## SYMPOSIUM A

# A: Micro- and Nanosystems

November 30 - December 3, 2003

## Chairs

## Arturo Ayon

College of Engineering University of Texas at San Antonio 6900 N Loop 1604 West San Antonio, TX 78245 210-408-2318

## Marc Madou

Dept. of Mechanical and Aerospace Engr University of California-Irvine Rm. S3231 4200 Engineering Gateway Bldg. Irvine, CA 92697-3975 949-824-6585

## David LaVan

Mechanical Engineering Dept Yale University 9 Hillhouse Avenue New Haven, CT 06511 203-432-9662

## Mark McNie

Malvern Technology Centre QinetiQ Ltd. St. Andrews Rd. Worcestershire, WR14 3PS UK 44-1684-894586

## Somuri Prasad

Materials and Process Sciences Center Sandia National Laboratories Albuquerque, NM 87185-0889 505-844-6966

Proceedings to be published in both book form and online (see ONLINE PUBLICATIONS at www.mrs.org)
as Volume 782
of the Materials Research Society
Symposium Proceedings Series

<sup>\*</sup> Invited paper

## TUTORIAL

FT A: Nanotechnology-Icarus Revisited - The Organic/Inorganic Challenge Sunday, November 30, 2003 8:30 AM - 5:00 PM Room 200 (Hynes)

The morning session of the tutorial will provide an overview of the technologies and processes available for creating nanostructures employing biotechnology and IC fabrication techniques, and combinations of the two. This session will also include an in-depth discussion of the issues and limitations related to manufacturing in the nanodomain and the problems associated with making measurements in the nanometer scale.

The afternoon session will explore biomimetics in NEMS. While biomimetics in the macro-domain often has led to failure in the past (airplanes do not flap their wings as birds do, see Icarus legend), it is believed that biomimetics in the nanodomain might be more successful. Nature has worked much longer on arriving at natural polymers and biological cells than it did at making trees or humans: nature excels at engineering in the nanodomain.

Also in the afternoon, we will address the many issues involved in combining organic with inorganic structures. Combining genetically engineered natural polymers, such as proteins and nucleic acids, with top-down machined structures, promises the advent of a totally new class of sensors and actuators. Many questions remain, though: how to contact neurons; how to go from ionic conduction (biology) to electrons (inorganic electronics); how to combine these widely different manufacturing techniques; how to apply amplification techniques such as PCR to inorganic particles, etc. This tutorial addresses for the first time the important compatibility issues between organic and inorganic manufacturing.

Instructors:

Marc Madou, University of California-Irvine Dennis Polla, University of Minnesota Carlo Montemagno, University of California-Los Angeles

> SESSION A1: Nanotechnology I Chair: David LaVan Monday Morning, December 1, 2003 Room 200 (Hynes)

## 8:30 AM \*A1.1

 $\begin{array}{c} \textbf{Developing Nanoscale Materials using Biomimetic Assembly} \\ \textbf{Processes.} \ \textbf{George D Bachand}^1, \ \textbf{Susan B Rivera}^1, \ \textbf{Andrew K Boal}^1 \end{array}$ 

Joseph M Bauer<sup>2</sup>, Ronald P Manginell<sup>2</sup>, Jun Liu<sup>3</sup> and Bruce C Bunker<sup>1</sup>; <sup>1</sup>Biomolecular Materials and Interfaces, Sandia National Laboratories, Albuquerque, New Mexico; <sup>2</sup>Micro-Total-Analytical Systems, Sandia National Laboratories, Albuquerque, New Mexico; <sup>3</sup>Chemical Synthesis and Nanomaterials, Sandia National Laboratories, Albuquerque, New Mexico.

The formation and nature of living materials are fundamentally different from those of synthetic materials. Synthetic materials generally have static structures, and are not capable of adapting to changing environmental conditions or stimuli. In contrast, living systems utilize energy to assemble, reconfigure, and dismantle materials in a dynamic, highly non-equilibrium fashion. The overall goal of this work is to identify and explore key strategies used by living systems to develop new types of materials in which the assembly, configuration, and disassembly can be programmed or "self-regulated" in microfluidic environments. As a model system, kinesin motor proteins and microtubule fibers have been selected as a means of directing the transport of molecular cargo, and assembly of nanostructures at synthetic interfaces. Initial work has focused on characterizing and engineering the properties of these active biomolecules for robust performance in microfluidic systems. We also have developed several strategies for functionalizing microtubule fibers with metal and semiconductor nanoparticles, and demonstrated the assembly of composite nanoscale materials. Moreover, transport of these composite assemblies has been demonstrated using energy-driven actuation by kinesin motor proteins. Current work is focused on developing mechanisms for directing the linear transport of microtubule fibers, and controlling the loading/unloading of nanoparticle cargo in microfluidic systems. \*Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

## 9:00 AM A1.2

Formation of Two- Dimensional Cellular Foams from Carbon

Nanotube Arrays. Nirupama Chakrapani<sup>1</sup>, Bingqing wei<sup>1</sup>, alvaro carrillo<sup>2</sup>, ravi s kane<sup>2</sup> and pulickel m ajayan<sup>1</sup>; <sup>1</sup>Department of material science an dengineering, Rensselaer polytechnic institute, troy, New York; <sup>2</sup>Howard P. Issermann department of chemical engineering, rensselaer polytechnic institute, troy, New York.

Carbon nanotubes have numerous potential applications as a result of their outstanding structural, mechanical, and electrical properties. The pursuit of these numerous applications is hindered by the difficulty of manipulating these materials and controlling their organization. We report the formation of intriguing and visually arresting cellular structures in various shapes and sizes in vertically aligned carbon nanotube arrays. The mechanisms giving rise to pattern formation would be discussed. We will also discuss how pattern formation may be influenced by controlling the chemistry of the substrate as well as by processing conditions. Applications of these nanostructured materials will also be discussed.

## 9:15 AM <u>A1.3</u>

Carbon Nanotube Modification Using BaF2 Vapor in Ultra-High Vacuum Environment. Francisco Santiago, Victor Gehman, Kevin A Boulais and Karen Long; Dahlgren Division Naval Surface Warfare Center, Dahlgren, Virginia.

Carbon nanotubes have attracted a lot of attention in the scientific community due to their unique properties and potential applications. One of the most promising application is carbon nanotube transistors. The motivation of this work is to find ways to connect carbon nanotubes directly to silicon using Ba as a chemical link. We studied the chemical interactions between carbon nanotubes and BaF2 vapors using x-ray photolectron spectroscopy (XPS) and atomic force microscopy (AFM). Surfaces of silicon wafers were chemically modified to allow the epitaxial growth of BaF2 using molecular beam epitaxy (MBE). Samples containing 2D single crystal islands of BaF2 were covered with carbon nanotubes with an average coverage of 10 nanotubes per um2. The samples were transferred to an outgas station inside the MBE system and heated to 900oC for two hours in a pressure of 10-9 mbar. XPS C1s data before and after heat show a major change in the nature of the carbon nanotube electronic states. In addition XPS shows formation of a Ba-C carbide like bond and no presence of fluorine. AFM images of the same region taken before and after heat exposure show remarkable changes in the surface morphology of the carbon nanotube wall. Results of the changes in the Ba-Si interface will be shown.

## 9:30 AM A1.4

Tunneling Current-Distance Characteristic Of Scanning Vibrating Probe/1-Alkanethiol Self-Assembled Monolayer (SAM)/Au(111) Structure. Yutaka Majima<sup>1,2</sup> and Yuhsuke

Yasutake<sup>1</sup>; <sup>1</sup>Dept. of Physical Electronics, Tokyo Inst. of Tech., Tokyo, Japan; <sup>2</sup>PRESTO, Japan Science and Technology Corporation, Tokyo, Japan.

Nanomechanical single-electron devices such as the electron shuttle are of great interest not only for application in electronics but also for precision measurement and metrology. For the creation of nanomechanical single-electron devices, it is important to control the tunneling resistance ratio in double barrier tunneling junctions (DBTJs), because the polarity of electric charge depends on the tunneling resistance ratio. Recently, we have developed the simultaneous measuring technique for displacement current and tunneling current spectroscopy[1]. By using our technique, we have observed the displacement current staircase as well as tunneling current staircase in the nanomechanical double barrier tunneling junctions that consist of scanning vibrating probe/vacuum/colloidal Au dots/1-alkanedithiol self-assembled monolayer (SAM)/Au(111) substrate. From the staircases of displacement and tunneling currents, the electron shuttle motion of the single electrons have been clarified[2-3]. Here we report the measurements of the tunneling current-distance (I-d) characteristic of scanning vibrating probe/vacuum/1-alkanethiol SAM/Au(111) structure, and estimate the tunneling resistance of 1-alkanethiol SAM on Au(111) when the top of the scanning tunneling microscopy (STM) probe is just in contact with the end of the alkyl chains of 1-alkanethiol SAM. We also demonstrate the dependence of the set-point current of  $\operatorname{STM}$ images (const. height mode) of 1-alkanethiol SAM on Au(111), and discuss the thickness of vacuum layer between the probe top and the end of the alkyl chains. [1] Y. Majima, Y. Oyama and M. Iwamoto, Phys. Rev. B 62 (2000) 1971. [2] K. Nagano, A. Okuda, Y. Majima, Appl. Phys. Lett., 81, (2002) 544. [3] Y. Azuma, K. Nagano, Y. Majima, Ling Land Land Phys. 12 (2002) 2452 Majima, Jpn. J. Appl. Phys. 42, (2003) 2458.

## 9:45 AM A1.5

Carbon Nanotubes for Sensors. Werner Haenni<sup>1</sup>, Thomas Stoeckli<sup>2</sup>, Christian Hinderling<sup>1</sup>, Helmut Knapp<sup>2</sup> and <u>Henry Haefke</u><sup>1</sup>; <sup>1</sup>Nanotechnology and Life Sciences, CSEM S.A., Neuchatel,

Switzerland;  $^2$ Microrobotics, CSEM S.A., Alpnach, Switzerland.

In recent years carbon nanotubes (CNTs) are proposed as a new material with outstanding properties like diamond for cold electron emission and for the next generation of field effect transistors (FET). A new application emerged by integrating CNTs in micro- and nanoelectromechanical systems (MEMS and NEMS) as an active sensor material. CNTs are promising material, based on their exceptional thermal and electrical conductivity, for their stiffness and chemical inertness. CNTs can be used to functionalize the surface or filled out with convenient molecules as highly selective sensor for a wide range of gas sensor applications. Key difficulties in making such sensors are the integration of CNTs in microstructure devices at elevated temperature from the chemical vapor phase. This paper presents the development of two different types of sensors based on CNTs in one case perpendicularly grown in the other case laterally grown CNTs at pre-defined sites. Iron has been used as a catalyst for the growth of CNTs as well as Ni and alloys of Ni and Fe are used. With an appropriate heat treating the catalyst cluster are fixed to the supporting material which can be either an insulator like silicon oxide or silicon nitride or an electrically conducting material like doped silicon, titanium nitride, tungsten silicide or even other forms of carbon materials. Classical thermal treatment process as well as hot filament activated chemical vapor deposition processes has been used to deposit CNTs in the temperature range of 700 to 800° C Appropriate E-fields are used for conducting the growth direction of CNTs. Electron microscopy and Raman spectroscopy are employed to for identification of the CNTs. The perpendicularly grown CNTs have been applied on AFM tips for nanorobotics and structure measurements. The laterally grown CNTs have been implemented in microstructures to construct a FET-based sensor.

> SESSION A2: Alternative Fabrication Techniques I Chair: Mark McNie Monday Morning, December 1, 2003 Room 200 (Hynes)

10:30 AM <u>\*A2.1</u>
Poly-SiGe: A Superb Material for MEMS. Ann Witvrouw, MCP/SSM, IMEC, Leuven, Belgium.

Micro-electromechanical systems (MEMS) such as infrared detectors, accelerometers, gyroscopes... are increasingly used. Monolithic integration of MEMS with the driving, controlling and signal-processing electronics on the same CMOS substrate can improve performance, reduce size and also lower packaging costs. The easiest approach for monolithic integration is post-processing MEMS on top of the electronics, as this allows the use of a standard CMOS fabrication process [1]. However, post-processing limits the maximum fabrication temperature of MEMS in order to avoid any damage or degradation in the performance of the underlying electronics. Polycrystalline silicon (poly-Si) has been widely used for MEMS applications, but this material normally requires a high processing temperature (>  $800~^\circ C$ ) to achieve a low tensile stress and to activate dopants. Poly-SiGe seems to be an atractive alternative to poly-Si as it has similar properties, while, as will be shown in this presentation, the desired electrical and mechanical properties can be realized at a temperature suitable for post-processing MEMS on top of standard CMOS wafers with Al interconnects [2]. Moreover, poly-SiGe has a 5 times lower thermal conductivity compared to poly-Si, making it an interesting material for thermopiles, bolometers... [3] with or without CMOS integration. Three types of CVD (chemical vapor deposition) processes to deposit poly-SiGe with an ideal intrinsic stress (low tensile), stress gradient (low) and resistivity (as low as  $1 \text{m}\Omega \text{cm}$ ) for MEMS structural layers were developed: CVD at 2 Torr pressure, CVD at 40 Torr pressure or RPCVD (reduced pressure CVD) and PECVD (plasma enhanced CVD). The use of these technologies for processing bolometers, capping layers for wafer-level packaging of MEMS devices and MEMS devices above standard CMOS will be demonstrated. The multitude of possible applications show that poly-SiGe is indeed a superb MEMS material and that surface micromachining of poly-SiGe is a candidate to be one of the very few 'generic' MEMS technologies. [1] A. Witvrouw et al., Microsystems Technologies, Vol. 6 (5), p 192-199 (2000). [2] S. Sedky et al., IEEE Trans. El. Dev., 48 (2), p 377-385 (2001). [3] S. Sedky et al., IEEE Trans. El. Dev., 46 (4), p 675-682 (1999).

## 11:00 AM A2.2

Fabrication of Micro- and Nanoscale SiC Structures Using Selective Deposition Processes. Li Chen<sup>1,2</sup>, Xiao-an Fu<sup>2</sup>, Christian A. Zorman<sup>2</sup> and Mehran Mehregany<sup>2</sup>; <sup>1</sup>Materials Science, Case Western Reserve University, Cleveland, Ohio; <sup>2</sup>Electrical Engineering and Computer Science, Case Western Reserve University, Cleveland, Ohio.

An ensemble of outstanding electrical, mechanical and chemical properties makes SiC an attractive alternative to Si for micro- and nanoelectromechanical systems (MEMS and NEMS). Unlike Si, the chemical inertness of SiC makes it a challenging material to pattern and micromachine. This paper reports on the fabrication of microand nanoscale SiC structures using selective deposition techniques for patterning. The process relies on significant differences in the nucleation of SiC on SiO2 and polysilicon surfaces to create patterned SiC structures without the use of SiC RIE. A process to fabricate free standing, porous SiC membranes begins with the deposition and patterning of a SiO2/polysilicon/SiO2 multilayer on (100) Si substrates. A carbonization-based epitaxial process is used to deposit the SiC films, which results in the formation of a uniform coating on the exposed sidewalls of the polysilicon and a continuous, yet porous film on the exposed SiO2 surfaces. The structure is then released by dissolving the sacrificial SiO2 layer atop the polysilicon in HF. This results in the creation of a porous SiC shell that is mechanically and chemically stable. Fabrication of nanoscale SiC beam structures capitalizes on the same selective growth processes used in making the porous membranes. As with the porous membranes, the starting substrate consists of SiO2/polysilicon/SiO2 multilayer patterned into long, narrow beams. The SiC growth process is also the same except that the SiC film thickness is restricted less than 200 nm. Under these conditions, only a collection of discontinuous SiC grains form on the oxide surfaces. These grains, as well as the SiO2 film, are easily removed by HF lift-off with ultrasonic agitation. The polysilicon is then removed in an aqueous Si etchant (KOH), resulting in free standing SiC beams with submicron widths. The extended paper will detail each process as well as possible applications.

## 11:15 AM A2.3

Manostructure fabrication using electrosprayed polymeric nanofibers. David Alan Czaplewski<sup>1,3</sup>, Jun Kameoka<sup>1,3</sup>, Robert Mathers<sup>2</sup>, Geoffrey Coates<sup>2</sup> and Harold Gene Craighead<sup>1,3</sup>; <sup>1</sup>Applied Physics, Cornell University, Ithaca, New York; <sup>2</sup>Department of Chemistry and Chemical Biology, Cornell University, Ithaca, New York; <sup>3</sup>Nanobiotechnology Center, Cornell University, Ithaca, New York.

We have demonstrated electrospray deposition of oriented nanoscale fibers as templates for nanostructure fabrication. This non-lithographic approach can be used to create a range of nanostructures in hard materials without the need for electron beam or exotic forms of optical photolithography. We utilized electrospray to fabricate nanometer dimensional fibers on the surface of substrates such as silicon and glass. We created a microfabricated tip holding a liquid drop. By applying an electrostatic potential between the tip and a counter electrode, a liquid jet is extracted from the tip to the counter electrode. The solvent evaporates from the solution in flight leaving a cylindrical polymer fiber that gets deposited on the substrate. By scanning the source relative to the counter electrode, we can orient the fibers relative to pre-existing structures on the substrate, such as trenches or electrodes. We can deposit fibers, with radii ranging from 25nm to several micrometers. The fiber composition has included polymers such as poly(ethylene oxide), polynorborene, poly(methyl methacrylate) (PMMA) and polycarbonate. We have also produced fibers from conventional photoresist solutions. By utilizing polynorborene and polycarbonate fibers as sacrificial structures, we have fabricated fluidic channels with radii less than 50nm. We have also used fibers made from PMMA and conventional photoresists as etch masks to pattern underlying materials. We hope to combine this capability with conventional photolithography to produce a range of NEMS structures by undercutting a sacrificial layer.

## 11:30 AM A2.4

Optimization of Poly-SiGe Deposition Processes for Integrated MEMS. Blake Ching-Yu Lin, Tsu-Jae King and Roger T Howe; Department of Electrical Engineering and Computer Science, University of California at Berkeley, Berkeley, California.

Monolithic integration of MEMS devices with driving and controlling electronics is advantageous for improving performance and lowering cost. Polycrystalline silicon-germanium (poly-Si $_x$ Ge1 $_-x$ , where 0 < x < 1), which has mechanical and electrical properties similar to poly-Si, is promising as a structural material for post-CMOS integration of MEMS because it can be deposited at much lower temperatures than poly-Si [1]. The use of Ge as a sacrificial material eliminates the need for a hydrofluoric (HF) acid release etch, which can damage unprotected electronic circuitry. In this work, the optimal deposition conditions for structural poly-Si $_x$ Ge1 $_-x$  and sacrificial Ge layers are determined. In-situ-doped p-type poly-Si $_x$ Ge1 $_-x$  films deposited at temperatures lower than 450°C generally exhibit strain gradient on the order of  $10^{-3}$   $10^{-4}$   $\mu$ m $^{-1}$  when thickness is about 2  $\mu$ m. To reduce the strain gradient in as-deposited films, the use of two layers (deposited under slightly different conditions) to form a single composite structural film has been investigated. The aim of this bi-layer approach is to create a bending-down moment, by depositing

a layer with more compressive (or less tensile) stress on top of a layer with less compressive (or more tensile) stress. With appropriate layer thicknesses, the bending-down moment can cancel the bending-up moment generally seen in as-deposited films, lowering overall strain gradient to the order of  $10^{-5}~\mu\mathrm{m}^{-1}$  without any high-temperature annealing. When undoped Ge is used as sacrificial material, a significant difference in dopant (i.e. boron) concentrations between the structural poly-Si<sub>x</sub>Ge<sub>1-x</sub> layer and the sacrificial Ge layer result in enhanced Ge inter-diffusion, resulting in a rough structural layer surface after release etch. For a multi-structural-layer MEMS process, this problem is exacerbated by the thermal budget of subsequent film depositions. An investigation is currently underway to characterize the dependence of Ge inter-diffusion on Ge doping concentration and thermal annealing budget. [1] A. Franke, J. M. Heck, T.-J. King, and R. T. Howe, J. MEMS 12, 160 (2003)

## 11:45 AM A2.5

Thin-Film Electrostatic Actuators On Flexible Plastic Substrates. Joao Gaspar<sup>1,2</sup>, Virginia Chu<sup>1</sup> and Joao Conde<sup>1,2</sup>;

<sup>1</sup>INESC Microsistemas e Nanotecnologias, Lisbon, Lisbon, Portugal; <sup>2</sup>Materials Engineering Department, Instituto Superior Tecnico, Lisbon, Lisbon, Portugal.

