscale. Impedance-based detection relies on measuring changes in the AC impedance of two electrodes immersed in a liquid where the bacteria are cultured, caused by the release of ionic species by metabolizing bacteria. Rapid detection of a few cells (1 to 10) is possible if the cells are confined into a volume on the order of nanoliters. A microfluidic biochip prototype has been fabricated incorporating sealed fluidic channels, planar fluidic interface ports, and integrated metal electrodes, to test this miniaturized assay. The biochip consists of a network of channels and chambers etched in a crystalline silicon substrate, with widths/lengths between 100 and 800 μm and a depth of 12 μm . The complex impedance of bacterial suspensions is measured with interdigitated platinum electrodes in a 5.27nl chamber in the biochip at frequencies between 100Hz and 1MHz. A circuit model of the electrodes is fitted to the measured impedance curve, and some of the parameters extracted from the fit are used as the detection signals. Impedance measurements on suspensions of the bacteria *Listeria innocua*, *Listeria monocytogenes*, and *Escherichia coli* in a low conductivity buffer demonstrate that, under the current experimental conditions, the minimum detection level is between 50 and 200 live cells, after two hours of incubation. This is the first demonstration of microscale impedance-based bacterial detection.

10:30 AM U4.7

DIELECTROPHORETIC SEPARATION OF LIVE AND HEAT-TREATED CELLS OF LISTERIA ON MICROFABRICATED DEVICES WITH INTERDIGITATED ELECTRODES. Haibo Li, School of Electrical and Computer Engineering, Rashid Bashir, School of Electrical and Computer Engineering, Department of Biomedical Engineering, Purdue University, West Lafayette, IN.

Listeria monocytogenes is a ubiquitous pathogenic bacterium that can be found in soil, water and food. This bacterium is very resistant to heat, acid or saline environments, and can multiply at temperatures as low as $+4^{\circ}\text{C}$. The effects of listeriosis include septicemia, meningitis, encephalitis, and intrauterine or cervical infections in pregnant women, causing 415 deaths in the US each year. As few as 1000 or less bacteria can cause illness in persons with weak immune systems (elderly, children, cancer patients, etc.). Our group is actively developing biochips to fast detect, manipulate and separate *Listeria monocytogenes* cells from food samples. It is of great importance to separate the dead bacteria from the live ones in order to detect effectively *Listeria monocytogenes* in food processing since dead L.

monocytogenes is not pathogenic. For safety purpose, a non-pathogenic subgroup of *Listeria*— *Listeria innocua* was used in our experiments. Dielectrophoretic separation of live and heat-treated *Listeria innocua* cells was achieved with great efficiency on the micro-fabricated devices with interdigitated electrodes by considering the difference of dielectric constant between alive and dead cells. The dependency of the applied AC signal's frequency on the dielectrophoresis of different cells is studied and discussed.

10:45 AM U4.8

ENVIRONMENTALLY SENSITIVE HYDROGELS PATTERNED ON SILICON MICROANTILEVERS. Rashid Bashir, School of Electrical and Computer Engineering, Department of Biomedical Engineering; J. Zachary Hilt, Biomaterials and Drug Delivery Laboratories, School of Chemical Engineering; Amit K. Gupta, School of Electrical and Computer Engineering; Nicholas A. Peppas, Biomaterials and Drug Delivery Laboratories, School of Chemical Engineering, Department of Biomedical Engineering; NSF IGERT Center on Therapeutic and Diagnostic Devices, Purdue University, West Lafayette, IN.