Microelectronics processing on large area and flexible substrates is of great interest for applications requiring low-weight integrated circuits (ICs), low-cost substrates, mechanical flexibility, biocompatibility, and/or throw-away electronics. The low temperature processing of amorphous-silicon-based thin-films allows a wide variety of substrates to be used. In addition, the mechanical, optical and electronic properties of these thin-films can be controlled by varying the deposition conditions. The field of flexible electronics has already given rise to devices such as thin-film transistors (TFTs) and image sensors fabricated on flexible plastic substrates. In this work we demonstrate MEMS electrostatic actuators fabricated on flexible polyethylene terephthalate (PET) and polyimide (PI) substrates using surface micromachining and thin-film technology. The structures consist of microbridges with an underlying metal gate electrode. The structural layer material is  $n^+$ -doped hydrogenated amorphous silicon  $(n^+$ -a-Si:H) with low residual stress. All the processing steps do not exceed 110°C. Typical microbridge dimensions are 4-100 µm for the length (L), 4-20 mm for the width (w) and 0.1-0.8  $\mu$ m for the thickness (t). Aluminum (Al) is used as the sacrificial layer material. The air-gap height (d) is  $0.5 \mu m$ . The structures are electrostatically actuated by applying a low-frequency voltage  $(itV_G)$  between the gate and the bridge and the resulting movement is optically detected. The quasi-DC (low frequency) deflection is monitored with sub-nm precision and is studied as function of the structure dimensions and driving electrostatic amplitude. The deflection is proportional to , which is in agreement with a 1D electromechanical model. Presently, work is under way to study the resonance behavior of these microbridges. This paper demonstrates that it is possible to integrate thin-film MEMS sensors and actuators on flexible plastic substrates.

> SESSION A3: Micro and NanoFluidics Chair: Prasad Somuri Monday Afternoon, December 1, 2003 Room 200 (Hynes)

## 1:30 PM <u>A3.1</u>

Scanning Probe Characterization of Localized pH Changes on a Sapphire Surface in the Presence of an Applied Field.

Joseph W Bullard, Ryan J Kershner and Michael J Cima; Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Single crystal sapphire substrates were lithographically patterned with a system of parallel platinum electrodes, which were used to manipulate 1.58  $\mu m$  silica particles in-plane, in the presence of an aqueous solution. Observation of the motion of these particles revealed the adhesion of some of them to the sapphire surface near the platinum working electrode, even in the range of pH where the  $\zeta$ -potentials of silica and sapphire are of the same sign. This phenomenon suggests the existence of localized differences in pH, attributable to the presence of potential determining ions produced in the faradaic processes occurring at the electrodes during the electrophoretic manipulation of silica particles. Atomic force microscopy (AFM) was used to corroborate this hypothesis, measuring the forces between a silica particle and a sapphire substrate in the presence of an applied field. The resultant force-distance curves demonstrate a change in the interaction forces beteween particle and substrate as a function of distance from the electrode. Variations in this interaction correspond to localized differences in the  $\zeta$ -potential of the substrate, which, in turn, are related to localized differences in pH. Quantifying these spatial variations in pH as a function of time further yields information related the diffusion of these faradaically

produced potential determining ions across the substrate.

### 1:45 PM <u>A3.2</u>

Monolithic Three-Dimensional Integration of Micro-Fluidic Channels and Optical Waveguides. Yves Bellouard<sup>1</sup>, Young Seok Oh<sup>1</sup>, Ali Said<sup>2</sup>, Mark Dugan<sup>2</sup>, Tom Sosnowski<sup>2</sup> and Philippe Bado<sup>2</sup>; <sup>1</sup>Center for Automation Technologies, Rensselaer Polytechnic Institute, Troy, New York; <sup>2</sup>Translume, Ann Arbor, Michigan.

Miniaturized biological sensors (Bio-MEMS or "Biochip"), chemical sensors and screening systems often require micro-fluidic channel networks of various complexity to perform functions as diverse as particles sorting, chemicals mixing or parallel assays. The manufacturing process must prevent leaks while being sufficiently simple to be commercially viable. In many applications, one must also dispose of the components containing the fluidic channels after usage. In this paper, we propose a unique fluidic platform with three-dimensional micro-tunnels and optical waveguides manufactured in a single piece of glass. The fabrication process combines femtosecond laser machining and chemical etching. Optical-waveguides are made by locally modifying the index of refraction through nano-restructuring of the glass using a femtosecond laser. Since the material is only modified at the focal point, waveguides can be written within the volume. Thanks to this technology, waveguides can be coupled, recombined or shaped to form complex optical devices like Mach-Zehnder interferometers for instance, integrated in a single substrate. The same femtosecond laser is also used in combination with chemical etching to form fluidic channels and tunnels. Carefully chosen laser-processing parameters are utilized to introduce spatially localized anisotropy in the glass Once the glass is immersed into a low concentration hydrofluoric bath, the etching rate is several orders of magnitude faster in the direction where the glass was previously exposed to the laser beam. This integrated platform opens new opportunities in bio- and chemical sensing. The flexibility of the process offers substantial new design capabilities in particular for single channels probing and massively parallel processing and sensing. This simple two-step process achieves a unique three-dimensional monolithic integration of fluidic micro-channels and optical waveguides. It bypasses most packaging issues that plague numerous MEMS applications. This paper details this new manufacturing process and demonstrates its viability through a test bed that contains high aspects ratio micro-tunnels and optical waveguides used to probe locally a fluid flowing in a channel.

### 2:00 PM A3.3

Damage and Failure Mechanisms in High Pressure Silicon-Glass-Metal Microfluidic Connections. <u>Dong-Jin Shim</u>, Hong-wei Sun, V.T. Srikar and S. Mark Spearing; Aeronuatics and Astronautics, MIT, Cambridge, Massachusetts.

The application of microsystems in power generation and propulsion necessitates the development microfluidic connection technologies capable of operating at high temperatures (500 C) and pressures (15 MPa). The damage and failure mechanisms in a design consisting of Kovar metal tubes attached to silicon microrocket devices using borosilicate glass seals has been analyzed. A key concern in such joints is the occurrence of cracks in silicon and glass due to residual stresses caused by large thermal excursion (>1000 C) during processing and the dissimilar coefficients of thermal expansion of the constituent materials. Such thermally-induced cracks may reduce the load carrying capability of the joint and lead to premature failure Connections with different glass compositions and bond configurations were fabricated and cross-sectioned to identify conditions that minimize thermally-induced cracks. Axial tension and pressure tests confirmed the increase in strength with reduction in pre-crack density. The similarities and differences in the damage and failure characteristics between the two loading conditions have been studied. Finite element models were used to analyze the residual stresses due to the manufacturing process and excellent correlation was found between the location of thermally-induced cracks and location of maximum principal stress for the different materials and geometries considered. A fracture mechanics approach was used to predict the failure of the bonded joints. Good agreement was found between the predicted failure loads for both the pressure and the axial tension test cases. The effects of other processing parameters such as voids in the glass, wetting of glass to Kovar and silicon, interactions between neighboring joints, and manufacturing-related variability will be discussed. This work clearly demonstrates that the nucleation and growth of thermal-induced cracks controls the strength of such joints and suggests guidelines for designing reliable microfluidic packages.

## 2:15 PM <u>A3.4</u>

Nanostructured Materials For Microfluidic Sensing Application. Nancy N Kariuki, Laura Moussa, Li Han, Jin Luo and Chuan-Jian Zhong; Chemistry, SUNY-Binghamton, Binghamton, New York. Microfluidc systems have great potentials in chemical and biomedical applications. One of the key challenges is to develop highly responsive sensing materials with large surface area to volume ratio for chemical/biological recognition. This presentation describes the fabrication of novel nanostructured materials on interdigited microelectrode (IME) arrays as sensors for integration into microfluidic devices. The goal is to produce functional devices at extremes of miniaturization. In one approach, gold nanoparticles are assembled as thin film coatings on the IME using molecular wires that are embedded in a microfluidic channel. Different materials have been used for the fabrication of the device. The IME were created on glass wafers using lithographic techniques. The microchannels were created using PDMS-based soft-lithography. Thin films composed of nanostructured carboxylic acid framework were found to provide enhanced sensitivity in chemical recognition. Implications of the findings to the design of the microfluidic devices for chemical/biological sensing applications will also be discussed.

## 2:30 PM A3.5

Characterization of 0.5 MHz Silicon-Based Ultrasonic Nozzles

Using Multiple Fourier Horns. Shirley C. Tsai<sup>1,2</sup>, Eugene YL Song<sup>1</sup>, W J Chen<sup>1</sup>, T K Tseng<sup>1</sup>, Y F Chou<sup>3</sup>, J H Yang<sup>4</sup> and Chen S Tsai<sup>1,5</sup>; Institute for Applied Science and Engineering Research, Academia Sinica, Taipei, Taiwan; <sup>2</sup>Department of Chemical California; Department of Chemical Engineering, California State University, Long Beach, Long Beach, California; Department of Mechanical Engineering, National Taiwan University, Taipei, Taiwan; Department of Mechanical Engineering, Chang Gung University, Taipei, Taiwan; Department of Electrical Engineering and Computer Science, University of California, Irvine, Irvine, California.

The production of ceramic nanoparticles for use in a wide variety of applications including photo catalysis and solid oxide fuels can be accomplished by spray pyrolysis of precursor drops. Current spray pyrolysis technology relies upon high temperature (>1000°C) or vacuum pressure (60mbar) to pyrolize 10-50um-diameter precursor drops into nanoparticles. We have developed a novel atomization technique using silicon-based ultrasonic nozzles, which can more efficiently produce precursor drops <10um in diameter. Such precursor drops can be pyrolized at much lower temperatures and atmosphoreic pressure, allowing efficient and inexpensive production of nanoparticles. Silicon-based ultrasonic nozzles for atomization have a number of advantages over conventional metal-based bulk-type ultrasonic nozzles. These advantages enable silicon-based ultrasonic nozzles to overcome the 120kHz-frequency limitations of the metal-based counterparts, and to produce uniform drops smaller than 10um in diameter. In this paper, following a brief description of the design basis and simulation results, characterization by impedance analysis and longitudianl vibration measurement of MEMS-fabricated  $0.5 \mathrm{MHz}$  Si-based ultrasonic nozzles are presented. The nozzle utilizes multiple Fourier horns in cascade to produce both large amplitude gain and adequate cross sectional area at the nozzle tip. Results of 3-D simulation using finite element method, the commercial ANSYS program, show existence of one pure longitudinal mode at the resonant frequency of 493±4kHz. Both impedance analysis and measurement of longitudinal vibration at the nozzle tip verified this simulation result. Furthermore, the measured longitudinal vibration amplitude at the nozzle tip of the 5-horn nozzle is much larger than that of the single horn nozzle, in excellent agreement with the theoretical values of  $2\hat{n}$ , where n is the number of Fourier horns. The large gain in vibration amplitude enables drastic reduction in the electric drive power requirement and frequency of transducer failure in ultrasonic atomization.

## 2:45 PM A3.6

A Scheme of Micromanipulation using a Liquid Bridge.

<u>Kenichi J. Obata</u><sup>1</sup>, Shigeki Saito<sup>1,2</sup> and Kunio Takahashi<sup>1</sup>; <sup>1</sup>Dept. of
International Develop Engineering, Tokyo Institute of Technology, Meguro-ku, Tokyo, Japan; <sup>2</sup>Department of Material Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

This paper presents a scheme of micromanipulation with a liquid bridge and an analysis of the capillary forces involved. The following procedure considered in this article: (a) PICK UP: a probe, with liquid in the tip, approaches the target object. (b) A liquid bridge forms between the object and the tip of the probe. (c) The object is picked up by means of the capillary force of the liquid bridge. (d) TRANSPORT. The probe ascends, moves to the target point, and descends towards a substrate. (e) PLACEMENT: At a given height, a second liquid bridge made from a drop previously applied at the target point on the substrate, forms between the object and the substrate. (f) The probe ascends and the probe-object bridge collapses. The collapse can be predicted through the stability analysis of the bridge and its condition can be controlled by the regulation of the liquid volume. The liquid volumes required for the manipulation, in the first and second liquid bridge, are calculated in this paper.

SESSION A4: Applied Micro and Nanotechnology I Chair: Marc Madou Monday Afternoon, December 1, 2003Room 200 (Hynes)

## 3:30 PM \*A4.1

Carbon Nanotube Field Emitters for Electron Microscopes. Niels de Jonge, philips research, eindhoven, Netherlands

A research project at Philips Research Eindhoven is aimed at developing a new type of electron source for electron microscopes to improve their resolution. Electron sources have been made by mounting individual carbon nanotubes (multi-walled type) on tungsten tips in a scanning electron microscope (SEM) equipped with a nano-manipulator. The electron emission of the new sources has been studied in an ultra-high-vacuum system containing specialized equipment to allow a full characterization of the emission process. These sources provide an extremely high brightness: 3x109  $A/(Sr^*m\hat{2}^*V)$ ; this is an order of magnitude larger than that of state-of-the-art commercial sources, i.e. Schottky emitters and cold-field-emission guns. The energy spread is more than two times smaller than that of Schottky emitters at a similar current stability, and is the same as that of cold-field-emission guns, which have a lower current stability.

## 4:00 PM <u>A4.2</u>

Characterization of Electrostatic Micromembrane Actuator Performance Using A Mass Probing Method. Xingtao Wu

Jeremy Hui<sup>1</sup>, Pat Kayatta<sup>3</sup>, Dedeian Kenneth<sup>3</sup>, Miriam Young<sup>1</sup> and Cardinal Warde<sup>2,1</sup>; <sup>1</sup>Optron Systems, Inc., Bedford, Massachusetts; <sup>2</sup>Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology, Cambridge, Massachusetts; Integrated Micromachines, Inc., Monrovia, California

We describe a mass probing technique for characterizing the electro-mechanical behavior of micromembrane array actuators under electrostatic loads. The technique utilizes an array of micromembrane actuators linked by one or more arrays of center-positioned posts to a top movable rigid mass plate, thus generating 1D motion of the top mass owing to motion transfer from the deformed membranes. With a large top mirror bonded to membrane actuator array, the observation of the mirror motion due to actuation from membrane actuator arrays was used to characterize the performance parameters of the micromembrane actuators beneath it. The latter is usually difficult to observe due to its small size (<100 um), especially during high-speed actuation (e.g. >10 kHz). The micromembrane actuator arrays are fabricated using a stacked architecture similar to MUMPs, in which the poly-Si structure material is replaced by metal-over-polymer composite, and final release is accomplished by a dry etch of a sacrificial silicon layer. Performance characteristics such as natural frequency, membrane stiffness, rise/fall time, damping ratio and phase delay of the actuator-mass systems were obtained. The mass probing technique is implemented by the hybrid integration of a thick (100 650-um), heavy, and rigid mass plate (with a polished surface as a reflective mirror) onto the membrane array in which individual membrane actuators have a center interface post to act as the mechanical linkage. The loaded heavy mass also behaves as a large-aperture (up to 12-mm at present) rigid optical mirror. This also simplifies the measurement of the actuated membrane motion, which was accomplished by using a standard Michelson interferometer setup. The large number (>2k elements) of membrane actuators that drive the mass load allows an averaging out of errors, leading to a statistically accurate measurement of the 1st natural frequency and spring stiffness. In real experiments we also compared Michelson interference results with diffractive measurements. Both static and dynamic properties of membrane actuators were measured. A preliminary test of a 40-um pitch membrane array shows a 1st natural frequency of 2.1 MHz of the membrane actuator. This was scaled down to 42 kHz by a 2.7mmx2.7mm 200-um thick mass load weighting 3.3-mg. Ignoring the small membrane mass, the lumped spring stiffness is estimated to be 50.2uN/um per actuator (4556 40-um actuators embedded beneath the probing mass). Thus, motion transfer principle from micro size actuators to macro size components was demonstrated.

## 4:15 PM <u>A4.3</u>

Development and Application of Intelligent Polymer Networks as Recognition Elements for Novel Microdevices. James Zachary Hilt<sup>1</sup>, Mark E. Byrne<sup>1</sup>, Rashid Bashir<sup>2</sup> and Nicholas

 $\overline{\text{A. Peppas}^1}$ ;  $^1\text{Department}$  of Chemical Engineering, University of Texas, Austin, Texas;  $^2\text{School}$  of Electrical and Computer Engineering, Purdue University, West Lafayette, Indiana.

Biological compounds, such as enzymes or antibodies, are typically applied as recognition elements in biosensor platforms. Although these biological compounds usually exhibit high affinity and specificity, their fragile nature limits the processing and operational conditions

for the corresponding sensor devices. The objective of this research is to develop and apply novel intelligent polymer networks as recognition elements. These synthetic networks are advantageous because they can be designed to mimic biological recognition pathways and at the same time exhibit other abiotic properties that are more favorable for microsensing applications. In our laboratory, methods have been developed to integrate these polymer networks with silicon substrates, since the majority of transducers for microsensor platforms are silicon based. In particular, a micro-reactor photolithography technique was developed, which enabled precise spatial and thickness control over the resulting polymer patterns. An organosilane agent was utilized to gain covalent adhesion between the polymer network and the silicon surface. Multiple intelligent polymer networks, which have been developed in our laboratory, have been applied in silicon based devices using these methods. For example, anionic hydrogels were precisely micropatterned onto silicon microcantilevers, for application as MEMS and bioMEMS sensors. Specifically, a crosslinked poly(methacrylic acid) network containing significant amounts of poly(ethylene glycol) dimethacrylate was utilized as a sensing element responsive to environmental pH. The bending response of patterned cantilevers with a change in pH was observed, and an ultrahigh deflection sensitivity of approximately  $1 \text{nm}/5 \text{x} 10^{-5} \Delta \text{pH}$  was demonstrated. This versatile biosensor platform can be extended to other intelligent polymer networks for detection of additional biologically significant analytes In another study, a biomimetic recognitive hydrogel that selectively recognizes D-glucose among similar molecules via non-covalent complexation was micropatterned onto silicon substrates and characterized by single and competitive fluorescent and confocal microscopy studies, SEM, and profilometry. Novel copolymer networks containing poly(ethylene glycol) dimethacrylate and functional monomers such as acrylic acid, methacrylic acid, and acrylamide were synthesized in polar, aprotic solvent (dimethyl sulfoxide). It was demonstrated that these recognitive networks were effectively micropatterned in fine dimensions and were specific for the target molecule. These novel materials and fabrication techniques that have been demonstrated are enabling technologies for innovative micro-/nanoscale biosensors and diagnostic devices. This research was funded by the National Institutes of Health.

4:30 PM A4.4

Polymer Microvalve Based on Anisotropic Expansion of Polypyrrole. Yevgeny Berdichevsky and Y.-H Lo; Electrical and Computer Engineering Department, University of California, San Diego, La Jolla, California.

A microactuator was fabricated from conductive polymer polypyrrole utilizing microfabrication techniques. The actuator exhibits high force density as well as large strain typical of anisotropic expansion of polypyrrole perpendicular to the substrate plane. The vertical anisotropic strain reaches as much as 25% as opposed to horizontal strains of 2-3% utilized in polypyrrole bilayer actuators previously. Pyrrole was electropolymerized on several substrates including electroplated gold and oxidized titanium in order to explore methods of improving the adhesion and increase the lifetime of devices based on this recently reported mode of actuation. A microfluidic valve was developed on the basis of large vertical expansion of polypyrrole micropillars. Soft lithography techniques and elastomer material (PDMS) were utilized to fabricate the microchannels and valve chamber. The use of PDMS enabled good valve seal and allowed encapsulation of the active part of the device.

## 4:45 PM A4.5

Transient Absorption at Telecommunications Wavelengths in Conjugated Polymers under Intense Picosecond Excitation at 800 nm. Sergei Musikhin, Vlad Sukhovatkin, Luda Bakueva and Edward H Sargent; ECE, UofT, Toronto, Ontario, Canada.

Conjugated polymers hold promise in electroluminescence, photovoltaics, and nonlinear optics and their optical properties in the visible spectral region have been already investigated quite well. Additionally, they may be combined with quantum dots to provide absorption, emission, modulation, and resonant nonlinearity at communications wavelengths, i.e. 1300-1600 nm [1]. These applications require thorough experimental analysis of the optical properties of relevant polymers in the near infrared region. We investigated MEH-PPV, PPV and CN-PPP via transient absorption using a pump (1 ps, 800 nm, 1 GW/cm2) and delayed probe (1 ps, 1150-1600, 1 MW/cm2) configuration. We observed two-photon absorption accompanied by photoluminescence in MEH-PPV polymer and not in pure PPV and CN-PPP polymers. The lifetime of the excited state responsible for photoinduced absorption is approximately 20 ps. The time-dependence of the observed transient absorption allows us to eliminate pump+probe [2] absorption and instead attribute the photoinduced absorption to two-photon absorption of pump photons followed by transfer to the excited state probed, as in [3]. At a high pump intensity the absorption is lower at 1500 nm than at 1350 nm, consistent with the energy level structure of the polymer as in [3]. [1]

L. Bakueva, S. Musikhin, M. A. Hines, T.-W. Chang, M. Tzolov, G. D. Scholes and E. H. Sargent. Appl. Phys. Lett. 82 2895 (2003) [2] U. Lemmer, R. Fisher, J. Feldmann, R. F. Mahrt, J. Yang, A. Greiner, H. Bassler, E. O. Gobel. Chem. Phys. Lett. 203, 28 (1993) [3] G. J. Lee, K. Kim, J.-I. Jin. Opt. Comm. 203, 151 (2002)

SESSION A5: Poster Session
Chairs: Arturo Ayon, David LaVan, Marc Madou, Mark
McNie and Prasad Somuri
Monday Evening, December 1, 2003
8:00 PM
Exhibition Hall D (Hynes)

### A5.1

Self-organized Criticality in Nanotribology. Micha Adler<sup>1</sup>, John Ferrante<sup>2</sup>, Alan Schilowitz<sup>3</sup>, Dalia Yablon<sup>3</sup> and Fredy R. Zypman<sup>1</sup>;

<sup>1</sup>Physics, Yeshiva University, New York, New York; <sup>2</sup>NASA-Glenn Research Center, Cleveland, Ohio; <sup>3</sup>Corporate Strategic Research, ExxonMobil Research and Engineering Co, Annandale, New Jersey.