A process was developed for patterning thin films of environmentally sensitive polymers on silicon microcantilevers. Extensive research has been focused on utilizing microcantilevers as highly sensitive chemical, physical, and biological microsensors. Hydrogels can be made sensitive to environmental conditions, such as temperature, pH, magnetic field, electric field, and ionic strength, through the choice of functional groups in their polymer network. By patterning these environmentally sensitive hydrogels onto silicon microcantilevers, microsensors can be prepared for MEMS and BioMEMS applications. We studied a cross-linked poly(methacrylic acid) network containing significant amounts of poly(ethylene glycol) dimethacrylate. This gel exhibits a swelling dependence on pH. By increasing the environmental pH above the pK_a of poly(methacrylic acid) to cause ionization of the carboxylic acid groups, electrostatic repulsion is produced along the main polymer chains causing the polymer network to expand and swell. Therefore, a pH change induces swelling or shrinking of the polymer network and creates stress on the microcantilever surface causing it to bend. In this study, silicon microcantilevers were fabricated on p-type (100) SOI wafers. Covalent adhesion was gained between the polymer film and the silicon surface through modification of the silicon surface with γ -methacryloxypropyl trimethoxysilane. Microcantilevers were fabricated with various lengths, widths, and thickness. Polymers were patterned onto the silicon microcantilevers utilizing standard photolithography techniques. A Karl Suss MJB3 mask aligner was utilized to achieve alignment with 0.1 μm precision. Images of the patterned hydrogels were obtained using optical microscopy and scanning electron microscopy. The bending of the microcantilever was observed optically utilizing an optical microscope. Quantitative measurements of the bending were obtained using an atomic force microscopy system. This research was funded by NSF Grant DGE-99-72770.

11:00 AM U4.9

ULTRA-SENSITIVE BIOMOLECULE SENSING VIA MICROSCALE SURFACE ENHANCED RAMAN SPECTROSCOPY. Elif Ertekin, Luke P. Lee, University of California at Berkeley, Berkeley Sensor and Actuator Center, Berkeley, CA.

The realization of a stand-alone, portable, fast microsystem for ultra-sensitive biomolecule sensing via surface enhanced Raman spectroscopy (SERS) is important for improved on-site detection. Applications for such a device include detection of toxins in water, food, and air. A complete microsystem must include the active sites required to induce surface plasmon resonances, which are critical to the signal enhancements in SERS. However, to fully-integrate a device, the active sites must be realized on substrates in a manner that is compatible with the integration of a microfluidic network for assaying, a light source, and a microspectrometer for detection of the Raman scattered light. We demonstrate two techniques based on self-assembly of polystyrene nanospheres for micropatterning active sites for SERS with feature sizes as small as 100 nm. First, a novel microfluidic technique is used wherein polystyrene nanospheres flowing through microchannels self-assemble into an ordered close-packed arrangement. The channels are encapsulated with polydimethyl siloxane (PDMS), which can easily be removed after the patterning has occurred. The ordered surface is then coated with a thin layer of gold for use as the active sites in SERS. Second, polystyrene nanospheres are spin-coated onto a surface that has been patterned with hydrophobic (Teflon-like) and hydrophilic (oxide) surfaces. The nanospheres selectively self-assemble on the hydrophilic areas; upon deposition of thin gold layers, patterned arrays of SERS active sites are realized. These two patterning techniques enable the inclusion of the active sites into a stand-alone biochemical sensor based on surface enhanced Raman spectroscopy. We have tested the patterned surfaces for Raman activity; as an example, we demonstrate

surface enhanced Raman spectra for two biologically relevant molecules: the DNA base adenine and hemoglobin.

11:15 AM U4.10

SENSITIVE IN-SITU MONITORING OF DNA HYBRIDIZATION USING CAPACITANCE MEASUREMENT. Joonsung Lee, Michael Pio, Ki-Hun Jeong, and Luke P. Lee, Berkeley Sensor & Actuator Center, Department of Bioengineering, University of California at Berkeley, CA.