We will present experimental results on dry friction, which are consistent with the hypothesis that the stick-slip mechanism for energy release is described by self-organized criticality. The data, obtained with a nano-tribometer (an Atomic Force Microscope set to measure lateral forces) examines the variation of the friction force as a function of time, or sliding distance. The materials studied were nominally flat surfaces of aluminum, M50 steel, gold and silver. An analysis of the data shows that the probability distribution of slip sizes follows a power law. In addition, the frequency power spectrum follows a 1/fa pattern with a closer but larger than unity. We performed a careful analysis of all the properties, in addition to the two just mentioned- that are required to guarantee the presence of self-organized criticality. Our data strongly supports the existence of self-organized criticality for nano-stick-slip in dry sliding friction. We also correlate the parameters of SOC with the metals under study and from the results propose a new method of materials characterization based on the slopes of SOC and power spectra.

### A5.2

Stiction Measurements Made with an Atomic Force Microscope on Test Structures Mounted with Various Die-Attach Materials. Erik J. Thoreson<sup>1</sup>, Jack Martin<sup>2</sup> and Nancy A. Burnham<sup>1</sup>; <sup>1</sup>Department of Physics, Worcester Polytechnic Institute, Worcester, Massachusetts; <sup>2</sup>Micromachined Products Division, Analog Devices Incorporated, Cambridge, Massachusetts.

An atomic force microscope (AFM) was used to determine the stiction between silicon oxide tips and silicon oxide substrates coated with a few angstroms of phenylsiloxane. The substrates were mounted in their usual packaging with three different types of die-attach materials, which were silicone, polyimide silicone, and silver glass. There was also a control group in which the substrates were not attached. The packages were opened and an AFM determined the adhesive force between the AFM tip and the substrate in force spectroscopy mode. A preliminary data set showed that the adhesive force normalized to the tip radius was respectively twice and four times as big for the polyimide silicone and silver glass as for the control group and silicone, the latter two being close in value. The percent variations in the measurements were 70% to 80% percent for the control group and silicone, 150% for polyimide silicone, and 25%for silver glass. Further work will verify these initial results and also study the dependence of adhesive force upon the tip radius.

## A5.3

Optimization of Mesoporous Silicon Microcavities for Proteomic-based Pathogen Sensors. Lisa A. DeLouise<sup>1</sup>, Scott Horner<sup>2</sup> and Ben Miller<sup>1</sup>; <sup>1</sup>Dermatology, Center for Future Health University of Rochester Medical School, Rochester, New York; <sup>2</sup>BioPhysics, University of Rochester Medical School, Rochester, New York.

Porous silicon (PSi) is a unique photonic material that can be fabricated into a variety of optical devices for direct biosensor applications. This project leverages the photoluminescence (PL) and highly reflecting properties of mesoporous PSi microcavities to develop proteomic-based optical sensors for detection of pathogenic organisms. The PSi microcavity is a multi-layer Fabry-Perot resonant device comprised of a photoluminescent active layer sandwiched between two highly reflective dielectric Bragg mirrors. The mirrors create a stop-band that prevents photons, generated within the active layer, from escaping the device except for those at the wavelengths coincident with the Fabry-Perot resonance's. The optical properties of the microcavity and its utility as a biosensor depend on the characteristics of the nanoporous regions within the 3D microstructure of the device. The microstructure, in addition to defining the base optical response, also serves as a scaffold to which

biomolecular probes are immobilized. Addition of the probes and subsequent binding of target molecules alters the porosity and consequently the index of refraction of the microcavity causing a shift in the optical response of the device. This paper describes key design considerations in optimizing the PSi microcavity for proteomic sensor applications. Critical parameters include the porosity, pore size and the orientation and index of refraction of the multi-layers within the microcavity stack. Pore size must be optimized relative to molecular weight and shape of the proteins. Theoretical modeling studies have been conducted to characterize the sensitivity of this device to porosity changes. Experimentally we have established how porosity can be altered by changing the electrochemical etch conditions. The impact of post-etch treatments, such as KOH treatment and thermal oxidation, on pore size, porosity and the width of the stop-band have been studied. The device must be designed such that the width of the stop-band is positioned properly with respect to the broad photoluminescence envelope. In addition to proper design of the device microstructure, for a biosensor to function reliably and accurately, strategic choices must be made in selecting the probe immobilization chemistry. An introduction to these issues will be addressed. The enterohemorrhagic stain of E. coli bacteria O157:H7 serves as our model system for sensor development. The 7KDa translocated intimin receptor (Tir) protein serves as the probe. A 35KDa adhesin protein, Intimin, isolated from the bacterial outer membrane serves as the target. The ability to detect intact organisms and whole cell lysates will be addressed.

Sub-500 nm Strutured Surfaces for Anti-reflective Applications. Thomas Sawitowski, Norbert Beyer, Sven Wagener and Schulz Frank; AlCove Surfaces GmbH, Gladbeck, NRW, Germany.

In modern display and solar energy technology there is a great demand for a method for simple and easy sub-500 nm surface structuring to improve the transmittance of electromagnetic waves. Currently mainly plasma coatings are applied to improve the spectral transmission which is a very cost ineffective method. An alternative to PVD processes is the AlCoStruct(TM) technology which allows stochastic struturing of for example PMMA, PC or Glass either in injection molding processes or by roller imprinting directly into the substrate or into an addional coating. By this process pillars ranging from 20 to 500 nm in diameter if desired with aspect ratios of up to 10 can easily be made. A PMMA plate (50 mm diameter, 2 mm thickness) made by injection molding with a 180 nm surface structure for example shows a transmission in the visible light region of up to 98 % which is an improvement of almost 5-6 % compared to the control. Beside display and solar energy application we can imagine application in MEMS technology to improve the incoupling of light or even to enhance the efficiency of certain MEMS devices which than become a real NEMS / MEMS combination device.

Thermal Effects in Plasma Treatment of patterned PDMS for Bonding Stacked Channels. Jin Zou and Peter Wong; Mechanical Engineering, TUFTS, Medford, Massachusetts.

This paper will present the results of a study to identify the critical thermal effects in plasma treatment of bio-compatible Polydimethylsiloxane (PDMS) for use as a bonding method for BIO-MEMS packaging. PDMS has been used to create irreversible seals to itself as well as strong seals with glass, silicon, and silicon nitride. There is potential to bond and seal several channels to create 3D systems for microfluidic and reactor applications (at low temperatures <200°C). Moreover, hybrid bonding approaches with intermediate metal layer bonding methods can be used to achieve system level packaging. PDMS bonding is guaranteed by applying plasma stripping (ashing) to all surfaces. This process activates the surface (produces hydroxyl groups) for several minutes to allow bonding. Traditionally, asher systems operate above 500°C; however, the investigators have had good results at low temperatures (under 150°C). This paper presents the results of a series of parametric studies for the effect of processing temperature and gas components on bond quality and quantified by measuring the work of adhesion between layers.

## A5.6

Statistical Thermodynamics of Two-Dimensional Fluids of Solid Ceramic Nanoparticles. Liudmila A Pozhar and John Maguire; MLBP, Air Force Research Laboratory, Wright-Patterson Air Force Base, Ohio.

Thin films of particulate fluids are widely used in processing of novel composite materials and other technological processes. When the film width is much smaller than the constitutive particles' dimensions the film can be treated as a two-dimensional system. This presentation discusses statistical mechanics and equilibrium molecular dynamics (EMD) simulations of equilibrium and transport properties of such

two-dimensional systems composed of solid ceramic nanoparticles. The statistical data (the density and pair correlation functions) have been obtained by a conceptually novel simulation procedure proposed recently by Maguire and co-workers. These data have been further used in simplified formulae derived from fundamental Pozhar-Gubbins expressions for the transport coefficients to calculate the diffusion and viscosity coefficients of the studied fluids. The virial expression for the surface tension was obtained by theoretical means and used to analyze equilibrium thermodynamics of the system.

## A5.7

Fabrication, Characterization, and Application of Nanowell Arrays. Myungchan Kang, Shufang Yu, Naichao Li and Charles R. Martin; Chemistry, U. of Florida, Gainesville, Florida.

Highly ordered nanowells are fabricated by etching substrates such as glass or glass /metal through ordered alumina membrane masks. The depth of the nanowells is controlled by changing the etching time. Inside and outside of the nanowells are modified with different chemistry. Modification of nanowells enables specific binding of nanoparticles to inside nanowells. So far, chemically modified nanoscale-silica particles have been selectively attached to modified nanowells. Combination of porous alumina and other porous pattern masks can generate different patterns. Atomic force microscopy (AFM), field emission scanning electron microscopy (FESEM), spectroscopy, and electrochemistry method will be used to characterize the nanopatterns. These arrays of the nanowells will be explored for several applications such as nanoassay for biomaterials and nanoelectrode arrays.

Interface Profiles Near Three-phase Contact Lines in Electric Fields. Juergen Buehrle, Stephan Herminghaus and Frieder Mugele; Applied Physics, University Ulm, Ulm, Germany.

Long-range electrostatic fields deform the surface profile of a conductive liquid in the vicinity of the contact line. We have investigated the equilibrium profiles by balancing electrostatic and capillary forces locally at the liquid vapor interface. Numerical results suggest that the contact angle at the contact line is equal to Young's angle. Simultaneously, the local curvature displays a weak algebraic divergence. Furthermore, we present an asymptotic analytical model, which confirms these results and elucidates the scaling behavior of the profile close to the contact line.

Cell Based Microelectrode Array Biosensors.

Cengiz Sinan Ozkan<sup>1</sup>, Mo Yang<sup>1</sup>, Xuan Zhang<sup>1</sup>, Shalini Prasad<sup>2</sup>, Mihrimah Ozkan<sup>2,3</sup> and Andre Morgan<sup>1,2</sup>; <sup>1</sup>Mechanical Engineering, University of California at Riverside, Riverside, California; <sup>2</sup>Electrical Engineering, University of California at Riverside, Riverside, California; <sup>3</sup>Chemical and Environmental Engineering, University of California at Riverside, Riverside, California.

Extracellular potential is an important parameter which indicates the electrical activity of live cells. Membrane excitability in osteoblasts plays a key role in modulating the electrical activity in the presence of chemical agents. The complexity of cell signal makes interpretation of the cellular response to a chemical agent very difficult. By analyzing shifts in the signal power spectrum, it is possible to determine a frequency spectrum also known as Signature Pattern Vectors ( $\overline{\mathrm{SPV}}$ ) specific to a chemical. It is also essential to characterize single cell sensitivity and response time for specific chemical agents for developing detect-to-warn biosensors. We used a 4x4 multiple Pt microelectrode array to spatially position single osteoblast cells, by using a gradient AC field. Fast Fourier Transformation (FFT) and Wavelet Transformation (WT) analyses were used to extract information pertaining to the frequency of firing from the extracellular potential.

Ellipsometry Measurement Accuracy of Gate Oxides Under Polysilicon. Gary Jiang, Daewon Kwon, Donald Pelcher, Jana Clerico and George Collins; Rudolph Technologies, Inc, Flanders, New

Precisely controlling the thickness of ultrathin SiO2 gate dielectric films is critical for high yield advanced generation semiconductor manufacturing. Advanced methods for producing ultrathin gates deposit both the gate dielectric and the polysilicon gate electrode in a single cluster tool. This avoids the opportunity for absorption of molecular airborne contamination between the two layers, but necessitates measuring gate oxide thickness under polysilicon. Because of the variation in polysilicon grain size, amorphous silicon content, and the roughness, it is very difficult to model the optical feedback in the visible spectral range to resolve ultrathin (10-20A) gate oxide with sufficient accuracy and repeatability to allow process control.

Even if the gate dielectric and poly-Si electrode are produced in separate tools, the gate dielectric metrology difficulties encountered in accurately and repeatably measuring gate dielectric thickness in atmosphere (which contains airborne molecular contaminants) make it advantageous to be able to measure the gate dielectric under poly-Si. This would allow the finished electrode/gate stack to be measured at this critical step before committing the wafer to further processing This paper studies the optical behavior of the poly-Si on gate filmstack from 190 nm to 900 nm, and the physical properties of the poly-Si layer and the roughness layer. A more accurate modeling method is proposed to characterize the poly and roughness layer using effective medium approximation (EMA) models. With the new model, both a spectroscopic ellipsometer and a multi-angle focus beam ellipsometer were employed to measure wafers with different poly thickness and different gate oxide thickness. TEM was used to characterize the film thickness while roughness was determined using AFM. Good correlation was obtained among TEM, AFM and ellipsometry results. Excellent repeatability (0.04A 1 sigma on 15A gate oxide for 10 days) across wafer uniformity (0.2A 1 sigma for 49 point map) were also achieved when measuring gate dielectric films under the poly-Si with the multi-angle focus beam ellipsometer.

### A5.11

Selective Positioning and Density Control of Nanotubes within a Polymer Thin Film. Emer Lahiff<sup>1</sup>, Chang Y Ryu<sup>2</sup>, Seamus Curran<sup>3</sup>, Andrew I Minett<sup>1</sup>, Werner J Blau<sup>1</sup> and Pulickel M Ajayan<sup>4</sup>; <sup>1</sup>Physics Department, Trinity College Dublin, Dublin, Ireland; <sup>2</sup>Chemistry Department, Rensellaer Polytechnic Institute, Troy, New York; <sup>3</sup>Nanotechnology Center, Rensellaer Polytechnic Institute, Troy, New York; <sup>4</sup>Materials and Engineering Department, Rensellaer Polytechnic Institute, Troy, New York.

It is well known that nanotubes can be used to enhance the mechanical, electrical and optical properties of a polymer matrix. Here we introduce a completely new and innovative method of producing polymer/nanotube composites where the density and position of nanotubes within the composite can be controlled. Carbon nanotubes are grown from organo-metallic micro-patterns. These periodic nanotube arrays can then be incorporated into a polymer matrix by spin-coating a curable polymer film on the as-grown tubes. The location and density of conducting channels within the polymer can be easily controlled by soft lithography patterning. This method ensures an even dispersion of nanotubes within a polymer matrix and prevents the tubes aggregating together to form bundles within the composite. This controlled method of producing free-standing polymer/nanotube composite films represents a more efficient method of combining the two materials for potential flexible electronic applications in an inexpensive and scalable manner.

## A5.12

Fabrication of Micro-Contact Probe by Using Mo-Cr Spring with Au Plating Bump. Chinami Kaneshiro and Kohji Hohkawa; Electrical & Electronic Engineering, Kanagawa Institute of Technology, Atsugi-shi, Kanagawa, Japan.

Electrode-pad densities on integrated circuit chips are beginning to be high. In wafer test using micro probe, it is necessary to establish a fine alignment between probe array and IC's electrode pads. Also, any damages to the wafer surface are caused by mechanical probing or measurement. The key issues of wafer probe on IC test are as follows ; 1) an impedance mismatching, 2) a mechanical damage of probe due to metal fatigue during probing cycle, and 3) a high frequency noise due to an isolation. Therefore, in order to respond such requirements, we propose a novel contact probe consisted of spring finger for prevent from mechanical damage in probing cycle. In this paper, we present results of a basic study on fabrication technology of micro-spring contact probe. We fabricate micro spring probe by using MEMS technology. In this approach, a metal cantilever of micro fingers is formed on Si substrate with SiO2 film by a standard lift-off process. To prevent from mechanical damage, the material of contact probe with fine pattern is selected hard and elastic metals. We deposited Mo-Cr as a spring metal on Si substrate. After metal deposition, we release tip of fingers by sacrifice etching SiO2 film. We examine the metal fatigue of micro fingers with respect to the fabrication condition of contact probe. The key factors of spring finger are to maintain its spring stress during contact cycle test, and to reduce mechanical damage. The condition of depositing metals is a key parameter for obtaining a hard and an elastic metal finger. We will show the experimental results of fabrication technology of micro contact probe array.

## A5.13

Microwave Syntheis of Molecular Sieves and Capillary Crystal Growth by Geometric Confinement. Steven B Ogunwumi<sup>1</sup>, John F Wight<sup>2</sup> and James C Fajardo<sup>3</sup>; <sup>1</sup>Crystalline Materials Reseach, Corning Incorporated, Painted Post, New York; <sup>2</sup>Inorganic Processing, Corning Incorporated, Painted Post, New York; <sup>3</sup>Surface

and Interfaces, Corning Incorporated, Painted Post, New York.

Microwave heating is an alternate means for material processing. The technique is promising for the accelerated synthesis of new materials. The technique is used successfully to synthesize Pollucite. Microwave synthesis of molecular sieves is combined with capillary confinement as a novel synthetic method. It is successfully demonstrated in the preparation of small ZSM-5 crystals by microwave hydrothermal synthesis within the 1-1.7  $\mu m$  channels of a capillary bundle. The geometric and spatial confinement offered by the capillary is expected to define and control the resulting crystal size distribution and orientation. MCM-41, a unidimensional mesoporous molecular sieve is investigated as a candidate for capillary confinement. The large porous channels of MCM-41(>35 Å) is attractive as a host for promoting inclusions of guest molecules or templates. The successful confinement of MCM-41 represents an important step towards the alignment of molecular sieves in a defined orientation within thin capillaries bundles (less than 5  $\mu$ m channels). Ultimately, such an approach may be useful for the fabrication of new molecular sieve devices.

#### A5.14

Micromechanical Characterization of GaSb by Microbeam Deflecion and Using Nanoprobe and Finite Element Analysis.

Maria Ospina<sup>1</sup>, S Vangala<sup>2</sup>, D Yang<sup>3</sup>, C Sung<sup>1</sup> and W. D Goodhue<sup>2</sup>;

Center for Advanced Materials, University of Massachusetts, Lowell, Massachusetts; <sup>2</sup>Photonics Center, Dept. of Physics and Applied Physics, University of Massachusetts; bowell, Massachusetts;

Hysitron, Inc., Minneapolis, Minnesota.

The commercial development of low-power electronics and electro-optics based on the antimonides in the coming years will demand a better understanding of the mechanical properties of ternary and quaternary thin-film alloys fabricated from the InGaAlAsSbP material system. Of particular importance is the determination of Young's modulus of the materials. In this paper, we describe a technique for studying the mechanical behavior of such thin films by microbeam deflection and then demonstrate the technique using GaSb as the model. A test structure consisting of an array of gallium antimonide microbeams was fabricated with lengths ranging from 80 to 500  $\mu$ m long using photolithography and angled bromine ion-beam-assisted etching techniques. The microbeams were deflected using a calibrated nanoprobe thereby generating load-displacement curves. Young's modulus of elasticity was then extracted from the data using beam bending theory and a finite element simulation of the structures under load. A total of five microbeams with the same trapezoidal cross-section and lengths of 80, 85, 200, 250 and 500  $\mu m$ were tested to study size scaling effects and technique applicability. It is observed that the 80 and 85  $\mu\mathrm{m}$  beams exhibited linear elastic behavior and the 200, 250, and 500  $\mu m$  beams exhibited non-linear elastic behavior before fracture. Using the load-displacement curves for the 250  $\mu$ m beam we determined a Young's modulus value of E = 49.36GPa which is within a 12% of the published value for bulk GaSb, E = 56.2GPa. Besides being useful in studying the mechanical properties of bulk crystals and thin films the method can also be used to study crystallographic orientation effects by fabricating the beams in various orientations on the surface. More detailed results of all the microbeams will be presented in the paper.

## A5.15

Doped Nano-Porous Xerogel-Based Materials as Tunable, Ultra-Stable Sensor Elements. Ying Tang, Zunyu Tao, Elizabeth C. Tehan, Rachel M Bukowski and Frank V. Bright; Chemistry, The State University of New York, University at Buffalo, Buffalo, New York.

Key figures of the merit for all sensors include: sensitivity, reproducibility, and stability. In general, sensor researchers focus on one of these figures of merit at a time. In addition, most researchers take a one-to-one strategy wherein an "optimized" sensor is developed for a particular analyte. In our research we focus on the entire problem and aim to develop arrays of micron-sized sensor elements that are highly stable, robust, and that exhibit tunable sensitivities. In this way we can develop redundant sensor platforms and multi-modal detection strategies. This presentation will focus on a simple model system: the quenchometric detection of gaseous and dissolved O2. In this approach, luminophores are sequestered within a tailored nano-porous xerogel glass. As the  ${\rm O}_2$  diffuses into the glass, it can quench the luminophore emission to a degree that depends on the bimolecular rate of interaction between the luminophore and quencher  $(\langle k_q \rangle)$  and the excited-state luminophore luminescence lifetime  $(\langle \tau_o \rangle)$ . The speaker will summarize work on developing ultra-stable sensor elements: (1) based on tailored, binary xerogels; (2) with tunable sensitivities that use binary mixtures of luminophores (adjust  $\langle \tau_o \rangle$ ); and (3) with tunable sensitivities that use binary mixtures of sol-gel precursors (adjust  $\langle k_q \rangle$  and/or  $\langle \tau_o \rangle$ ). Together, these strategies yield sensor elements with continuously tunable responses that can be adjusted by nearly an order of magnitude.