A capacitance-based biosensor featuring metal/insulator/metal (MIM) structure for direct in-situ detection of DNA hybridization is described. Differential capacitance measurements provide fast and sensitive in situ monitoring of DNA hybridization without DNA labeling. This is an alternative to conventional DNA hybridization detection techniques (such as radiochemical, enzymatic, fluorescent, and electrochemiluminescent methods) which can be cumbersome because of the need for DNA labeling and gel-based separations. In this device, the bottom electrodes are deposited by evaporating 100nm of gold film on a Pyrex glass wafer patterned with photoresist. For the top electrode, 100nm of gold is deposited on a thin glass slide. Parylene, which acts as the spacer between the top and bottom electrodes, is deposited and patterned along the bottom electrodes by a liftoff procedure. The bottom electrodes were then treated with aminoethanethiol, a self-assembling monolayer (SAM) whose amine groups can be attached to the 5' end of ssDNA. The capacitance measurements were taken for various 35-mer polynucleotide sequences. Poly T and poly C oligonucleotides are immobilized on the gold electrodes and a capacitance change is observed when poly A and poly G probes are added. Hybridization of DNA with immobilized DNA on the Au/Cr surface induces charge effects, altering the dielectric properties of the biolayer, and can be detected by the associated change in the measured capacitance. The permittivity and dielectric loss of DNA were investigated at frequencies between 20Hz and 1GHz. The DNA immobilization and hybridization events are verified independently by FTIR and in situ monitoring using fluorescence microscopy. Poly T and poly C oligonucleotides were 3' labeled with fluorescein. Poly A and poly G sequences are tagged with TAMRA at the 3' end. The hybridization events are further characterized by DAPI, a fluorescent marker that is activated upon intercalating to dsDNA.

11:30 AM U4.11

NANOWIRE ELECTRODES FOR IMPROVED NEURAL RECORDING. Karen C. Cheung, Luke P. Lee, Dept of Bioengineering, University of California Berkeley, CA.

The interface between neurons and electrodes is one of the key issues in implantable microdevices and bioelectronics. The ideal electrode requires maximum selectivity and minimum impedance. Selectivity refers to the ability to select a single neuron from a multitude of interconnected cells or even damaged cells. High impedance in an electrode attenuates and filters the measured signal. In addition, a sufficient signal-to-noise ratio is required for data analysis; low electrode impedance gives high signal gain. However, an increase in electrode selectivity with larger geometric electrode size results in increased impedance and noise. To optimize the electrode, the impedance per unit of geometric surface area must be decreased by altering its nanostructure. With the introduction of surface roughness or nanostructures, a square 20 μm x 20 μm electrode has only 400 square microns of geometric (planar) area but a much larger effective area. Electrode materials historically used in neuroscience are gold, platinum, and iridium. The most common surface modifications are wet chemical etching for gold, electroplating platinum black or ion milling the platinum surface, or activating iridium to form iridium oxide. However, they suffer from some drawbacks such as harsh chemical treatment in the case of gold etching, and poor adhesion of platinum powder to the electrode. We present a new method of increasing the effective electrode surface for improved neural recording. Recently, nanowires have been widely studied for use in micro- and nanoscale electronic devices and structures. Here silicon nanowires are grown on a silicon substrate and then coated with gold. Tungsten is used as the metal interconnect; tungsten wires have also long been used as a biocompatible electrode material. These nanowire based neuroMEMS devices with improved neural interfaces are characterized for monitoring neural activity in vitro and compared with control electrodes. Nanowire robustness in cell culture is also examined.

11:45 AM U4.12

PATCH-CLAMP ARRAY DESIGN FOR NEURAL MEMS APPLICATIONS. Loren F. Bentley, Berkeley Sensor and Actuator Center, UCSF/UCB Joint Graduate Group in Bioengineering, Berkeley, CA; Luke P. Lee, Berkeley Sensor and Actuator Center, Department of Bioengineering, Berkeley, CA.

Microfabricated patch clamping devices comprising planar arrays of

individually addressable nozzles, fluidic channels and electrodes have been developed. Patch clamp based electrophysiological techniques are among the most widespread methods in neurophysiology and are used to address a broad range of problems. Among the limitations of the technique are the difficulty of obtaining multiple patches on connected cells or on the same cell, limited stability of patches, and constraints on chemical and optical access to the patched membrane. The parallel array device will enable the formation of multiple seals simultaneously. The structure facilitates visualization of the interior of the patched membrane during electrical recording, as well as delivery of chemicals. The microfabrication technique gives precise control over the capacitive and resistive characteristics of the electrode channels, as well as the flow resistance, which are important factors in patch clamp recording. The device is fabricated using an SOI wafer and Deep Reactive Ion Etching to create an array of cylindrical nozzles, each of which has a core of silicon dioxide and interior walls of silicon nitride. Vertical channel segments and plumbing holes are fabricated by deep reactive ion etching through the wafer. The connecting horizontal channels are formed using SU-8 negative photo resist patterned on Pyrex substrates and bonded to the back of the SOI wafer. The flow properties of the channels were characterized, as well as the effect of varying fabrication parameters such as nozzle geometry, channel geometry, and thin film deposition characteristics on the electrical properties and performance of the patch clamp device.

SESSION U5: DEVICES AND CHARACTERIZATION

Chairs: Ronald P. Manginell and Peter J. Hesketh
 Wednesday Afternoon, April 3, 2002
 Olympic (Argent)

1:30 PM *U5.1

LOW-TEMPERATURE LPCVD MEMS TECHNOLOGIES.
 Roger T. Howe and T.-J. King, Univ. of California at Berkeley,
 Berkeley, CA.

This paper describes recent research on LPCVD processes for the fabrication of micro-mechanical structures after the completion of a standard CMOS. Polycrystalline silicon germanium (poly-SiGe) alloys are attractive candidates for low-temperature MEMS processes, since they can be deposited at much lower temperatures than poly-Si films, yet have excellent mechanical properties. A post-process maximum temperature of 450 C is a conservative estimate for avoiding damage to foundry CMOS wafers with either aluminum or copper metallization. In order to fabricate MEMS at or below this temperature ceiling, it is attractive to use p-type in situ doped poly-SiGe films since adequate conductivity can be achieved without post-deposition annealing. We will review experiments at Berkeley and elsewhere on the mechanical properties of p-type poly-SiGe films. Furthermore, poly-Ge films can be selectively etched in 90 C hydrogen peroxide. Integration with CMOS is greatly simplified by the elimination of HF-etching of oxide sacrificial layers, which can damage the underlying electronic layers. Low-resistance interconnects between the poly-SiGe layers and the CMOS metallization can be accomplished by deposition onto typical barrier metals exposed in contact windows. The implication of post-CMOS integration of LPCVD poly-SiGe MEMS is that the barrier to including MEMS in systems-on-a-chip is lowered dramatically. Over the past two decades, surface micromachining technologies based on LPCVD poly-Si films and SiO₂ sacrificial layers have become highly developed. The design principles and micromechanical library elements, such as flexures, hinges, bearings, and electrostatic actuators, can be imported into an integrated LPCVD poly-SiGe MEMS process with only small modifications.

2:00 PM U5.2

DESIGNING THERMALLY UNIFORM MEMS HOT MICRO-BOLOMETERS. Nicholas Moelders, Martin U. Pralle, Mark P. McNeal, Irina Puscasu, Lisa Last, William Ho, Anton C. Greenwald, James T. Daly, Edward A. Johnson, Ion Optics, Inc., Waltham, MA; Thomas George, Dan S. Choi, Jet Propulsion Laboratory, Pasadena, CA.

Here we describe the evolution of a silicon, MEMS-based chip design developed for infrared gas and chemical detection. The 'sensor-chip,' with integrated photonic crystal and reflective optics, employs narrow-band optical emission/absorption for selective identification of gas and chemical species. Gas concentration is derived from attenuated optical power, which results in a change in device steady point. This change in temperature results in a change in device resistance, via the TCR of the Si. Thermal non-uniformity across the device results in optical 'noise' and accelerates localized thermal and electrical failures. This paper reports the influence of processing and design, on achieving uniformly heated, high reliability devices. Specifically, we examine the role of contacts, drive scheme, electric field distribution, and device thermal distribution on chip design.