A5.16
The Influence of InGaAs Strain-Relaxed Layer on Photoluminescence and Surface Morphology of InAs/GaAs Quantum-Dot Structure Grown by MOCVD. Shiang Feng Tang<sup>1</sup>, Shih Yen Lin<sup>2</sup>, Cheng Der Chiang<sup>1</sup>, Ray Ming Lin<sup>3</sup> and Ya Tung Cherng<sup>1</sup>; <sup>1</sup>Materials & Electro-Optics Research Division, Chung-Shan Insitute of Science & Technology, Tao-Yuan, Taiwan, Taiwan; <sup>2</sup>Union Chemical Laboratories, Industrial Technology Research Institute, Hsinchu, Taiwan; <sup>3</sup>Electronic Engineering, Chang Gung University, Tao-Yuan, Taiwan.

Three-stacked InAs/GaAs quantum-dot samples without and with InGaAs strain-relaxed layer denoted as sample A and B are grown by MOCVD at 500° C on (100) 2° toward (111) GaAs substrate. Under ultra low AsH3 background pressure, the growth conditions of InAs/GaAs quantum dot structure are the same for the two samples while an additional thin InGaAs layer is grown prior the quantum dot growth for sample B. The room temperature photoluminescence have revealed similar FWHM 70 nm, and 1267 and 1280 nm peak wavelength for sample A and B, respectively. Longer peak wavelength of sample B is attributed to bandgap lowering resulted from the strain relaxed effect on the InAs/GaAs QD structure and similar FWHM to the unchanged dot size distribution with the additional thin InGaAs layer. The 2 order of reduction of PL intensity for sample B is attributed to the reduction of quantum efficiency resulted from relaxed strain compared with sample A. The AFM images of the two samples in  $1000 \times 1000$  nm view region have revealed similar dot size distribution for the two samples and denser dot density for sample B. The phenomenon is attributed to the longer migration length for In adatoms on a less strained surface such that nucleation possibility for each In adatom would increase and the desorption of In atoms under low AsH3 background pressure would therefore decrease to form a denser dot density. In summary, the room-temperature PL and AFM images of the QD samples have revealed unchanged FWHM, longer peak wavelength, reduced PL intensity and denser dot density with the additional InGaAs strain relaxed layer. Relaxed strain induced bandgap lowering, quantum efficiency reduction and decreasing desorption possibility of In atoms are main mechanisms responsible for these phenomenon.

Small angle X-Ray Scattering (SAXS) on Deformed PVDF -Foils. Guenther Maier<sup>1</sup>, Gernot Wallner<sup>2</sup> and Peter Fratzl<sup>3</sup>; <sup>1</sup>ESI Inst. f. Metal Physics, MCL University of Leoben, Leoben, Austria; <sup>2</sup>IBWK, PCCL University of Leoben, Leoben, Austria; <sup>3</sup>ESI Inst. f. Metal Physics, Austrian Academy of Sciences University of Leoben, Leoben, Austria.

PVDF is an polymer witch shows a piezoelectric effect, can be used as matrix in photovoltaic cells and act as a pyroelectric transducer. For that the nanostructure of this material is very interesting and, because of the change in the electrical properties during deformation, studies on the nanostructure of the material are necessary to understand the difference between native and stressed samples. Small angle X - ray scattering is a good method to study the nanostructure of polymers and is very sensitive for changes. We applied tensile stress to thin PVDF foils with a different degree of crystallinity and studied the change in the structure while tensile stress was applied. For the change in the nanostructure, scanning three dimensional SAXS methods have been applied. For this purpose an uniaxially stretched foil was scanned over the deformed region of the specimen. Stretched foils visibly show 3 areas: undeformed regions are transparent, then some milkiness appears upon deformation and the area of plastic flow again is transparent. In the non deformed stage the samples show an isotropic orientation of the lamella, in the milky area randomly orientated crazes are formed and in the area of plastic flow a fibrous structure appears , showing an lamellar - angle of 60 degrees with respect to the stress direction. The distance of the lamella packages also changes from 11.0 nm to 6.5 nm upon stretching, which is a sign that the deformation is largely caused by microscopic shearing.

Stress Development in PZT/ZrO<sub>2</sub>/SiO<sub>2</sub> Stacks For MEMS Piezoelectric Unimorph Diaphragms Using Interdigitated Transducer (IDT) Electrodes. <u>Eunki Hong</u><sup>1</sup>, Robert L. Smith<sup>2</sup>

Silai V. Krishnaswamy<sup>2</sup>, Carl B. Freidhoff<sup>2</sup> and Susan Trolier-McKinstry<sup>1</sup>; <sup>1</sup>Materials Research Institute, The Pennsylvania State University, University Park, Pennsylvania; <sup>2</sup>Northrop Grumman Electronics Systems, Baltimore, Maryland.

Proper assessment of the stress of each layer in MEMS structures is an important aspect of their design and performance. In this work, stress development in piezoelectric MEMS diaphragm actuators was studied to understand its impact on resonant frequencies and the behavior. The stresses of 1.6  $\mu m$  Pb(Zr\_{0.52}Ti\_{0.48})O\_3[PZT]/0.3  $\mu m$  ZrO\_2/0.5  $\mu m$ 

 $SiO_2$  stacks are presented. In this stack, PZT acts as the active layer while the SiO<sub>2</sub> layer is the passive layer. Zirconia acts as a diffusion barrier between the PZT and SiO<sub>2</sub> layers. The stress of each layer was characterized by measuring the wafer curvature. This data is compared with results from load-deflection (bulge test) test on MEMS diaphragm structures. The measured stresses of PZT,  $\rm ZrO_2$  and  $\rm SiO_2$ were 100-150, 230-270 and -147 MPa. The total stress of the stack was around 80 MPa. In load-deflection measurements, the total stress was in 80-86 MPa range. The effect of poling and actuation voltage on the stress was also investigated; it was found that using IDT electrodes, the structure softens on actuation, yielding a net decrease in stress of around 0.2 MPa/V. This is in contrast to through-the-thickness poled samples, where actuation stiffens the membrane

Photo-Oxidation of Polysilane Deposited Using CVD for the Production of Waveguides. John P Lock and Karen K Gleason; Chemical Engineering, MIT, Cambridge, Massachusetts.

Polysilanes consist of a silicon backbone with organic sidegroups. Unlike polysiloxanes (silicones), polysilanes have no oxygen atoms between the silicon atoms. Plasma enhanced chemical vapor deposition (PECVD) of hexamethyldisilane gives a crosslinked, amorphous polymer film with a chemical composition resembling that of polydimethylsilane (PDMS). CVD presents the only pathway for some traditionally intractable materials like PDMS. UV irradiation of PDMS decreases its refractive index by about 5%, making it a good candidate material for the fabrication of waveguides and other optical devices. Waveguides with a refractive index contrast of 5% can have a turning radius of 1mm, which is sufficient for use in the interconnect layer of a microphotonic chip. A Si-Si linkage in PDMS has a bond strength of 80 kcal/mol, which corresponds to light with a wavelength of 350 nm. When polysilanes are irradiated with energetic UV light, they undergo an irreversible photo-oxidation reaction. The reaction inserts oxygen into Si-Si bonds along the polysilane backbone. The molecular density of the material decreases, resulting in a lower refractive index. The maximum achievable index contrast is between 5 and 10%. Irradiating PDMS with 900 mJ/cm<sup>2</sup> of light from a 193 nm laser dropped the refractive index of the material from 1.55 to about  $1.50. \ \mathrm{Modeling}$  predicts that a square waveguide with deposited PDMS as the core and irradiated PDMS as the cladding should be 1 um tall and 1 um wide to contain 850 nm light. This wavelength is important since photonic chips will likely integrate AlGaAs lasers and Si detectors. A shadow mask has been designed to test PDMS as a waveguide material and characterize its performance.

 $\frac{\textbf{A5.20}}{\textbf{Effects}} \ \textbf{of Softbake Parameters on a Benzocyclobutene (BCB)}$ Adhesive Wafer Bond. Daniel N. Pascual, Bonders, SUSS MicroTec, Waterbury Center, Vermont.

This study explores the effects of softbake parameters including temperature and duration on the homogeneity and strength of a Benzocyclobutene (BCB) adhesive bond. Experiments were initially performed on silicon wafer pieces and then verified using quartered wafers bonded on a different machine. A four-point bend delamination test was employed to quantitatively measure bond strength while uniformity was analyzed visually. A softbake temperature of 150°C for 10 minutes yielded void free bonds with a fracture toughness of 23  $J/m^2$ 

Characterization of Mechanical Properties of Silicon Nitride Thin Films for Space Applications. Wen-Hsien Chuang<sup>1,2</sup>

Thomas Luger<sup>1,2</sup>, Rainer K. Fettig<sup>3</sup> and Reza Ghodssi<sup>1,2</sup>; <sup>1</sup>Electrical and Computer Engineering, University of Maryland, College Park, Maryland, <sup>2</sup>The Institute for Systems Research, University of Maryland, College Park, Maryland, <sup>3</sup>NASA/Goddard Space Flight Center, Greenbelt, Maryland.

Mechanical properties of micro-electro-mechanical systems (MEMS) materials at cryogenic temperatures are investigated to extend MEMS sensors and actuators into space applications. A measurement setup is developed to measure the mechanical properties of MEMS materials from room to cryogenic temperatures. This setup is installed inside a focused-ion-beam (FIB) system, which can provide scanning electron microscopy (SEM), maskless ion milling and ion-induced platinum deposition. T-shape, low-stress LPCVD silicon nitride cantilevers suspended on a silicon substrate are fabricated using bulk micromachining technique. A lead-zirconate-titanate (PZT) translator powered by a function generator and a DC voltage is utilized as an actuator, and a silicon diode is used as a temperature sensor in the measurement setup. To measure Young's modulus, one cantilever is driven to its first resonant mode by the PZT translator. The resonant frequency is measured by pointing the electron beam in a fixed position where the vibrating cantilever moves in and out of the electron beam path. This modulates the secondary electron detector

signal with the frequency of vibration. This signal is acquired with an oscilloscope and the resonant frequency is determined. The Young's modulus of the silicon nitride thin film is then calculated from the resonant frequency. The quality factor is also determined from the vibrating amplitude near the resonant frequency, and is estimated as high as 1156 at room temperature. Therefore, the difference between resonant frequencies and natural frequencies can be neglected. For bending tests, a micro-needle mounted on the PZT translator is utilized to push the cantilever. The maximum bending angle for the cantilever before it breaks is measured to obtain the fracture strength. The Young's modulus of the silicon nitride thin film varies from 260.9 GPa at room temperature (298 K) to 273.3 GPa at 30 K and the fracture strength ranges from 28.01 GPa at room temperature to 37.35 GPa at 30 K. Detailed configuration of the experimental setup and measurement methods and results will be presented.

## A5.22 TRANSFERRED TO A1.2

### A5.23

Characterizing the Mechanical Response of Silicon Nanostructures. W. M. Mook, J. M. Deneen, C. R. Perrey, C. B. Carter and W. W. Gerberich; Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, Minnesota.

The mechanical response of freestanding silicon nanostructures to an applied stress is important to applications such as nanoimprint lithography (NIL) and microelectromechanical systems (MEMS). In the case of NIL, silicon nanostructures are etched to form a stamp that repeatedly contacts a substrate which transfers the etched pattern. This cyclic loading can lead to wear and eventually to nanostructure fracture. Similarly, MEMS are frequently designed where the silicon structures are supporting normal and/or tangential contact forces. In the above cases, the mechanical response (whether elastic, elastic-plastic or plastic) of the structure is critical in determining the reliability, and consequently the lifetime of the device. In this study, silicon nanostructures with lateral dimensions ranging from 50 to 200 nm were patterned using electron-beam lithography and etched into both bulk silicon and silicon-on-sapphire substrates. The mechanical response of the nanostructures to compressive stresses was evaluated by nanoindentation as a function of load, loading rate and structure size. The deformed structures were characterized using scanning probe and transmission electron microscopy

### A5.24

Fabrication of Nanochannel Array on Functional Oxide Thin Films. Shuhei Sato, Shusaku Akiba, Akifumi Matsuda, Jin Liu, Satoru Aida and Mamoru Yoshimoto; Materials & Structures Laboratory, Tokyo Institute of Technology, Yokohama, Kanagawa, Japan.

There has been great interest in fabrication methods for nanostructures with a wide range of applications that include electronic devices, chemical chips and nanoelectromechanical systems. And oxide materials have many useful properties about mechanical, thermal and electrical functions. We have reported the preparation of single crystal sapphire substrates with straight atomic steps and ultrasmooth terrace surface (Appl. Phys. Lett. Vol.67 (1995) 2615) By using these substrates, we have constructed nanostructures such as nanowires or nanodots of some oxide materials. Here we report on the novel method to create the straight nanochannel array on the oxide thin films, which uses the selective vaporization along the straight step edges of the ultrasmooth substrates at high temperatures. The YSZ (yittria-stabilized zirconia) thin films were deposited at 700°C by pulsed laser deposition (PLD) process on the ultrasmooth sapphire substrates. YSZ epitaxial growth was confirmed by reflection high energy electron diffraction (RHEED) and X-ray diffraction (XRD) Then, YSZ thin films were annealed in air at temperatures in the range of 1000°C to 1200°C. The film surfaces were observed by atomic force microscopy (AFM). When the epitaxial films were annealed at 1100°C, the channel structures on the film surfaces were generated, which showed the width of about 20 nm and the depth of about 2 nm. The nanochannels were well self-aligned along the step edges of the ultrasmooth sapphire substrate, and the separation between each channel corresponded to the atomic terrace width on the substrate before deposition. The nanochannel structures were also fabricated for other epitaxial oxide thin films of NiO, TiO2, and so on.

## A5.25

The Application of Combinatorial Approach in the Hydrothermal Syntheses of Open-framework Zinc Phosphates. Jihong Yu, Yu Song, Ruren Xu and Shouhua Feng; Department of Chemistry, Jilin University, Changchun, China.

Combinatorial methods have attracted considerable attentions because of their high efficiency in the discovery of new materials. The application of combinatorial approaches to hydrothermal synthesis has demonstrated their potential strengths in the discovery of

open-framework materials.1 Recently, we have applied this approach in the hydrothermal syntheses of open-framework zinc phosphates.2 Here we will describe the combinatorial method for the discovery and characterization of these zinc phosphate phases. The experimental design includes variation of the components of the reagents, the study of influence of transition metal cations, temperature and solvent on the crystallization. The multiautoclave consists of a stainless steel block with 64 Teflon chambers (0.7 cm in diameter, 3.0 cm in depth, 800 l in volume per chamber). The reagents were added into each individual Teflon chamber using a Tecan CH Miniprep 75 pipette robot. The autoclave was placed in an oven for 60 h at 160-200C. The separated products were characterized by automatic X-ray diffraction analysis carried out on Bruker D8 Discover GADDS. Applying combinatorial technique we have systematically investigated the influence of variation of the components of the reagents on the products in the reaction system 1.0 Zn(OAc)2- x H3PO4- y N, N-dimethylpiperazine-H2O. Two zinc phosphate phases, i.e. a new phase A ([Zn6P5O20(H2O)]&#8226;0.5C6H16N2&#8226;C5H14N2&#8226;3H2O),and a known phase B (Zn3P2O8H2O), have been discovered. At 180C, phase A forms in the range of 0.5  P/Zn  4 and 2  R/Zn  6 with the pH values of 7-9, while phase B forms in the areas of 0.5  P/Zn  4 and 0.33 R/Zn  1.67, and 6  P/Zn  7 and 3 R/Zn  6, respectively, with pH values of 5-7. It is found that some transition metal cations, such as Co2+, Fe2+, Ni2+, Mn2+, etc., have influence on the crystallization of phase A and B. Adding certain amount of Co2+ cations to the crystallization field of phase A causes the formation of a new phase C ([Zn5P4O16(H2O)]•C4H14N2) whose structure is similar to that of a reported hybrid zinc phosphate. Structural analysis indicates that A possesses a novel 3-D open-framework with intersecting 16-10-, and 8-membered ring channels along the [100], [010] and [001] direction, respectively. Its structure consists of tetrahedral ZnO4, ZnO3(H2O) and PO4 units. The influence of temperature, and solvent on the crystallization of these zinc phosphate phases has been investigated. References 1 Akporiaye, D. E.; Dahl, I. M.; Karlsson, A.; and Wendelbo,R. Angew. Chem. Int. Ed. 1998, 37, 609; Klein, J.; Lehmann, C. W.; Schmidt, H.-W.; and Maier, W. F. Angew. Chem. Int. Ed. 1998, 37, 3369; Choi, K.; Gardner, D.; Hilbrandt, N.; and Bein, T. Angew. Chem. Int. Ed. 1999, 38, 2891. 2 Song, Y.; Yu, J.; Li, G.; Li,Y.; Wang,Y.; Xu,R. Chem.Comm., 2002, 1720.

### A5.26

Detection of DNA Hybridization by Nano-scaled Electronic Devices. Chil Seong Ah, Yong Ju Yun, Dong Han Ha, Se Il Park and Wan Soo Yun; Electronic Device Group, Korea Research Institute of Standards and Science, Daejeon, South Korea.

We report on the detection of DNA hybridization using an electronic device with two electrodes separated by 30 nm from each other. Single-stranded DNA molecules were immobilized in the area between the electrodes by covalently attaching thiol-modified DNA molecules onto self-assembled monolayer of silane. Specific hybridization of the targeted DNA molecules tagged with Au nanoparticles of around 30 nm in diameter leads to an increase in the electric conductance between the electrodes readily measurable at room temperature. To ensure the high sensitivity of DNA detection, a micro-fluidic channel was used to guide sample-flow onto the active area of the device. We expect that this work should provide a promising way to developing DNA nano-arrays and nano-scaled biosensors.

## A5.27

Abnormal Contact Resistance Reduction in Bonded Cu Interconnects Using Pre-Bonding HCl Cleaning. Kuan-Neng Chen, Andy Fan, Chaun Seng Tan and Rafael Reif; Microsystems Technology Laboratories, Massachusetts Institute of Technology, Cambridge, Massachusetts.

The contact resistance of bonded Cu interconnects in three-dimensional integration technology was measured from a novel test structure. An abnormal gradual contact resistance reduction was observed in interconnects cleaned by HCl prior to bonding when the current was ramped from 1 mA to 100 mA. It is also observed that the contact resistance value decreases and converges to a constant value. However, no reduction in the contact resistance was observed for interconnects that skipped pre-bonding HCl clean. This abnormal contact resistance reduction can be explained by investigating the surface roughness prior to bonding and the interfacial microstructure after bonding. The surface roughness is higher in samples cleaned by HCl than samples without HCl cleaning. As a result, a high proportion of interface and voids remain at the original bonding interface of samples with pre-bonding HCl clean particularly when the bonding duration or the bonding temperature is not sufficiently high to remove the interface and voids. The interface and voids introduce a high contact resistance in the bonded interface. When the bonded interconnects with pre-bonding HCl clean are stressed with current

that increases gradually, a reduction in contact resistance is measured. Energy introduced by current in the bonding interface might assist refining the interface structure and shrinking the voids. By increasing the stressing current, the contact resistance eventually converges to a lower constant value. This also implies that the microstructure of bonded layers has reached a steady state.

Multi-Analyte Screening Using an Array of On-Chip Pores. Troy Christopher Messina<sup>3</sup>, Omar A. Saleh<sup>2</sup> and Lydia L. Sohn<sup>1</sup>, <sup>1</sup>Mechanical Engineering, University of California at Berkeley, Berkeley, California; <sup>2</sup>Physics, L'Ecole Normale Superieure, Paris, France; <sup>3</sup>Physics, Princeton University, Princeton, New Jersey.

We demonstrate multi-analyte sensing using an array of pores on a single chip. The pores are embedded in PDMS using micromolding techniques that allow rapid, reproducible fabrication with a high degree of structural and chemical flexibility. Using our previously demonstrated resistive pulse method of particle sizing [1-3], we can detect up to four different-sized colloids, each derivatized with a different analyte, per pore. By addressing N x M pores in our device, we can potentially screen for  $4 \times N \times M$  analytes simultaneously. Advantages to our multi-array pore beyond multi-analyte screening include the ability to screen without labeling the reactants, rapid detection using as little as nanoliters of solution (ng/mL concentrations), and device fabrication using the simple, inexpensive means of soft-lithography. In this report, we will show how we fabricate our multi-array pore device, and we will present preliminary results demonstrating the multianalyte screening capabilities of our device. 1. O. A. Saleh and L. L. Sohn, Rev. Sci. Inst. 72, 4449 (2001). 2. O. A. Saleh and L. L. Sohn, NanoLetters3, 37 (2003). 3. O. A. Saleh and L. L. Sohn, PNAS 100, 820 (2003).