FEM analysis was used to validate device performance.

Experimentally the temperature uniformity was characterized using an infrared camera. Experimental results indicate that the design of the contact areas in combination with the device design is essential for the reliable performance of the sensor-chip. Redesigned devices were fabricated and demonstrated as highly-selective gas and chemical sensors.

2:15 PM U5.3

HIGH FREQUENCY MEMS-FABRICATED ULTRASONIC NOZZLES FOR NANOPARTICLES SYNTHESIS. S.C. Tsai, California State Univ at Long Beach, Dept of Chemical Engineering, Long Beach, CA; Y.F. Chou, National Taiwan Univ, Dept of Mechanical Engineering, Taipei, TAIWAN; Y.L. Song, T.K. Tseng, C.S. Tsai, Academia Sinica, Cent for Applied Science and Engineering Research, Taipei, TAIWAN; J. Yoo, Univ of California, Irvine, Dept of Electrical and Computer Engineering, Irvine, CA.

At the 2001 MRS Fall Meeting, we reported that uniform spherical particles 150 nm in diameter have been produced by spray pyrolysis of 6-9µm precursor drops through the new vapor condensation mechanism. Further, for smaller than 30µm-diameter precursor drops, the product particle diameter is independent of the drop size. Thus, spray pyrolysis, chemically flexible and operating at ambient pressure, of uniform precursor drops holds promise for mass production of nanoparticles of advanced functional materials. In this paper, we will report on design, fabrication, and characterization of a 0.5 MHz Si-based ultrasonic nozzle. Uniform drops 15µm in diameter have been produced by air-assisted ultrasonic atomization using such a nozzle. The nozzle is composed of PZT transducers and a multiple-horn silicon resonator with a central channel for liquid flow. It is geometrically configured such that excitation of the transducer creates a standing wave through the nozzle with maximum longitudinal vibration at the nozzle tip. As liquid issues from the nozzle tip, a capillary wave is generated and travels axially in the direction of liquid flow. This temporally unstable wave eventually collapses into drops. Very large gain in the longitudinal vibration amplitude at the nozzle tip can be obtained through the use of multiple sections of Fourier horn of half-wavelength design. Therefore, the required electric drive power of this Si-based multi-horn nozzle is drastically reduced, and transducer failure can be more easily avoided. Silicon-based ultrasonic nozzles have several advantages over conventional metal-based ultrasonic nozzles: stronger electro-mechanical coupling strength, higher acoustic velocity, and potential for mass production of any resonator profile by semiconductor fabrication technology. To the best of our knowledge, no ultrasonic nozzles at such a high frequency have been fabricated heretofore. Supports from the National Science Foundation, USA, the National Science Council and the Academia Sinica, Taiwan are acknowledged.

3:00 PM U5.4

A NOVEL, MICRO- CONTACT POTENTIAL DIFFERENCE PROBE. Matthew Moorman, Peter Hesketh, Jiantao Zheng, and Steven Danyluk, Mechanical Engineering, Georgia Institute of Technology, Atlanta, GA.