Polyurethane Nanofiber Webs for Sensor and Actuator Applications in Microelectromechanical Systems (MEMS). yusuf ziya menceloglu, Yasar Gurbuz, Thomas Bechteler, Mansoor Naseer and Mustafa M Demir; Faculty of Engineering and Natural Sciences, Sabanci University, Istanbul, Turkey.

Multiphase, segmented polyurethanes are one of the most interesting classes of polymers. Due to their excellent elastomeric and physical properties, polyurethane was used in the production of high performance mats via electrospinning method to be utilized in mems-based transducers applications. The fabrication of the discontinuous mat made by electrospinning technique which a uniform electrical potential was applied to polyurethane solution (30-1600 centipoises) in dimethylformamide within a cupper probe of high voltage generator and grounded conductive sheet. The diameters of the nanofibers were obtained from 20 nm to 1.2 mm and depend mainly on the viscosity of the solution. Scanning Electron Microscope and Atomic Force Microscope were used to determine the fiber morphology and measure the fiber diameters. Thickness of the resulting film was adjusted by changing spinning time. We have implemented these elastic nanofibers in a mems-based pressure sensor to measure fluid flow. The pressure sensor consists of a parallel plate capacitor. One electrode is an aluminum that could be deposited on a silicon substrate. The other electrode is made of a flexible, conductive elastic nanofiber and is exposed to the pressure. The area of this membrane is 20x100mm2. The distance between the two electrodes under no pressure is 3mm. With increasing pressure the membrane is displaced and the electrode distance is decreased. This results in a larger capacitance. The simulated displacement of the membrane has been found to be linearly changing up to 1mm over a pressure range of 0.001 to 0.01 MPa. The resulting capacitance has been calculated and ranges with the above mentioned sensor dimensions between 6fF and  $12f\bar{\mathrm{F}}$  at zero and maximum displacement, respectively. These simulation results have shown that with very good elastomeric and physical properties, successful physical implementations of polyurethane-based elastic nanofibers could realizes for applications such as leak detection

Ceramic Microfabrication Techniques for Microdevices with 3-Dimensional Architecture. Balakrishnan Nair, Merrill Wilson, Akash Akash and Charles Lewinsohn; Ceramatec, Inc., Salt lake City,

The term "microfabrication" has been primarily used as an acronym for silicon based device fabrication. Recent developments in ceramic processing technology have resulted in cost-effective, scalable options of ceramic microfabrication that offer the potential for fabrication of devices with a number of advantages over silicon-based microdevices for specific applications. These advantages include the ability to fabricate devices with three-dimensional architecture, high-temperature operation up to 1200 C, porous layers for gas diffusion, and textured substrate properties for specific applications

through wider materials selection. Processing routes for these ceramic microdevices with three-dimensional architecture include established processes such as tape casting, laser machining, lamination and sintering, or new processes such as reaction bonding and lost-mold techniques. The ability to fabricate three-dimensional feature geometries allows the application of these ceramic microfabrication techniques for device fabrication targeted at a number of applications such as point-of-use high purity gas generation, microchannel devices, microreactors, fiber-optic connectors and heat-pipes for microelectronics.

Fluctuation microscopy studies of medium-range order structures of hydrogenated amorphous diamond-like carbon films. Xidong Chen<sup>1,2</sup> and Jacqueline Johnson<sup>3</sup>; <sup>1</sup>Cedarville University, Cedarville, Ohio; <sup>2</sup> Materials Science Division, Argonne National Lab, Argonne, Illinois; <sup>3</sup>Energy Technology Division, Argonne National Lab, Argonne, Illinois.

Hydrogenated amorphous diamond-like carbon (DLC) films have commanded great interest because of their useful properties, such as high wear resistance, low friction coefficients, chemical inertness, thermal stability, wide optical gap, high electrical resistivity and potentially low dielectric constant. They have been found or being considered for many applications, such as wear and corrosion protection of magnetic storage media, biological implant, micro-electromechanical systems (MEMS) and low dielectric materials for ultra-large-scale integrated circuits (ULSI). The film structure and properties are determined by the hydrogen content and the ratio of three-fold sp2 and four-fold sp3 carbon atoms. Hydrogen in DLC is known to be important for obtaining a wide optical gap and a high electrical resistivity, removing defect states and stabilizing the network. Yet very little is known about structures of DLC due to the amorphous nature of the material. We have studied medium-range order structures in these amorphous diamond-like films with fluctuation microscopy. By analyzing speckle dark-filed images taken over different areas as a function of momentum transfer in reciprocal space, we obtain a measure of the medium-range order. This is exhibited by the intensity fluctuation as a function of the momentum transfer vector and measured as a function of hydrogen content. We have found that the hydrogen content is directly correlated with the medium-range order, which explains property changes in these films. Future studies will be focused on systematic exploration of annealing effects.

 $\frac{\textbf{A5.32}}{\textbf{Localized, In-Situ Vacuum Measurements For MEMS}}$ **Packaging.** <u>Nicholas Moelders</u>, James T Daly, Anton C Greenwald, Edward A Johnson, Mark P McNeal, Ramesh Patel, Martin U Pralle and Irina Puscasu; Ion Optics, Inc., Waltham, Massachusetts.

MEMS devices have unique packaging considerations compared to conventional semiconductor devices. MEMS devices tend to have relatively large die size and many devices cannot tolerate elevated temperatures. Often these devices require a vacuum environment for efficient operation. While advances have been made in hermetic packaging of MEMS devices, vacuum packaging remains elusive. One significant problem in developing vacuum sealing has been the inability to determine, readily and non-destructively, the vacuum level inside the package. We have previously described the development of a silicon MEMS-based chip design, SensorChip $^{TM}$ , with integrated photonic crystal and reflective optics, which uses narrow-band optical emission and absorption for selective identification of gas and chemical species.‡ Because the power consumption required to maintain a specific temperature is directly related to the vacuum level, these devices effectively serve as microscopic Pirani gauges local vacuum sensors in the moderate vacuum range (0.01 to 1.0 torr) of interest to MEMS devices. Using the membrane itself as a vacuum gauge during sealing has proven to be an invaluable tool in developing a robust vacuum seal in a leadless chip carrier package. This paper reports progress in die-level hermetic packaging using the MEMS device as its own pressure gauge to optimize choice of solder, to optimize mounting design reducing trapped gas in bonding film, and to reduce adsorbed gases on package surfaces Designing Thermally Uniform MEMS Hot Micro-Bolometers,

Nicholas Moelders, et. al., MRS Symp. Proc. v.729, paper U5.2.

Electromechanical Behavior in Micromachined Piezoelectric Membranes. M. C. Robinson, J. Raupp, I. Demir, C. D. Richards, R. F. Richards and  $\overline{D}$  F Bahr; Washington State University, Pullman, Washington

Piezoelectric materials can convert mechanical and electrical energy, a particularly useful tool in developing micro and nanoscale systems. Characterizing the electromechanical behavior is essential to the design and optimization of the material and device performance.

Using piezoelectric thin films in MEMS requires the use of support structures and electrical isolation layers which do not participate in the piezoelectric transduction of energy. This paper will examine the influence of boundary (clamping) conditions, relative thickness variations between the active one to two micron thick piezoelectric membrane and underlying passive support structure, and the electrode coverage on the electromechanical behavior. Membranes were fabricated with silicon and silicon nitride and lead zirconate titanate with a ratio of Zr to Ti of 40:60 (PZT) thicknesses that provide thickness ratios between 1:2 and 2:1 by depositing the PZT using sequential solution deposition. PZT films contain a tensile stress that accumulates during processing, therefore a compressive stressed layer of tungsten was sputtered on bulk micromachined membranes to produce a near zero net residual stress. Piezoelectric coefficients were directly measured for various thickness films at strains exceeding 0.1% to determine the effect of both PZT thickness and support structure compliance, d31 of 70 pC/N have been demonstrated. The effects of thermal treatments on membranes versus bulk wafers, which impact the heat transfer during processing, have been compared using both conventional and rapid thermal annealing. A nonlinear finite element model is utilized for the analysis of the composite thin film. A comparison between the behavioral trends determined by modeling and experimental methods will be discussed.

### A5.34

Enhanced Initial Attachment of Fibroblast Cells on Titanium Oxide and Nitride Thin Films. Zuruzi Abu Samah<sup>1</sup>, Blaine Butler<sup>2</sup>, Cyrus Safinya<sup>2</sup> and Noel MacDonald<sup>1</sup>; <sup>1</sup>Materials Department, and Mechanical and Environmental Engineering Department, University of California, Santa Barbara, California, <sup>2</sup>Materials Department, Physics Department and Biomolecular Science and Engineering Program, University of California, Santa Barbara, California.

For some bio-micro-electro-mechanical system (BioMEMS) applications, it is desirable for cells to adhere on devices in the shortest time possible. In this work cell adhesion and spreading of mouse fibroblast cells cultured on thin (thickness < 1um) film oxides and nitrides of both silicon and titanium were studied to determine material systems for use as cell adhesion layers. Mouse fibroblast cells were cultured for times ranging from 1 to 24hr. It was found that these films have an effect on cell adhesion for short culture times with enhanced adhesion for titanium-based films and least on silicon nitride. Adhesion was significantly reduced when cells were cultured in serum-free media and suggests adsorption of serum-proteins mediated cell adhesion on these films. Cell culture and X-ray photoelectron spectroscopy studies indicated that differences in titanium oxide composition on surfaces of Ti and titanium oxide films did not result in changes in initial cell attachment to these films. These observations suggest that titanium-based thin films are suitable for integration as cell adhesion layers on devices.

## A5.35

Reliability of Microresonator as Derived from Stiffness Degradation Analysis. Jong-jin Kim, Dongil Son, Dong-Won Kim and Dongil Kwon; School of Materials Science and Engineering, Seoul National University, Seoul, South Korea.

Reliability issues such as fatigue and lifetime are more and more crucial in the development of commercial microelectromechanical systems (MEMS). Because conventional methods have significant limitations in MEMS applications due to their very small scale, more advanced methods are required. This study presents a new methodology based on analytical and statistical analysis of time-dependent degradation of resonating structures. To predict reliability from the degradation behavior, we introduced a new concept, degradation rate. Silicon and nickel microresonators with small sharp notches were designed and fabricated by micromachining and LIGA, respectively. They were then operated electrostatically at their resonance mode and the decrease in resonant frequency with operation cycles was measured. To investigate the effect of a notch as a local defect on the failure of the microresonator, we formulated a relation between the stiffness of a notched beam and the resonant frequency of the microresonator. Using this relation, we quantified the effect of notch depth ratio and crack growth near the notch tip on stiffness degradation and ultimate failure time. Finally, using the degradation rate concept in a statistical analysis of the measured resonant frequency, we evaluated a reliability function and hazard rate. In addition, we predicted the failure time of the microresonator and compared it with experimental results to verify the validity of the proposed methodology.

## A5.36 Abstract Withdrawn

## A5.37

Dissipation Mechanisms in Thin-Film Silicon Microresonators

on Glass. <u>Joao Gaspar</u><sup>1,2</sup>, Virginia Chu<sup>1</sup> and Joao Conde<sup>1,2</sup>;

<sup>1</sup>INESC Microsistemas e Nanotecnologias, Lisbon, Lisbon, Portugal;

<sup>2</sup>Materials Engineering Department, Instituto Superior Tecnico, Lisbon, Lisbon, Portugal;

Microresonators are technologically important since they can be used in a wide range of applications, such as RF filters and mixers, sensitive mass detectors and sub-nm positioners. Because of the low temperature processing of most thin-film technologies, thin-film microelectromechanical systems (MEMS) extend the applications of MEMS to large-area substrates such as glass and plastic. We characterize the performance of thin-film silicon MEMS microresonators fabricated on glass substrates. The structural layer material is n<sup>+</sup>-doped hydrogenated amorphous silicon (n<sup>+</sup>-a-Si:H) and the maximum processing temperature is 110°C. The resonance frequencies  $(f_{res})$  and quality factors (Q) of the structures are measured as function of pressure from atmosphere to below  $10^{-6}$ Torr, where intrinsic mechanisms rather than extrinsic air damping dominate the energy dissipation (1/Q). Since energy dissipation limits the resonators performance, it is important to understand the damping mechanisms involved. In order to identify the dominant energy dissipation mechanism, the resonance behavior is studied as a function of the resonator length (L), width (w), thickness (t) and the actuating electrostatic voltage  $(itV_G)$ . Resonance frequencies up to 70 MHz and quality factors up to 5000 are measured. It is experimentally observed that  $(f_{res})$  is proportional to  $1/L^2$  and t, and independent of w, in agreement with an electromechanical model. The Young's modulus, E, of n<sup>+</sup>-a-Si:H is extracted from experimental results to be  $\sim$  160 GPa, in agreement with values quoted in literature for a-Si. Qincreases slightly with L and varies linearly with the surface-to-volume ratio of the resonators, which is an indication that surface losses are limiting the  $\mathcal{Q}.$  The highest  $\mathcal{Q}\text{-values}$  obtained approach the thermoelastic limit of a-Si:H. This paper demonstrates that the performance of thin-film Si based resonators fabricated at low temperatures ( $\leq 110^{\circ}$ C) is approaching that of the best c-Si and poly-Si resonators fabricated at much high temperatures (~700-1100°C).

#### A5.38

Microfriction of stainless steel: Combined effect of grain size, load, and relative humidity. Giuseppe Bregliozzi<sup>1</sup>, Andrea Di Schino<sup>2</sup>, Imad Ahmed<sup>1</sup>, Jose Maria Kenny<sup>2</sup> and Henry Haefke<sup>1</sup>; <sup>1</sup>Micro and Nanomaterials, CSEM S.A., Neuchatel, Switzerland; <sup>2</sup>Materials Engineering Center, University of Perugia, Perugia, Italy.

Research on stainless steels is still continuing to further improve their properties. Some studies in literature also indicate their utility for microsystems. Among the various steel types, austenitic stainless steels have good corrosion resistance and formability. However, they also have a relatively low yield strength. One way of increasing the yield strength is by alloying it with nitrogen. Another way of increasing the yield strength without severely affecting the ductility is grain refining. The mechanical characterization of the austenitic steels with different grain sizes showed that the microhardness increases with decreasing grain size, as does the Young's modulus; however a slight decrease in ductility was also observed with decreasing grain size. This works present a study on the effect of atmospheric air humidity on the microfriction and wear of AISI 304 austenitic stainless steel characterized by two different grain sizes: 2.5  $\mu m$  and 40  $\mu m$ . The influence of low loads upon the friction behavior of the steel were examined with a reciprocating microtribometer. In all cases, sapphire balls up to 2 mm diameter were used as counterbodies. Friction-load curves reveal that, for applied forces in the micronewton regime, capillarity plays a dominant role. At high humidity both, the fine- and large-grained steels have high friction coefficients relative to measurements performed under dry conditions. A wear map generated for both grain sizes at a fixed relative humidity and loads of up to 15 mN shows differences in friction between the fine- and large-grained steel. At higher loads (20 mN) a reversal in the microfriction as well as wear behavior is observed: Under dry condition the friction is greater than under moist conditions.

## A5.39

Microtribological Properties of Differently Prepared Silicon Oxide Surfaces. Giuseppe Bregliozzi, <u>Imad Ahmed</u> and Henry Haefke; Micro and Nanomaterials, CSEM S.A., Neuchatel, Switzerland.

Silicon is the most common MEMS material and, for systems with moving parts in contact, the microtribological properties of the oxide covered surface plays an important role in defining the operational characteristics and lifetime of silicon-based MEMS devices. In this paper, the microfriction and adhesion properties of differently prepared silicon oxide surfaces were examined with a microtribometer. Experiments were performed in air at different relative humidities (RH) ranging from 5% to 95% RH. In each case, single crystal silicon (100) or (111) as well as polysilicon were used as substrates and the

counterbodies were 1-2 mm diameter silicon spheres. The various preparation methods consisted of applying a variety of cleaning and oxide growing techniques commonly used in industry and found in literature. These range from simply cleaning the already native oxide surface present on the silicon surface, applying wet chemical methods (for e.g. using piranha solution), creating oxides in vacuum, to generating thermal oxides. The surfaces were characterized by contact angle measurements and the topography was examined using atomic force microscopy. The pull-off force was determined as a function of time at different relative humidities following the fresh preparation of the oxide surfaces. Results indicate that change in the pull-off force between the oxide surfaces corresponds well with increase in the contact angle over time under ambient conditions. The pull-off force (adhesion) and contact angle results also correspond well with the microfriction experiments showing that capillary effects play a major role under low load conditions, especially for flat samples. Experiments with the relatively rougher polysilicon surfaces show a decrease in the pull-off force and microfriction. In experiments performed at different relative humidities, the microfriction and adhesion properties result from a combination of the chemical change of the oxide surface over time and increase of water adsorption on the

### A5.40

Effect of surface roughness on sliding friction in a vacuum. Masahiro Tosa, Akira Kasahara, Masahiro Goto and Tetsuo Ooishi; Materials Engineering Laboratory, National Insitute for Materials Science, Tsukuba, Ibaraki, Japan.

We have studied vacuum tribological materials to obtain precise and smooth sliding motion in a vacuum so far and shown that the materials with surface roughness around 100nm can offer as low friction in a vacuum as one at an atmospheric pressure. The surface roughness of the materials can be easily controlled not only by chemical polishing, mechanical polishing and electrochemical buffing but also by sputter deposition. This smooth sliding behavior is thought to arise from adsorption gas as a lubricant kept in hollows of nanoscopic asperities on the materials surface. We accordingly tried to estimate adsorption force by decrease in sliding load with the developed friction measurement system to study the effect of surface roughness on adsorption force and the effect on smooth sliding in a vacuum. We found existence of adsorption force less than 0.1mN on a stainless steel substrate with surface roughness over 600nm at an atmospheric pressure and in a vacuum. The substrate with the surface roughness under 40nm also showed adsorption force below 1mN in a vacuum, while sample with the surface roughness from 70nm to 250nm showed higher adsorption force over 1mN even in a vacuum. The surface roughness can also reduce desorption rate of adsorption gas and keep friction small in a vacuum. It is therefore concluded that the surface with 100nm roughness exhibiting similarly low friction in a vacuum as at an atmospheric pressure is ideal modified surface structure for strongly trapping adsorbed gas to act as good lubricant in a vacuum.

 $\frac{A5.41}{}$  Three-Dimensional Imaging of Magnetic Fields. R. Lloyd Carroll<sup>1</sup>, B. Wilde<sup>2</sup>, S. Washburn<sup>1</sup> and R. Superfine<sup>1</sup>;

<sup>1</sup>Physics and Astronomy, University of North Carolina - Chapel Hill, Chapel Hill, North Carolina; <sup>2</sup>Computer Science, University of North Carolina - Chapel Hill, Chapel Hill, North Carolina.

Control of the forces applied to microstructures in magnetic fields is emerging as a useful means of positioning, moving, and investigating behaviors on the microscale. To exert such control, a precise understanding of the shape and intensity of applied magnetic fields is necessary. Finite Element Modeling it is the standard for such modeling. Until now, comparison of such modeling to control experiments has proven difficult. We have developed a simple, straightforward technique wherein the magnetic fields in a device may be imaged on the nanoscale. Our technique allows us to prepare a high-resolution three-dimensional map of the shape and relative intensity of magnetic fields around complex structures. Analysis of the shape and intensity of magnetic field images near complex devices allows us to modify device designs to optimize the desired field patterns.

## A5.42

Surface Micromachined MEMS Pumps Using Piezoelectric Diaphragm Actuators Driven by Interdigitated Transducer (IDT) Electrodes. Eunki Hong<sup>1</sup>, Robert L Smith<sup>2</sup>, Silai V

Krishnaswamy<sup>2</sup>, Timothy T Braggins<sup>2</sup>, Carl B Freidhoff<sup>2</sup> and Susan Trolier-McKinstry<sup>1</sup>; <sup>1</sup>Materials Research Institute, The Pennsylvania State University, University Park, Pennsylvania; <sup>2</sup>Northrop Grumman Electronics Systems, Baltimore, Maryland.

MEMS pump structures with piezoelectric diaphragm actuators were fabricated by surface micromachining [1]. The diaphragm actuators

were driven with ring-shaped IDT electrodes. In this configuration, stress was concentrated at the center of the diaphragm, creating sharp deflection and reduced stroke volume in the pump structures. To overcome this problem, the IDT electrodes were modified. For the novel electrode design, finite element analysis showed flatter deflection at the center. However, the amplitude of the deflections decreased at the same time. The fabricated diaphragm actuators, fabricated by bulk micromachining, gave flatter deflections, as predicted. For example when actuated by 100 V, some actuators were flat over 15%of the diaphragm. Deflections of 760  $\mu\mathrm{m}$  diaphragms were about 5 microns for an activation voltage of 150V. MEMS pumps utilizing this novel design were fabricated. Characteristics of the pumps will be presented. [1] E. Hong, S.V. Krishnaswamy, C.B. Freidhoff and S. Trolier-McKinstry, Fabrication of Micromachined Piezoelectric Diaphragm Pumps Actuated by Interdigitated Transducer Electrodes, Mat. Res. Soc. Symp. Proc. Vol. 741, 2003.

Enzyme Catalyzed Polymerization in situ Self-Assembled Polyelectrolyte Microcapsules. Rohit C. Ghan<sup>1</sup>, Tatsiana Shutava<sup>1</sup>, Amish Patel<sup>1</sup>, Vijay John<sup>2</sup> and Yuri Lvov<sup>1</sup>; <sup>1</sup>Institute for Micromanufacturing, Louisiana Tech University, Ruston, Louisiana; <sup>2</sup>Department of Chemical Engineering, Tulane University, New Orleans, Louisiana.

This study presents the results of polymerization of inorganic monomer to yield fluorescent polymers encapsulated within shells fabricated via layer-by-layer (LbL) assembly. The model used for demonstrating this synthesis is polymerization of 4-(2-aminoethyl) phenol hydrochloride commonly known as tyramine hydrochloride to its corresponding polymeric form by reacting it with hydrogen peroxide catalyzed by enzyme horseradish peroxidase (HRP). Hollow polyelectrolyte microcapsules were prepared using weakly cross-linked melamine formaldehyde resin (MF) particles. Characterization of the adsorption of oppositely charged polyelectrolytes onto the MF cores was done using zeta potential measurements. Changing the pH of the core solution to 1.1 dissolved the MF cores. Formation of the hollow capsules was confirmed by observations from fluorescence microscope. Horseradish peroxidase (HRP) was encapsulated in these capsules by means of regulation of the pH of the shell solution. The shells were then suspended in a concentrated solution of monomer. Since the monomer is a low molecular weight species, it was able to freely permeate through the capsule wall into the capsules. Addition of aliquots of hydrogen peroxide initiated the polymerization reaction and the polymer formed from the ensuing reaction was confined in the hollow shells due to its high molecular weight. Fluorescence spectrometry (FS) and confocal laser scanning microscopy (CLSM) confirmed that the polymer formed in situ hollow shells exhibited fluorescence. Emission and excitation wavelengths for this polymer were found to be  $\lambda_{excitation} = 350$  nm and  $\lambda_{emission} = 404$  nm respectively. We are currently characterizing properties of the fluorescent polymer such as structural conformation, mechanical strength, and long-term fluorescence retainment studies, which we plan to present at the conference. Encapsulation of synthesized polymers and other species like enzymes or pharmaceutical chemicals in situ polyelectrolyte microcapsules offer various applications as controlled chemical reaction systems, drug-delivery systems, sensors for hydrogen peroxide and photoactive systems for optical applications.

## A5.44

Effective Temperatures and Fluctutation Dissipation Relation  ${\bf in~Structural~Glasses.}$  Shomeek  ${\bf Muk\underline{hopadhyay}}$  and Nathan Israeloff; Physics Department, Northeastern University, Boston, Massachusetts

Understanding the thermodynamic and microscopic basis of glass forming liquids and polymers remain one of the outstanding problems in Condensed Matter Physics. The concept of effective temperature has been proposed on theoretical grounds to explain how the slow degrees of freedom reach equilibrium to bath temperature. We present experimental studies on supercooled glycerol to measure effective temperature using dielectric spectroscopy and noise measurements and to probe whether there are deviations from the fluctuation dissipation relation.

## A5.45

Investigation of Leakage Current in Silicon Nitride MIM Structures for Capacitive Switches. Abuzer Dogan, Eugene Furman, Suzanne Mohney and Susan Trolier-McKinstry; Materials Engineering and Science, The Pennsylvania State University, University Park, Pennsylvania.

Silicon nitride thin films dielectrics can be used in capacitive radio frequency micro-electromechanical systems switches. The lifetime of the switches is believed to be adversely affected by charge trapping in the silicon nitride. In this work, the leakage current of

metal-insulator-metal structures with silicon nitride dielectric layers and top electrodes with different work functions have been studied over a wide range of electric fields. The silicon nitride dielectric layers and electrodes had thickness of  $0.25~\mu m$  and  $0.15~\mu m$ , respectively. The magnitude of the leakage current was inversely correlated to the work function of the metal with the largest leakage current for the Pt, the smallest for the Al, and intermediate for the Au top electrodes. This electrode dependence was observed for both voltage polarities and up to the highest electric field of 4 MV/cm. From the study of the sequence of applied voltages and their polarities, charge injection and trapping play important roles. Specifically, at moderate fields, a larger leakage current was observed for both polarities after high field stressing.

## A5.46 TRANSFERRED TO A10.1

## A5.47 Abstract Withdrawn

### A5.48

Dielectrofluidic Assembly and Integration of Nanotube and Nanowire Nanoelectromechanical Systems. Stephane Evoy,

<sup>1</sup>Electrical Engineering, Univ. of Pennsylvania, Philadelphia, Pennsylvania; <sup>2</sup>Mechanical Engineering and Applied Mechanics, Univ. of Pennsylvania, Philadelphia, Pennsylvania; <sup>3</sup>Chemistry, Penn State Univ, University Park, Pennsylvania; <sup>4</sup>Electrical Engineering, Penn State Univ, University Park, Pennsylvania; <sup>5</sup>Electrical Engineering, Virginia Tech, Blacksburg, Virginia.

Nanoelectromechanical systems (NEMS) represent a powerful platform for the detection of physicochemical processes with high sensitivity. However, surface structural issues impede their viability as resonant sensors. In addition, cantilever functionalization without sacrificing mechanical quality becomes problematic at these scales. Surface machining therefore does not provide intrinsic handles for the production of NEMS offering high-quality resonance and sensing specificity. We here report on a novel approach to nanomechanical design and integration that is based on the bottom-up synthesis and assembly of functional nanostructures. We report the dielectrofluidic assembly of multi-walled carbon nanotube NEMS onto specific sites of a prefabricated circuit. The diameters of these tubes range from 100 to 250 nm, their suspended lengths vary from 1 to 5 um, and their resonance range in the low MHz. Such frequency is unexpectedly low given the relatively high Young's modulus of carbon tubes. However, close inspection reveals the tubes to be partially collapsed, thus possessing a reduced bending stiffness compared to what would be expected from a cylindrical beam. We will present data obtained from smaller diameter, uncollapsed tubes, as well as from recently acquired GaAs nanowires. Finally, we will discuss the integration of such functional structures with standard VLSI circuits.

## A5.49

Parametric Approach to Development of a MEMS Chemical Sensor. <u>Daniel Arecco</u> and Ryszard Jan Pryputniewicz; Mechanical Engineering, Worcester Polytechnic Institute, Worcester, Massachusetts

There currently exist several different types of chemical sensors, each based on a different method of operation: capacitive, thermal, resistive, etc., depending on specific applications. While there is a number of ways for the detection of chemicals it is usually based on either adsorption or absorption of the target chemical by means of a special material. Once the target chemical is attached to the sensor it can then be measured by one of several methods. For the sensor to absorb chemicals or organisms special materials and polymers are used that attract the intended chemical. One of the developments in this field has been based on advancement of cantilever type sensors. These cantilevers are usually on the order of only about 150 mm in length, 30 mm in width, and just a few mm in thickness. During fabrication, a special coating, usually a polymer, is applied to the cantilever. This coating attracts and bonds target chemicals to itself. Once the chemical absorbs on the polymer, mass of the cantilever alters changing its resonance frequency. This change in frequency can then be detected and analyzed to determine concentration of the target chemical in the medium surrounding the sensor. While chemical sensors are fairly advanced, there is still a lot of work to be done to optimize their functional operation. This paper focuses on design, analysis, and characterization of a cantilever type MEMS chemical sensor, including determination of uncertainties. Results indicate that parametric approach provides effective means for development and optimization of a cantilever type MEMS chemical sensor.

## A5.50

Effect of polymer substrates on nano scale hot embossing. Jin Hyung Lee, Hyun-Woo Lim, Jin-Goo Park, Eun-Kyu Lee and Yang-Sun Kim; Micro Biochip Center, Hanyang University, Ansan, South Korea.

Hot embossing has been widely accepted as an alternative to photolithography in generating patterns on polymeric substrates. The optimization of embossing process should be accomplished based on polymer substrate materials. In this paper, the effect of polymer substrates on nano scale hot embossing process was studied. Silicon molds with nano size patterns were fabricated by e-beam direct writing. Molds were coated with self-assembled monolayer (SAM) of (1, 1, 2, 2H perfluorooctyl)-trichlorosilane to reduce the stiction between mold and substrates. For an embossing, pressure of 55, 75 bar, embossing time of 5 min and temperature of above transition temperature were performed. Polymethylmethacrylates (PMMA) with different molecular weights of 450,000 and 950,000, MR-I 8010polymer (Micro Resist Technology) and polyaliphatic imide copolymer were applied for hot embossing process development in nano size. These polymers were spun coated on the Si wafer with the thickness between 150 and 200 nm. The nano size patterns obtained after hot embossing were observed and compared based on the polymer properties by scanning electron microscopy (SEM). The imprinting uniformity dependent on the pattern density and size was investigated. Four polymers have been evaluated for the nanoimprint. By optimizing the process parameters, the four polymers lead to uniform imprint and good pattern profiles. A reduction in the friction for smooth surfaces during demoulding is possible by polymer selection.

### A5.5

Dielectric Material Effects On Electrowetting-On-Dielectric Pumping For Mems Biological Sensor. Ryan T. Marinis and Ryszard J. Pryputniewicz; Mechanical Engineering, Worcester Polytechnic Institute, Worcester, Massachusetts.

Microelectromechanical Systems (MEMS) have integrated traditional microelectronics and mechanical components in a variety of fields, including biological sensing. MEMS biosensors have the ability to conduct near real-time analyte detection in a very reliable fashion. In order to successfully utilize this analyte detection technology there is a need for understanding of flow characteristics of fluids on the microscale. The scaling of fluids to the microscale introduces forces that may be neglected on the macroscale such as surface tension. Due to the high area to volume ratio surface tension forces become large as size decreases. This concept can be harnessed in order to utilize the scaling effects of the fluid for beneficial purposes, such pumping by electrocapillary or thermocapillary effects. Preliminary results show that electrocapillary, also referred to as electrowetting (EW), effect can generate high pressures if a dielectric layer is used instead of the electric double layer (EDL) between the fluid and electrode in a microfluidic channel. This process, known as electrowetting-on-dielectric (EWOD), has been explored for a variety of dielectric materials. In this paper, results of analytical calculations of EWOD pumping with different dielectric materials are presented. There is also a need to understand the working fluid to properly design a microfluidic system. In many biological fluids there are particulates, such as cells, that may require special design consideration. Analysis of the effects of particulate size on flow is also discussed. It is shown that EWOD is an effective method of pumping fluid with no moving parts that can be incorporated into a biosensor application.

## A5.52

Evaluation of Design and Performance of a Cryogenic MEMS Micropump Realized With Si-Based Diaphragm Using Combination of ZYGO/WYCO Interferometer and Raman Spectroscopy. Yi Zhao¹, Biao Li², Daryl Ludlow¹ and Xin Zhang¹; ¹Department of Manufacturing Engineering, Boston University, Brookline, Massachusetts; ²Fraunhofer USA Center for Manufacturing Innovation, Brookline, Massachusetts.

The MEMS pumps usually consist of multiple layers of silicon that form the diaphragm. An accurate determination of diaphragm deformation is critical to micropump design, especially to better control of variable pumping rate and/or pumping output. As a solution to the problem of cooling satellite instrumentation to cryogenic temperature, a MEMS-size pump was designed and characterized to transport liquid nitrogen/oxygen between an evaporator (attached to the instrumentation) and a condenser (in contact with a cryocooler). This approach yields a system with minimal hardware, greatly reduced mass, and a significantly smaller working fluid charge. As a start-up of such cryogenic micro-pumped transport system, a series of square silicon diaphragms were fabricated using wet chemical etching; the diaphragm thickness was measured using Fourier Transform Infrared Spectroscopy. In this study, a large range of pressure was applied to the diaphragm samples using a compressive nitrogen gas, and a ZYGO laser displacement measurement system was employed to estimate the diaphragm deformation. For the purpose of calibration and mapping, a WYCO interferometer was further applied. As a result, the relationship between the pumping output (volume change) and the applied

pressure was achieved. Additionally, it was observed that if the diaphragm has sustained a high pressure, there was a plastic deformation existing in the diaphragm. The effect of the plastic deformation on the pumping output was also investigated. In order to optimize the diaphragm design and performance, a finite element analysis was preliminarily applied and stress distribution was further evaluated/mapped using Raman spectroscopy. It was found that the stress increased with increasing the lateral size of diaphragm, and concentrated at the middle point of each edge of the diaphragm. This information was then used to size an appropriate micropump assemblage for cryogenic applications, in which a decreased stress peak and better stress uniformity were successfully achieved.

### A5.53

The Influence of Interfacial Atomic Steps on the Leakage Current in MOS Capacitors. Valerian Ignatescu and Jack M. Blakely; Materials Science and Engineering, Cornell University, Ithaca, New York.

The 90 nm process for future microprocessors will feature CMOS transistors with 50 nm wide gates and 1.2 nm thickness of SiO2 gate oxide layer (only 5 atoms thick!) [1]. Tunneling currents through such small thicknesses should be extremely sensitive to interfacial imperfections that cause variations in thickness. Oxides with higher dielectric constants than that of silicon oxide are under study, but they are more difficult to grow. In the present paper we investigate the influence of surfaces steps on the tunneling current through thin oxides at small thicknesses. We first prepare large step-free rectangular zones (30x30  $\mu$ m) on the Si(111) surface through a process already described in several recent papers (see for example [2]). After this, a thin layer of thermal oxide is grown and then rectangular gold contact pads with sides of 20  $\mu$ m are deposited inside the step-free areas. Similar gold pads are also deposited on the surrounded stepped regions as described in the Ref. [3]. In this way we obtain arrays of MOS capacitors. We will report on the variation of leakage current with applied voltage for a range of oxide thicknesses in the nanometer range. References: [1] S. Thompson et al., Intel Technology Journal 06 (2002) 5 [2] D. Lee and J. Blakely, Surface Science 445 (2000) 32 [3] A. Oliver and J. Blakely, MRS Fall Meeting, December 2-6 2002, Boston, V4.6

### A5.54

High Temperature Micromolding of Aluminum with Surface Engineered LiGA Inserts. Dong Mei Cao and Wen Jin Meng; Mechanical Engineering, Louisiana State University, Baton Rouge, Louisiana.

The LiGA (Lithographie, Galvanoformung, Abformung) technology is a leading micromanufacturing technique for fabricating high-aspect-ratio microscale structures (HARMs) out of non-silicon-based materials. The ability to fabricate metallic HARMs is key to the realization of many harsh-environment-compatible micromechanical devices. In particular, the ability to fabricate Al-based HARMs would enable the construction of high efficiency micro heat exchangers for stand-alone and embedded applications. Micromolding of Al or other reactive metals has, however, not been achieved to date. We report, for the first time to our knowledge, successful repeated high-temperature micromolding of Al, utilizing LiGA fabricated microscale Ni inserts conformally coated with a Ti-containing hydrocarbon (Ti-C:H) coating. Our results demonstrate that the key to successful micromolding of reactive metals is to alter the chemical/mechanical interactions between the insert and the molded metal. With an instrumented molding apparatus, important molding parameters such as the maximum molding force, demolding force, and work of molding are measured. The effectiveness of surface engineered Ni inserts in Al micromolding will be illustrated through comparison with Al molding with conventional, non-surface-engineered Ni inserts.

## A5.55

Adhesion Force and Nanotribological Characterization of Chemical Vapor Deposited Fluorocarbon Films.

Nam Kyun Kim<sup>1</sup>, Taegon Kim<sup>1</sup>, Jin-Goo Park<sup>1</sup>, Hyung-Jae Shin<sup>2</sup> and Woon-Bae Kim<sup>2</sup>; <sup>1</sup>Metallurgy and materials engineering, Han yang university, Ansan, Kyung Ki do, South Korea; <sup>2</sup>Samsung Advanced Institue of Technology, Suwon, South Korea.

In this study, adhesion force and nano tribological characteristics of chemical vapor deposited fluorocarbon (FC) thin films were reported. Teflon-like FC surface has been widely used as passivation layer because of its low coefficient of friction, low adhesion force and high hydrophobicity. FC surfaces have been commonly prepared by self assembled monolayer (SAM) and chemical vapor deposition (CVD). The films by SAM are easy to deposit, but do not have enough nano tribological characteristics. They are also not compatible with manufacturing process due to its lacks of reproducibility and controllability. FC thin films were prepared by PECVD to obtain

desired chemical and nano-mechanical properties. The cleaning of Al surface was performed in O2 plasma with a RF power. C4F8 was supplied to chamber to deposit FC thin films. Contact angles of water and diiodomethane (CH2I2) were measured to calculate the surface energy. In order to investigate nano-tribological characteristics of FC thin films, atomic and lateral force microscopy (AFM/LFM) have been used for the measurements of adhesion forces and the friction force. Fourier transform infrared spectroscopy (FTIR) was used to analyze chemical structure of the FC thin films. Variable angle spectroscopic ellipsometry (VASE) was used to measure thickness and optical properties. The presence of FC films on substrates changed its contact angle from 10° to 110°. The surface energy was measured to be around 15 dynes/cm. The contact angle hysteresis, which usually indicates the surface heterogeneity, was less than 26°. The friction force of films coated surface was three times lower than bare Al. The adhesion force between AFM tip (Si3N4) and FC films was decreased in the presence of FC films. FTIR analysis showed the presence of C-F groups. Thermal stability and tribological reliability were also tested

#### A5.56

A Novel Device for Fountain Pen Nanolithography - Design and Microfabrication. Horacio D. Espinosa, Nicolaie A. Moldovan, Keun-Ho Kim and Changhong Ke; Mechanical Engineering, Northwestern University, Evanston, Illinois.

A novel atomic force microscopy (AFM) probe has been developed to expand the capability and applications of dip-pen nanolithography (DPN) technology. Using AFM tips, chemical ink can be deposited by DPN with a line-width resolution of less than 20 nm. However, the ink needs to be replenished periodically, which requires dismounting the AFM probe. In order to resolve this shortcoming of DPN, a new AFM probe has been designed and produced which can continuously feed ink without interrupting the DPN process. This new probe has integrated microchannels and reservoirs, which allow "fountain-pen" writing called "Fountain-Pen Nanolithography" (FPN). Ink is transported from the reservoir through embedded microchannels and eventually dispensed onto substrates via a volcano tip that is a completely novel design for microfluidics. Numerical simulations were performed to select optimal materials and suitable tip shapes providing stable fluid-air interface in the writing tip. AFM probes with integrated microchannels and volcano tips were produced by surface micromachining technology, specifically, employing three stacked layers of film. Fluid flow was tested through implemented microchannels using water-soluble fluorescence dye. The proposed FPN device will be arrayed and actively actuated to accomplish nanolithography in a massively parallel way.

## A5.57

Novel Opto-Ceramic Materials for Microchip and High Power Ceramic Lasers. Xuesheng Chen<sup>1</sup>, Kewen Li<sup>2</sup>, Kevin Zou<sup>2</sup>, Run Zhang<sup>2</sup>, Hua Jiang<sup>2</sup>, Gonul Ozen<sup>3</sup> and B. Di Bartolo<sup>3</sup>; <sup>1</sup>Dept. of Physics & Astronomy, Wheaton College, Norton, Massachusetts; <sup>2</sup>Boston Applied Technologies, Woburn, Massachusetts; <sup>3</sup>Dept. of Physics, Boston College, Chestnut Hill, Massachusetts.

Ceramic laser materials have recently received a lot of attentions because they are potentially great candidates for efficient microchip or high power lasers. Contrast to popular single-crystal laser materials such as the rare-earth ion Nd doped single crystal YAG, ceramic materials are easy to be fabricated into large size and can accommodate high rare-earth ion (laser ion) concentrations. The first ceramic (Nd:YAG) laser output was obtained in 1995, and the first Yb:Y2O3 ceramic laser was just demonstrated in April 2003. These two materials are the only ceramic materials that have shown lasing and some promising future in their applications for microchip or high power lasers. Their polycrystalline structures show average grain size in the micron range and grain boundary size in the nanometer range. To make ceramic lasers possible, the ceramic materials have to be made highly transparent with very low porosity and scattering loss. This presentation reports a new, transparent ceramic material, Er-doped Pb1-xLaxZryTi1-yO3 (PLZT), which we have recently fabricated and extensively studied, for possible high efficient microchip or high power laser applications. Outstanding optical devices such as active variable optical attenuator used in telecommunication have been developed in recent years using this host ceramic material PLZT, which is highly transparent and has exceptionally high electro-optic effect. Detailed optical property studies of this newly developed opto-ceramic Er-doped PLZT at different Er concentrations will be reported. These studies will provide crucial information in its promising applications in new optical devices including lasers.