This paper details the fabrication, and testing of a novel, micro-contact potential difference (CPD) probe that investigates the chemical and physical properties of surfaces such as wear[1], corrosion or contamination. The 50-micron diameter of the probe, in conjunction with an integrated shielding scheme, has been fabricated to allow high-resolution surface studies. The probe uses Parylene as a flexible substrate material[2] which allows the probe to be bent away from the integrated electronics. This allows the probe to closely approach the surface of study, which yields higher quality signals. A four-mask process was used to create the micro-CPD probe. The first mask defines the circuit pads, wires, and the probe and shielding seed layer on the Parylene by DC sputtering Platinum. The next mask defines a large, 500-micron, annulus of SU-8 that provides mechanical support for the probe and the shielding. SU-8 is an epoxy resin that is deposited and defined in the manner of standard negative photoresists. The mold lines for the electroplating of the external shielding were defined through the 3rd mask using AZ4620 positive photoresist. Gold electroplating follows to create the probe and side shielding. Finally, gold is E-beam evaporated over the probe to define the upper shielding. Probe signals were obtained by scanning a spinning hard disk drive. The probe was electrically connected to the disk, and positioned .5 mm above the surface. Defects such as scratches and adhesives were placed on the disk, which was then rotated under the probe at 1000 rpm. A computer program samples the probe's signal and creates images of the disk. Peak heights in this image show the amplified contact potential difference between the probe and the features. From these images we can reconstruct the features' dimensions, their CPD values, and determine the probe's resolution.

3:15 PM U5.5

A MICROMECHANICAL STUDY OF FREE-STANDING ALUMINUM MICROBEAMS FOR MEMS APPLICATIONS.

Ping Zhang, John C. Bravman, Stanford University, Department of Materials Science and Engineering, Stanford, CA.

We present a study on micromechanical testing of free-standing thin films. We have developed a dedicated sample fabrication process (compatible with standard IC processes) to produce free-standing thin films of different materials and different thicknesses. We have built a custom-designed micromechanical testing apparatus with a load resolution of ± 0.2 mN and a displacement resolution of ± 10 nm. We study the stress and strain responses of pure Al and Al-Ti alloy free-standing thin films under monotonic and cyclic loading conditions. The samples are fabricated on silicon wafers using pure Al and Al-Ti alloy of different Ti concentrations (1 at%, 3 at% and 5 at%) deposited at room temperature and subsequently annealed at 250°C, 400°C and 550°C, respectively. Each microbeam measures 500x50x1 μm ; each die (i.e. the silicon supporting frame) measures 20x7 mm. Monotonic tests include microtensile tests and stress relaxation tests. In microtensile tests, we find that the yield strengths of these microbeams range from 100 MPa to 500 MPa. Yield strength increases with increasing Ti concentration but decreases with increasing annealing temperature. Ductility, on the other hand, decreases with Ti concentration but increases with annealing temperature. In stress relaxation tests, we find that over the same period of time, Al-Ti thin films relax less than pure Al films, while Al-Ti films of higher Ti concentrations relax even less, with the Al-5%Ti films showing almost no relaxation within the equipment resolutions. We fit the relaxation data by a proposed two-Maxwell-element anelastic model. We also present results for cyclic loading. We use transmission electron microscopy (TEM) to reveal the microstructural features of the samples both before and after micromechanical testing, which facilitates our understanding of the mechanical properties of these thin film materials.

3:30 PM U5.6

MICRO-SCALE LIGHT SOURCE AT 253 NM FOR UV SPECTROSCOPY. Paul von Allmen, Claudia Jensen, Mary Harper, Frederic Zenhausern and Dave Wilcox, Motorola Labs, Solid State Research Center, Tempe, AZ.

Gas micro-discharges have received considerable attention over the past years as potential micro-scale light sources in the UV and thereby as an alternative to Hg based devices. A number of experiments have been published using a variety of platforms such as silicon, polymers, glass, and low temperature co-fired ceramics (LTCC). We will present results using commercial LTCC material and we will focus on the characterization of the emission at 253.2 nm stemming from the B to X transition in the XeI excimer. We have considered several geometric arrangements and compositions of the electrodes and we have identified the most favorable pressure range and electrical operating conditions for each of them. In the best configuration, the emission is found to be sufficiently intense and stable for potential micro-scale spectroscopic applications.