## A5.58

The Nanotribology of Ultrananocrystalline Diamond: An Ideal Candidate for MEMS/NEMS? David S. Grierson<sup>1</sup>, Anirudha V. Sumant<sup>1</sup>, Jennifer E. Gerbi<sup>2</sup>, James P. Birrell<sup>2</sup>, Orlando O. Auciello<sup>2</sup>, John A. Carlisle<sup>2</sup> and Robert W Carpick<sup>1</sup>; <sup>1</sup>Engineering Physics, University of Wisconsin - Madison, Madison, Wisconsin;

 $^2\mathrm{Materials}$ Science Division, Argonne National Laboratory, Argonne, Illinois.

As devices shrink in size, the need for materials that exhibit low friction, adhesion, and wear becomes more critical due to the increased role of surface forces at high surface-to-volume ratios. Ultrananocrystalline diamond (UNCD) films have unique mechanical and tribological properties that exhibit significant potential for addressing this need because of the high stiffness, strength, fracture toughness, and surface chemical inertness of these films. One particular application is in microelectromechanical systems (MEMS) devices, where UNCD has the potential to alleviate, if not eliminate, current critical tribological problems in these devices. Our study is aimed at probing both the top "as-grown" surface and the bottom "nucleation" surface (exposed by etching the substrate) of UNCD films. The bottom surface plays a particularly critical role in dynamic MEMS since it is the surface that will be in more frequent contact with other underlying surfaces in devices such as cantilevers, membranes, and gears. Atomic force microscopy is used to examine the nanotribological properties of these surfaces, and Auger electron spectroscopy (AES), near-edge x-ray absorption fine structure (NEXAFS), x-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and scanning electron microscopy (SEM) are used to characterize the surface structure and chemistry. We have probed the effects of hydrogen termination on both surfaces by examining the morphological, adhesive, and frictional properties of the H-terminated vs. untreated UNCD films as a function of load, sliding speed, and relative humidity. Our initial results indicate that H-terminated UNCD exhibits extremely low adhesion, essentially indistinguishable from that of H-terminated single crystal diamond. We will also discuss the resulting relationships between nanotribological behavior, surface morphology, and surface chemistry of UNCD and how this relates to UNCD MEMS performance. This work was supported by the US Department of Energy, BES-Materials Sciences, under Contracts DE-FG02-02ER46016 and W-13-109-ENG-38.

### A5.59

Opening of multi-walled carbon nanotubes by controlled oxidation. Fabrice Valsaque, Moulay Rachid Babaa, Nicole Dupont-Pavlovsky, Xavier Duval and Edward Mcrae; UMR UHP - CNRS 7555, Laboratoire de Chimie du Solide Mineral, Vandoeuvre les Nancy, France.

The opening of carbon nanotubes, which gives various elements access to their interior surface, is of great importance for creating new material functionalities or for enhancing adsorption properties. As first shown by Ajayan and Iijima [1], carbon nanotubes can be opened by means of a chemical reaction with oxygen heated at an elevated temperature. In the present work, we further investigate this technique in the case of multi-walled carbon nanotubes. In particular, we try to determine the optimal amount of oxygen required to open such tubes without damaging them significantly. By gradually injecting small quantities of oxygen into a cell containing multi-walled carbon nanotubes, we study the oxidation process step-by-step through TEM imaging. In order to specify the optimal amount of oxygen required to open the maximum number of tube with as little damage to their outer surfaces as possible, the microscopic studies are completed by measurement of krypton and xenon adsorption isotherms. The optimal amount is determined through comparison of the results with those obtained under the same conditions before oxidation. [1] Ajayan and S. Iijima, Nature (London) 361, 333 (1993)

## A5.60

A multi-scale approach to predicting friction and wear in MEMS. Robert W Carpick, Erin E. Flater, Can K Bora and Michael E. Plesha; Engineering Physics, University of Wisconsin - Madison, Madison, Wisconsin.

The design of reliable MEMS devices that involve sliding or rolling surfaces requires a predictive capability for friction and wear. In this study, we use atomic force microscopy (AFM) to resolve critical roughness features of silicon MEMS surfaces from the nm-to- $\mu$ m scale on the top and bottom surfaces of a contacting interface. From this information, we derive surface roughness parameters that are used as inputs to predict the interfacial mechanics from generalized models based on the Greenwood-Williamson approach using transformation methods developed by McCool. We also employ a fractal approach in which we derive parameters that are optimized to match the measured topography. These methods allow us to determine parameters such as the real contact area as a function of the applied pressure. Using single crystal silicon surfaces coated with hydrophobic monolayers, we measure single-asperity constitutive laws using AFM. The AFM tip radii are determined using calibration techniques and are in the same range as the MEMS surface asperities, which ensures the methodology is relevant. From the calculations of the real contact areas, and using the measured constitutive friction laws, we make predictions for the measured friction behavior. We will discuss the validation of these

predictions with reference to multi-asperity MEMS friction test device experiments. This work was supported by the US Department of Energy, BES-Materials Sciences, under Contract DE-FG02-02ER46016, and by Sandia National Laboratories.

### A5.61

Core-shell domain structures in Pb(Fe<sub>2/3</sub>W1/<sub>3</sub>)O<sub>3</sub>-PbTiO<sub>3</sub> at the morphotropic phase boundary. zhenrong Li<sup>1</sup>, Paula Vilarinho<sup>1</sup> and Ian Reaney<sup>2</sup>; <sup>1</sup>Department od Ceramics and Glass Engineering, University of Aveiro, Aveiro, Portugal; <sup>2</sup>Department of Engineering Materials, University of Sheffield, Sheffield, United Kingdom.

 $Pb(Fe_{2/3}W_{1/3})O_3$  (PFW) is a relaxor ferroelectric that exhibits a diffuse and frequency dependent dielectric permittivity maximum at low temperature (~185K). Similar to other complex, Pb based perovskite relaxors, the macroscopic properties of PFW can be tailored by the formation of multicomponent systems, e.g., 1-xPb(Fe $_{2/3}$ W $_{1/3}$ )O $_3$  - xPbTiO $_3$  (PFW-PT). The low transition temperature (T $_c$ ) of these compounds make them particularly attractive for space-based applications. Previous studies (1-3) on the PFW-PT system have shown that, as the PT content increases, the temperature of the dielectric maximum increases and the relaxor ferroelectric behaviour gradually becomes ferroelectric. A maximum in dielectric permittivity was observed for compositions in the range of 0.20 < x < 0.37. Structural studies (1-3) revealed the presence of a morphotropic phase boundary (MPB) in the region 0.20 < x < 0.37, in which a rhombohedral (pseudocubic) and a tetragonal phase coexist. The relative amount of the phases in the MPB was found to be dependent on PT concentration and temperature. A detailed microstructural analysis of MPB 1-xPFW - xPT is presented using a combination of scanning electron microscopy (SEM) in - situ transmission electron microscopy (TEM) from room temperature down to 15K. The development of the grain structure is discussed and results indicate the existence of a core-shell microstructure. At 300K, the grains exhibit a core with 90 degree domains and no domain structure in the shell. With decreasing temperature, the domain structure within the core expands and the shell gradually shrinks. A 90 degree macrodomain structure in the whole grain is observed at 15K. The chemical composition of the core and shell has been analyzed by EDS. These microstructural features are discussed with respect to macroscopic properties. References: 1. L. Mitoseriu, P. M. Vilarinho, J. L. Baptista, Phase Coexistence in Pb(Fe2/3W1/3)O3 -PbTiO3 solid solutions, Appl. Phys. Lett., 80, 23, 4422-4424, 2002. 2. L. Mitoseriu, P. M. Vilarinho, M. Viviani, J. L. Baptista, Structural sudies of Pb(Fe2/3W1/3)O3 - PbTiO3 system, mat. Lett., 57, 3, 609 -614, 2003. 3. L. Feng., Z. - G. Ye, Phase diagram and phase transitions in the relaxor ferroelectric Pb(Fe2/3W1/3)O3 - PbTiO3 system, J. Solid State Chemistry, 163, 484 - 490, 2002.

## A5.62

**2D** InP etching simulation under high density plasma of chlorine. Ahmed Rhallabi, Bo Liu, Gregory Marcos and Jean Pierre Landesman; LPCM, IMN - CNRS, Nantes, France.

It is now evident that the improvement of the optical and electrical performances of the III-V components depends on the optimization of the critical process steps such as the dry etch processes especially for the submicron devices. The simulation of plasma surface interaction may widely contribute to the optimization of such process type. In the present study, a gas phase kinetic model of Cl2 plasma combined to 2D surface model is developed to predict the submicron etching profiles as a function of the plasma parameters. The gas phase kinetic model is based on the mass balance equations of reactive species diffusing toward the surface. The kinetic constants of electron impact reactions are established as a function of electron temperature assuming maxwellian distribution of electron energy. The additional equation of power balance in the ICP reactor allows to determine the electron temperature evolution with the plasma discharge parameters (Rf power, reactor pressure and the chlorine flow rate). One of the advantage of our model is the coupling between the plasma chemistry model and the surface etching model. The later is based on the calculating of the etch rate on each discretized surface element. The etching rate model uses the Langmuir surface concept which is based on the calculation of the covered surface fraction by GaClx sites. The direct fluxes of the reactive species such as Cl2+, Cl+ and Cl are determined from the gas phase kinetic model and introduced as the input parameters in the InP etching model. The simulation results show the role of different plasma parameters on the etched surface topographies through the mask. Some etching phenomena such as ARDE are analyzed

## A5.63

Layered molecular assemblies as interface isolators and adhesion enhancers for devices. P. G. Ganesan and G. Ramanath; Materials Science and Eng., Rensselaer Polytechnic Institute, Troy, New York.

Preserving the structural and functional integrity of interfaces is crucial to realize devices with nanoscale components. Hence, there is a need to explore new nanomaterials and processing strategies to isolate components from interdiffusion, mixing, and phase formation, and enhance interfacial adhesion. For instance, < 5-nm-thick conformal layers are needed to prevent diffusion and enhance adhesion at Cu/dielectric interfaces in sub-100-nm features of integrated circuits. Here, we demonstrate a completely new approach of using  $\sim 0.7$  to 5-nm-thick self-assembled molecular layers (SAMs) and polyelectrolyte layers to inhibit interfacial diffusion and enhance interfacial adhesion. The rationale of using molecular layers for these applications is based on anchoring the termini of the molecular constituents to the overlayers and underlayers through strong, but highly local, chemical interactions. We show that this strategy inhibits Cu diffusion and effects as much as ~10-fold increase in lifetimes of test capacitor structures and decreases leakage currents by ~6 orders of magnitude. Four-point bend tests of the Cu/SAM/dielectric structures show ∼3 times the debond energy of structures without SAMs. Systematic studies of diffusion and interfacial adhesion, combined with XPS analysis of fracture surfaces show that the nature of the terminal functional groups of the molecular structures are a critical factor determining the diffusion barrier and adhesion properties. In particular, strong bonding of Cu with thiols and carboxyl groups via covalent interactions or polychelation, or steric hindrance of aromatic groups contribute to Cu ion immobilization. These results are reinforced by the fact that there is no observable improvement in properties with increased barrier layer thickness. In fact, in many cases, the barrier and adhesion properties degrade with increasing thickness depending upon the terminal group in contact with the adjacent layers. Based on the above, we will present a model to explain important factors that influence interfacial diffusion and adhesion in Cu/molecular layer/dielectric structures.

### A5.64

Interface Adhesion and Reliability of Microsystem Packaging.

Marvin I Francis and Junghyun Cho; Mechanical Engineering, State
University of New York, Binghamton, New York.

Microsystem packaging requires a reliable and economic platform to meet with the challenges as the system becomes smaller. Since the packaging involves numerous interfaces among dissimilar materials, the reliability of the packaging system is often influenced by interfacial adhesion strength. The flip-chip packages, which have advantage for use in the microsystems due to a high-density interconnect capability, contain the organic underfill material to fill the space between the chip and the substrate. It can minimize the stresses developed between the underfill material and the adjoining components due to a CTE mismatch. Enhanced adhesion and integrity of the interfaces are, however, needed for long-term reliability that can be degraded under thermal and moisture exposure. This study focuses on interfacial failure phenomena between the underfill material and the chip as well as between the underfill material and the organic substrate by measuring interfacial toughness. For this purpose, we employ a linear elastic fracture mechanics (LEFM) approach that determines the critical strain energy release rate  $(G_c)$ . Numerical modeling and simulation will also be an integral part of this work to guide experimental efforts. Further, a depth-sensing indentation technique will be utilized to complement the interfacial toughness measurements as well as numerical simulation work by assessing the temperature-dependent properties of associated materials. In order to correlate microstructure and microchemistry of the interfaces with interfacial properties, atomic force microscope and electron microscopes will be employed. It is expected that a better understanding of microstructure as well as mechanistic evolution at the interfaces will further promote life extension and reliability of the microsystem packaging while providing affordable packaging processes.

## A5.65

Scaling of Microstructure Selection in Directional Solidification. Michael Greenwood and Nikolas Provatas; Materials Science and Engineering, McMaster University, Hamilton, Ontario, Canada.

The prediction of microstructure selection in solidification will play a vital role in the optimization of next-generation alloys. In this work we examine the fundamental nature of microstructure formation in directional solidification of binary alloys. We begin by re-visiting pattern selection in cellular growth in directional solidification experiments of binary alloys of pivalic acid (PVA) and succinonitrile (SCE). Various studies have characterized cell spacing versus pulling speed by fitting the cell wavelength to power laws of velocity over different growth ranges. We show that through suitable scaling, microstructure selection can be described in terms of a universal scaling function generic to the cellular growth regime. These results are confirmed by phase-field simulations of directional solidification, performed at experimentally relevant parameters through the use of a

recent multi-scale adaptive-mesh refinement technique. We characterize the details of the cellular scaling function, and examine its extension into the dendritic growth regime.

### A5.66

Controlled Field Emission From The Carbon Nanotubes Grown Over Cylindrical Surface. Debajyoti Sarangi and Ayat Karimi; EPFL, Lausanne, Switzerland.

Carbon nanotubes are excellent field emitters due to the high aspect ratios and small radii of curvature. These properties of the nanotubes offer tremendous possibilities for the realization of field emission devices. For the realization of the field emission devices controlled placement and alignment of the nanotubes over the substrate surface is desirable. Furthermore, field emission of the nanotubes in cylindrical geometry has added advantages over planar geometry. But, it is difficult to grow nanotube uniformly over non-planar substrate like long metallic cylinder by conventional CVD technique. In the present investigation we have adopted a very simple technique called as Cold Plasma Chemical Vapor Deposition (CP-CVD) to grow carbon nanotubes over the catalyst-supported metallic wire by resistive heating in hydrocarbon atmosphere at about  $700^{\circ}\mathrm{C}$ . The localized dissociation of the hydrocarbon gas over metallic wire allows well control growth of the nanotubes. The field emission performances of these nanotubes coated wires were carried out in a cylindrical geometry where the aluminum tube was used as anode. Emission site density is found to be the most important parameter to control the field emission performance. Comparisons of the field emission properties were made using nanotubes grown in different hydrocarbon gases like methane, acetylene and dimthylamine. The effect of other parameters like growth time, temperature, pressure, and catalysts were also investigated.

### A5.67 TRANSFERRED TO A13.3

### A5.68

Materials Integration for an Ultrananocrystalline Diamond-Based MEMS Technology, Jennifer E Gerbi<sup>1</sup>, James Birrell<sup>2,1</sup>, Jian Wang<sup>1</sup>, Xingcheng Xiao<sup>1</sup>, John A Carlisle<sup>1</sup> and Orlando Auciello<sup>1</sup>, <sup>1</sup>Materials Science, Argonne National Laboratory, Argonne, Illinois; <sup>2</sup>University of Illinois, Urbana-Champaign, Illinois.

Ultrananocrystalline diamond (UNCD) is a fine-grained (3-5nm), smooth (25 nm RMS roughness) phase-pure diamond material with abrupt high energy grain boundaries. Thin UNCD films have extremely attractive properties for an extensive range of MEMS devices: mechanical (high hardness and fracture strength); tribological (very low coefficient of friction and negligible stiction); electronic (high conductivity when doped); and chemical stability in corrosive environments. While the surface and bulk properties of UNCD are particularly well suited for fabricating MEMS structures themselves, the integration of UNCD with various materials is crucial for the realization of real-world MEMS devices such as resonators, RF switches and chemical sensors. Here, we discuss the processing of UNCD films to achieve integration with a selection of materials relevant to the fabrication of UNCD-MEMS devices. We will specifically detail the use of various seeding processes and growth chemistries for different substrates, including cross-sectional electron microscopy studies of the interface structure. We also discuss the fabrication and characterization of various metallic contacts and gate oxides on as-grown UNCD films, including the influence of post-deposition surface treatments. The impact of these results on both current and future applications of UNCD to MEMS devices will be presented. This work was supported by the US Department of Energy, BES-Materials Sciences, under Contract W-13-109-ENG-38.

## A5.69

Electrical Characterization of BCB for Electrostatic Microelectromechanical Devices. Alireza Modafe<sup>1,2</sup>, Nima Ghalichechian<sup>1,2</sup>, Benjamin Kleber<sup>1,2</sup> and Reza Ghodssi<sup>1,2</sup>; <sup>1</sup>Department of Electrical and Computer Engineering, University of Maryland, College Park, Maryland; <sup>2</sup>The Institute for Systems Research, University of Maryland, College Park, Maryland.

We report the results of electrical characterization of thick dry-etch benzocyclobutene (BCB) used as insulating dielectric film in electrostatic micromotors and microgenerators. The electrical efficiency of micromotors and microgenerators increases substantially using thick BCB instead of conventional silicon dioxide dielectrics. Reduction in stray capacitances due to low dielectric constant and large thickness, and increase in mechanical reliability due to low residual stress are main advantages of thick BCB. As an enabling tool for design and fabrication of BCB-based micromachines, we have developed a test scheme and an environmental test system that allow the study of correlation between electrical properties and design, process, and environmental parameters. The electrical characterization reported here consists of measuring the dielectric

constant, the I-V characteristic, and the effect of moisture uptake in the dielectric film. The developed test structures resemble the electrodes used in electrostatic micromachines and enhance moisture absorption in the dielectric film. The test structures are five interdigitated capacitors (IDCs) with 500 fingers, equal line width and spacing of 3 or  $4 \mu m$ , and finger length of 1 mm. The IDCs consist of metal thin films sandwiched between two layers of thick BCB (26  $\mu m$ for the first layer and 9  $\mu$ m for the second) deposited on a Pyrex wafer to minimize stray capacitances. The thickness of the BCB layers is not critical provided that it is bigger than the largest finger pitch of the IDC under test (8  $\mu$ m in this case). The dielectric constant is measured based on a geometry extraction method with two capacitance measurement steps before and after deposition of the second BCB layer. The average dielectric constant is found to be 2.830 (6.8% higher than the nominal value of 2.65) with a standard deviation of 0.028. The I-V characteristics are measured with stepping up the applied voltage in 10 V increments, holding the voltage for several minutes and measuring the current. The I-V characteristic of IDCs is measured after the samples have been exposed to room environment (23-25 degree C, 40-50 %RH) for several days. It is also measured after a 2-hour nitrogen bake at 200 degree C to drive the moisture out. After nitrogen bake, the leakage current decreased by one order of magnitude, and the breakdown voltage and the maximum current at thermal runaway are almost doubled. For the dry film, the dielectric strength is between 2 MV/cm to 3 MV/cm and the leakage current is several pA at voltages much lower than the breakdown voltage. This work demonstrates that the critical electrical properties of BCB vary drastically with process and environmental conditions. The characterization results reported here enable the optimization of design and fabrication processes to increase efficiency, reliability and robustness of BCB-based micromachines.

### A5.70

Investigation of Diamond Thin Films on Silicon Carbide for MEMS Applications. Scott E. Holmes and Ingrid St. Omer; Department of Electrical Engineering, Northern Arizona University, Flagstaff, Arizona.

MEMS devices are primarily fabricated from silicon due to the extensive process technology that has been developed. Unfortunately, silicon does not possess the mechanical or chemical attributes that are often demanded by harsh environments. Low young's modulus and Knoop hardness values indicate the mechanical limitations of silicon structures. Temperature sensitivity also complicates the suitability of silicon materials. Future MEMS devices need to be constructed from materials that possess the characteristics that will allow them to be used under the broadest of circumstances. Diamond and silicon carbide are two such materials whose properties prove to be most attractive. Both diamond and silicon carbide have enticing mechanical properties including high strength, and low friction coefficients. In addition, the materials are virtually chemically inert, thermally preferable, and potentially biocompatible according to preliminary studies. Unfortunately, those properties that make diamond and silicon carbide so attractive also make them extremely difficult to shape and utilize. Through the use of hot filament chemical vapor deposition, and photolithography processes diamond films have been deposited and patterned on 6H-silicon carbide to fabricate a MEMS cantilever structure. Physical and material properties of this structure are discussed, and the present stage of development is evaluated relative to standard silicon processes.