3:45 PM U5.7

MICROPACKAGING OF MEMS DEVICES USING THIN FILM ANODIC BONDING. Lauren E.S. Rohwer, Melissa V. Collins, Andrew D. Oliver, Sandia National Laboratories, Albuquerque, NM.

A wafer scale packaging technology has been demonstrated. This technique involves the anodic bonding of Pyrex wafers to released surface micromachined wafers to form transparent micropackages. Besides providing a hermetic seal, the micropackages allow wafer scale release, provide protection during die separation, and offer the possibility of integration with optoelectronics. Full wafer glass-to-polysilicon and glass-to-glass bonds were formed to create the micropackages. Pyrex 7740 glass was selected as the micropackage material and was shown to bond to a variety of thin films used in MEMS (MicroElectroMechanical Systems) fabrication. In this study, Pyrex wafers were anodically bonded to a variety of thin films. The process involves aligning then bonding a Pyrex wafer to 500 μm wide polysilicon rings surrounding the surface micromachines. The Pyrex wafers contain machined recesses to allow the surface micromachines to move out of plane and for electrical access to the bondpads. In another approach, a second Pyrex wafer is bonded to the first, using a thin film intermediate layer. Anodic bonding was performed under applied voltages up to 1000 V, and temperatures ranging from 280 to 400°C in high vacuum (1×10^{-6} Torr), nitrogen, or air. The quality of the bonded interfaces was evaluated using shear strength testing, transmission electron microscopy, energy dispersive x-ray analysis, leak testing, and temperature cycling. The shear strength of the bonds was measured to be 10-15 MPa, with shearing predominantly at the bonded interface. The micropackaging process was found to be compatible with self-assembled monolayer (SAM) coatings. Functionality and yield of micropackaged devices will be presented.

4:00 PM U5.8

WAFER STEPPER ALIGNMENT FOR MEMS APPLICATIONS USING DIFFRACTION GRATINGS. H.W. van Zeijl, J. Slabbekoorn, Delft University of Technology, Laboratory of Electronic Components, Technology and Materials, Delft, THE NETHERLANDS.

In MEMS fabrication the substrates are not always optically flat, and for dual side processing, alignment on the backside is necessary. This put demands on the lithography, especially when a production proven lithography tool, like a waferstepper is required. In this work we investigate the alignment accuracy of a waferstepper on substrates with a 500 micrometer focus offset of the alignment markers and the feasibility of front-to-backwafer alignment. In the ASM PAS 5000 waferstepper, used in this study, a set of diffraction gratings is used as an alignment marker. The alignment marker is illuminated with a laser and the interference pattern is projected through the lens on corresponding gratings on the reticle. The transmitted light is used as an alignment signal. Because the 0th-order diffraction is filtered out the interference pattern at the reticle level is, in theory, independent of focus offset. On a test wafer, arrays of mirror symmetric alignment markers [1] are exposed and etched into the silicon (layer 1). Next this wafer is anodic bonded to a glass wafer of 500 micron thick and a second array of alignment markers (layer 2) is exposed on the glass with a programmed alignment offset. After development, the markers positions of layer 1 and layer 2 are measured using the alignment system. The overlay accuracy between layer1, measured through the glass, and layer 2 is 0.43 micrometer (3 sigma). The silicon-glass wafer is turned upside down, and with KOH etch through the silicon, the layer 1 alignment markers are cleared. Now the mirrored alignment markers on the frontside can be used to align layer 2 on the backside. The measured overlay accuracy is 0.52 micrometer (3 sigma). The overlay accuracy on flat silicon wafers is 60 nm (3 sigma) and with a large focus offset or front- to backwafer alignment the overlay errors are much larger. However, characterization and correction of the sources of overlay errors [2] may improve the overlay performance on MEMS devices.

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2. H.W. van Zeijl, J. Slabbekoorn, Proceedings of the 1st International Conference on Semiconductor Technology, The Electrochemical Society, Vol. 2001-17, may 2001, pp 356-367.