## A5.71

Combining Multivariate TOF-SIMS and SPM for Microscale Characterization of Microsystems Surfaces. Kevin R. Zavadil<sup>1</sup>, James A. Ohlhausen<sup>1</sup>, Diane E. Peebles<sup>1</sup>, Mathew G. Hankins<sup>2</sup> and Richard A. Plass<sup>2</sup>; <sup>1</sup>Materials and Process Sciences Center, Sandia National Laboratories, Albuquerque, New Mexico; <sup>2</sup>Microelectronics Science, Technology and Components Center, Sandia National Laboratories, Albuquerque, New Mexico.

Minimizing electrostatic bonding or stiction between closely spaced polycrystalline Si surfaces in surface micromachined (SMM) devices is typically accomplished by creating a low energy, low polarizability, and hydrophobic surface. Organosilane-based self-assembled monolayers (SAM) are used extensively to control surface properties of silicon. The long-term reliability of devices is a function of ensuring these SAM films are not significantly degraded in the packaged, operational environment. Our goal is to develop small length-scale characterization tools capable of providing physical or chemical signatures of degradation processes like SAM modification or displacement. Time-of-flight secondary ion mass spectrometry (TOF-SIMS) appears as a particularly valuable tool because it provides spatial resolution, surface sensitivity, and very high chemical information content. The key to successful application of TOF-SIMS is fully extracting this chemical information and we have developed multivariate techniques capable of accomplishing this task. We are currently investigating a combined approach of using high spatial

resolution scanning probe microscopy (SPM) and high chemical information content TOF-SIMS to characterize degradation mechanisms in these films. As a test case, we are exploring humidity and thermal degradation processes for a series of perfluorinated triand mono-dimethylaminosilanes on Si(100) and polycrystalline Si. TOF-SIMS analysis shows that subtle differences in fragmentation of the pendant chain can be detected for the SAM deposited onto occluded and line-of-sight regions on a polycrystalline Si surface. The source of this difference is, in part, a variation in co-adsorbed water at the oxide/SAM interface produced by mass transport constraints at the surface prior to and after SAM deposition. This result indicates that it may be possible to extract early stage chemical signatures of eventual SAM modification and displacement. We envision our approach as one component of a series of combined techniques that could be used to establish a surface chemical inventory in a microsystem. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

#### A5.72

Measurement of shape and deformation of MEMS at the wafer level. Cosme Furlong, Curtis F. Ferguson, Michael J. Melson and Ryszard J. Pryputniewicz; Mech. Eng. Laser Lab, WPI, Worcester, Massachusetts.

 ${\bf Microelectromechanical\ systems\ (MEMS)\ are\ integrated\ micron-sized}$ electrical and mechanical devices used for sensing, actuation, and control. MEMS structures are fabricated using very large-scale integration (VLSI) techniques adapted from those used in the microelectronics industry. Production of MEMS devices involves microfabrication technologies, e.g., surface micromachining, to define MEMS structures on single crystal silicon wafers, dicing of the wafers to separate MEMS structures, and packaging of the individual MEMS structures. Dicing and packaging of MEMS structures accounts for nearly 85% of the production costs. Therefore, identification of defects at the wafer level will result in improved production yield. Testing of MEMS at the wafer level includes measuring electrical, optical, and mechanical responses to electrical signals and environmental conditions. In this paper, optoelectronic holography (OEH) methods for nondestructive and noninvasive testing of MEMS components at the wafer level are described. Using OEH, quantitative measurements of shape and deformation are obtained with sub-micrometer spatial resolution and nanometer measuring accuracy. To facilitate evaluation of MEMS at the wafer level, the OEH methods incorporate: (1) imaging systems based on high-spatial, high-frame rate, and high-digital resolution cameras, (2) advanced nanometer resolution positioning stages and piezoelectric devices, (3) automated, high-resolution, computer controlled positioning and data acquisition systems, (4) computer controlled instrumentation for loading and controlling of MEMS samples, (5) analysis software capable to provide quantitative and full-field of view shape and deformation data in form of 3D information, and (6) routines to export quantitative 3D shape and deformation information into advanced computer-aided design (CAD) software packages. To inspect the entire wafer with OEH methods, measurements of overlapping regions of interest (ROI) on the wafer are recorded and adjacent ROIs are stitched together through correlation analysis. Capabilities of the OEH methods are illustrated with representative applications, including determination of optimal inspection conditions to minimize inspection time while achieving sufficient levels of accuracy and resolution.

## A5.73

Advanced confocal optoelectronic microscopy for materials and structural characterization of MEMS. Cosme Furlong, Peter Hefti and Ryszard J. Pryputniewicz; Mech. Eng. Laser Lab, WPI, Worcester, Massachusetts.

Surface micromachined MEMS are fabricated using lithographic processes and sequential deposition of thin film materials, which define three-dimensional microstructures containing nanometer size geometrical features. Effective study and optimization of MEMS depend on high accuracy and resolution three-dimensional measurements of, both, their absolute shape and changes in shape due to operational and loading conditions. Confocal microscopy (CM) is capable to accurately measure surface features of MEMS and changes in these features. Using CM, structures are imaged layer-by-layer and a three-dimensional representation is build up by synthesizing and stacking individually recorded layers of images, with each image recorded at the best focal plane and requiring specific image processing and quantification. Accuracy and resolution of CM measurements is highly dependent on resolution and repeatability of positioning devices used for image acquisition, spatial resolution of the imaging device, confocal optics, image processing algorithms used for quantifying individual images, as well as on the algorithms used for synthesis and stacking of imaging data. In this paper, a combined confocal and optoelectronic methodology for characterizing shape and

changes in shape of MEMS is presented. The methodology builds on the capabilities of confocal microscopy to resolve features at the best focal plane, and the capabilities of optoelectronic methodology to provide high-resolution measurements at the best focal plane, as defined by the confocal and optical instrumentation. It is demonstrated that the combined confocal and optoelectronic methodology is able to structurally characterize MEMS with high accuracy and resolution. Most importantly, this characterization is capable to include large discontinuity ranges, which are found in high aspect ratio (HAR) MEMS. Application of the methodology to study structural uniformity of MEMS demonstrates applicability of the advanced confocal optoelectronic microscopy to study and optimization of the microdevices.

### A5.74

Thermal Conductivity of Ultrananocrystalline Diamond Films. Maki A Angadi, John Carlisle, Jeff Eastman and Orlando Auciello; Materials Science Div, Argonne National Lab, Argonne, Illinois

The study of thermal transport in nanostructured materials offers the opportunity to study what happens when the wavelength of thermally active phonons is comparable to the grain size. In this presentation we will discuss initial experimental results on thermal conductivity measurements performed on ultrananocrystalline diamond (UNCD) films deposited on Si wafers. Samples were prepared by microwave plasma chemical vapor deposition using argon-rich Ar/CH<sub>4</sub>plasma chemistries, and consist of 3-5 nm diamond grains and 0.5 nm wide high energy, high angle grain boundaries. Thermal conductivity was measured on UNCD films in the thickness range 1-7.5  $\mu m$  and the temperature range 200K-400K using the 3- $\Omega$  technique. We have studied thermal conductivity in UNCD films as a function of (a) film thickness (b) temperature and (c) and hydrogen and nitrogen doping. Our results show that the thermal conductivity of UNCD films shows a strong dependence on nitrogen and hydrogen doping levels. The effects of grain size and film thickness on thermal conductivity will also be described. The implications of these results for the thermal stability of UNCD MEMS devices will be discussed. Comparisons will be made with thermal transport in polycrystalline and single-crystal diamond. This work was supported by the US Department of Energy, BES-Materials Sciences, under contract W-13-109-ENG-38

### A5.75

Evolution of Mesas on Si(111) Surface Under Sublimation.
Kee-Chul Chang and Jack Blakely; Materials Science & Eng., Cornell University, Ithaca, New York.

How the surface morphology of a low index crystal surface evolves during annealing below the roughening temperature is still a open question that is being investigated both theoretically and experimentally. This research is needed to understand the stability of nanostructures and to devise methods to produce controlled atomic step arrays which may have application in technological applications. We have approached this problem from an experimental point of view by studying at the step distributions on the tops of mesa patterned on Si(111) after flashing at 1200C and annealing between 1000-1100C We find that after the annealing, nanometer height ridges develop on the edges of our mesa pattern except for one edge which becomes a source for steps advancing into the mesa top. The edge that does not develop a ridge is mainly determined by the initial miscut of the surface. The results have relevance to the formation of quantum dot arrays on patterned substrates. We will attempt to explain our results through step dynamics and present some applications of this phenomena in creating novel patterns of steps for preferential growth and adsorption.

## A5.76

Fabrication and integration of nanomechanical disk resonators for RF range oscillating circuitry. Stephane Evoy, T.R. Jaruhar, P. Baude, M.L. Cohen and J. Li; Electrical Engineering, Univ. of Pennsylvania, Philadelphia, Pennsylvania.

Off-chip filtering and resonating elements currently preclude the complete single-chip integration of wireless systems. Electromechanical resonators have therefore been proposed as on-chip replacements of such devices. Prototype nanoelectromechanical resonators with resonant frequencies reaching the GHz range have been fabricated in silicon, SiC, GaAs and other materials. However, as result of poor clamping and structural issues, these RF range nanomechanical structures typically offer insufficient resonance quality for filtering applications. We report on the fabrication and characterization of nanomechanical disk resonators in single-crystal silicon. This disk design offers access to various higher-order modes of vibration that minimizes stresses at the clamping point. We will present finite element analysis of these disks predicting high-quality resonance modes reaching the GHz range even in micron-size disk diameters. A first generation of resonators has been successfully

fabricated in silicon-on-insulator layers, and resonance in the low MHz range has been observed. However, the highly-slanted nature of the support stubs induces the resonant flexion of the support structure rather than the true oscillation of the Si disk. Our second generation will rather involve the undercut etching in a dry environment to improve the structural integrity of the stub. We have also performed the full electromechanical modeling of these disks in support of the design of electronic circuits embedding these disks. Specifically, we report the design of a Pierce oscillator that integrates these disks as filtering feedback, as well as of integrated monitoring circuits for disk-based sensing. We have demonstrated the simulated oscillation of such electromechanical circuits at frequencies reaching 100 MHz.

### A5.77

Application of FIB/SEM and TEM to Single Bit Failure Analysis in SRAM arrays. Wentao Qin, Alex Volinsky, Larry Rice and David Theodore; Process and Materials Characterization Lab, Motorola, Tempe, Arizona.

Many microelectronic chips contain embedded memory arrays. A single SRAM bit-cell contains several transistors. Failure of any of the transistors makes the entire bit-cell inoperable. Dual-beam Focused Ion Beam (FIB) combines the slicing capability of FIB with in-situ SEM imaging. The combination offers unparalleled precision in looking for root causes of failures in microelectronic devices. Once a failure site has been located, an FIB lift-off method can be used to prepare a TEM sample containing the area of interest. Further structural, elemental and chemical bonding information can then be acquired from the failure site. We report here, analyses of single-bit failures in SRAM arrays carried out using FIB/SEM followed by TEM imaging and EDS/PEELS. Root causes of bit failures including remnant seed-layer metal between stacked vias have been identified.

### A5.78

Study of high aspect ratio etching in silicon by a mixture of SF6/O2 using a 2-D model based on a Monte Carlo approach. Ahmed Rhallabi<sup>1</sup>, Gregory Marcos<sup>1</sup> and Pierre Ranson<sup>2</sup>; <sup>1</sup>LPCM, IMN - CNRS, Nantes, France; <sup>2</sup>GREMI, ESPEO - CNRS, Orleans, France.

For the new microelectronic applications such as MEMS, the industry of plasma etching must succeed significant challenges. In particular, the problem concerns the control of kinetics and shape of structures. In GREMI laboratory [1], studies in dry etching processes have been performed in obtaining deep silicon trenches by using a cryogenic method in an Inductive Coupled Plasma reactor with an SF6/O2 plasma discharge. The experiments have shown that undercut and bowing formation is strongly correlated with plasma parameters, mask shape and time during processing. In order to understand the involved plasma-surface interaction mechanisms, we have developed a two dimensional etching model based on a Monte Carlo approach. This etching simulator includes different surface processes due to neutral reactive species (fluorine and the atomic oxygen). Their flux is assumed to be isotropic. A transport model through the RF sheath is connected with the surface model to calculate angular and energetic ion function distribution. With Monte-Carlo techniques, the physical processes can be introduced with probabilistic considerations adsorption/desorption, spontaneous chemical etching, ion preferential sputtering, incident species reflection, passivation layer formation and redeposition. Kinetic parameters of the plasma phase are introduced as input data obtained by in-situ measurements. The etched substrate is discretized by a series of uniform square cells which size defines a real number of silicon atoms. Local surface displacement is modelled by "full" cells dis/re-appearance when an etching or redeposition process occurs. This method gives an instantaneous picture of surface state during the process. In particular, it permits to follow the F/Si and O/Si surface coverage on the sidewalls versus depth and time These information are useful to understand the transport of species in the trench and their role on the ARDE characteristics. Defects as roughness, microtrenching and faceting are studied and novel hypotheses are given to explain their formation.

## A5.79

Physical Mechanism of SOI MOSFET Photodetector with a Nanometer Scale Wire. Hong Goo Choi, Young Chang Jo and Hoon Kim; NANO Scale Quantum device research center, Korea Electronics Technology Institute, PyungTaek-Si, KyoungGi-Do, South Korea.

Photodetectors are of considerable importance as image processing units. Especially, for mobile applications the photodetector, which can be operated under low power and can be easily downscaled, is more needed. In this aspect MOSFET in its lateral bipolar mode has been considered a possible candidate instead of conventional photodiode. We compare the MOSFET photodetector with and without a wire in their power consumption and high integration and the mechanism of optical characteristics of the device with the wire is investigated. SOI

MOSFETs used in the experiments were fabricated using p-type <100> SIMOX wafers with a top silicon layer thickness of 190nm. By electron beam lithography and re-oxidation a nanometer scale wire is obtained. Optical SOI MOSFETs were of a fully depleted type and characteristics of the devices were measured using He-Ne laser(wavelength=632.8 nm). In the conventional SOI MOSFET without the narrow channel, both photocurrent and sensitivity increased with the intensity of light and sub nA of dark current was detected. This device is not easily downscaled because of the increase of leakage current caused by short channel effect. On the contrary, in SOI MOSFET with the wire, the highest sensitivity to light for weak light and abnormal increase in photocurrent were found. At higher intensity of light, the abnormality occurred at an earlier time. Because similar results were observed for several other devices, we believe these phenomena reflect a device structure resulting from the wire. The physical mechanism of the operation of our device can be interpreted as hole accumulation model and lateral bipolar action. We believe that photodetector with the wire is easy to be downscaled and to operate under low power of 0.1V because that it is controlled only by the wire region and forms a MOSFET-type.

### A5.80

Advanced Anodic Bonding Processes for MEMS Applications. Viorel Dragoi<sup>1</sup>, Paul Lindner<sup>1</sup>, Thomas Glinsner<sup>1</sup>, Markus

Wimplinger<sup>2</sup> and Sharon Farrens<sup>2</sup>; <sup>1</sup>Technology, EV Group, Schaerding, Austria; <sup>2</sup>Technology, EV Group Inc, Phoenix, Arizona.

Due to the specific characteristics of MEMS devices (3D architectures, containing moving parts, controlled atmosphere encapsulation, etc.) the device packaging has very specific constraints. Wafer bonding is one of the most powerful techniques used for MEMS devices fabrication and packaging. Different wafer bonding approaches are used in the MEMS industry: fusion, adhesive, eutectic, thermocompression bonding are normally used for device fabrication and generation of 3D structures, while anodic bonding is one of the most used wafer level packaging procedures. Among the wafer bonding techniques, anodic bonding is a very mature technology. Anodic bonding is used today mainly to bond a silicon wafer to a glass wafer with high content of alkali oxides. The bond occurs under heating conditions in an applied electric field due to the fact that at a certain temperature depending on the glass composition the oxides dissociate and the mobile alkali ions are driven by the electric field into the glass, creating an oxygen rich layer at the silicon-glass interface. The oxygen ions will be driven by the electric field to the silicon surface and will produce oxidation of Si. The resulting bond strength is very high and the process is irreversible. Some MEMS applications like accelerometers, gyros and microfluidic devices are using two anodic bonding processes in order to obtain a 3 wafers structure (triple-stack). The present paper describes a process developed for the fabrication of triple-stacks using a single anodic bonding step. The process conditions for Si-Glass-Si and Glass-Si-Glass triple-stack bonding will be detailed. Compared with the approach using two anodic bonding steps, the advantages offered by the single process are the secure packaging of double side processed wafers with minimum contamination risk by using only half of handling steps and increased throughput.

## A5.81

Characterization of a cross-junction of multi-walled carbon nanotubes fabricated by using nanomanipulators. Seongchu Lim<sup>1</sup>, KeunSoo Kim<sup>1</sup>, DongJae Bae<sup>1</sup>, YoungHee Lee<sup>1</sup>, Shinje Cho<sup>2</sup> and Jae-Eun Yoo<sup>2</sup>; <sup>1</sup>Center for Nanotubes and Nanostructured Composites, Suwon, South Korea; <sup>2</sup>Nanotechnology Center, Iljin Nanotech, Seoul, South Korea.

We have installed two nanomanipulators inside field emission-scanning electron microscope (FE-SEM). Using these manipulators, we are able to precisely move, position, and even integrate an each individual multi-walled carbon nanotube (MWCNT) into a desired form of device. For example, we have fabricated a cross-junction of multiwalled carbon nanotubes (MWCNTs) using nanomanipulators. The detailed properties of the junction will be discussed together with other characteristic studies.

## A5.82

Mechanical Characterizations of Laser Microwelds for MEMS Packaging. Wei Han and Ryszard Pryputniewicz; Mechanical Engineering, Worcester Polytechnic Institute, Worcester, Massachusetts.

Laser micromachining has proven to be a very powerful and successful tool for precision machining and microfabrication with applications in electronics, MEMS, medical, and biomedical fields. For bonding techniques required in MEMS fabrication and packaging, several approaches based on localized heating and bonding have been proposed such as localized eutectic bonding, fusion bonding, solder bonding, and chemical vapor deposition (CVD) bonding. However,

these approaches require resistive heating which result in intimate contact at the electrodes, and this is not preferred for MEMS packaging applications. As a good alternative, laser microwelding has advantages of non contact, low heat distortion, high speed, high precision, and consistent weld integrity. Therefore, it is finding increasing use in MEMS fabrication and packaging applications. In laser microwelding, the material in the HAZ of the workpiece experiences heating, melting, and re-solidifying stages, and the final characterizations of the laser microwelds can be influenced by various factors such as the laser beam properties, the system cooling condition, and the surface roughness/reflectivity of the component. This paper presents characterization of laser microwelds, with emphasis on study of deformation and strength of the microwelds made by a Nd:YAG pulsed laser. Optical interferometry is used to nondestructively record fringe patterns of the shape/deformation of the laser microwelded workpiece before, during, and after the microwelding process, and tensile tests are performed to study the strength of the laser microwelded workpiece as the function of different welding parameters. Furthermore, the material properties of the laser microwelds are determined by nanoindentation. Results indicate that quality of the laser microwelds depends on selection of an appropriate set of parameters controlling the microwelding process.

## A5.83

Electrochemical Synthesis, Design Concept and Application of Nano-Patterned Alumina Bio-Interface. Paul Takhistov, Rutgers University, New Brunswick, New Jersey.

Nano-structured bio-substrate high surface area materials with nano-scale dimensions can be attained by creating materials where the void surface area (pores) is high compared to the amount of bulk support material. Increased surface area of a nano-structured material leads to the increasing of the surface chemical reaction rate and intensification of the electron transfer (i.e. biosensor's output signal) through the solid-liquid interface. Furthermore, ordered nano-scale structure of a surface substrate stimulates ordering and self-assembly of deposited specific biological components at the molecular level. Template based electrochemical synthesis techniques appear to be particularly attractive since they are take advantage of the self-patterning of natural systems, where the biointeracce material is synthesized in the size and shape of the desired nano-structure. An ideal template for biosensor applications should provide: (a) good surface constraints/features size control and periodicity, (b) good structural and mechanical integrity, (c) chemically inert material for a wide variety of deposited bioactive materials, (d) easily tailored template size parameters, (e) cost-effective fabrication process and (f) high transparency over a wide range of the media matrix. The use of anodized aluminum substrate for bio-applications (porous membranes, nano-electrodes, biosensors, etc. is discussed. Fabrication of the thin film alumina template in proposed fabrication technique is based on anodization, or an electrolytic oxidation, of aluminum into porous alumina. During this process a porous film of alumina is formed characterized by the ordered pore structure that follows 2-D lattice. Microorganisms-surface interactions can be described in terms of the physicochemical processes that are associated with bacterial adhesion and biofilm development. Surfaces with nano-scale patterns, such as aluminum oxide are of special interest in this case. The interaction of cell membrane's protein receptors with nano-scale surface constraints could cause the resonance-type effect in bacterial adhesion dynamics. This interaction is a very complex process including non-linear interstitial liquid film instability, transport phenomena at nano-scale and biochemical reactions. Information about the specific impact of the surface topography on the dynamics and efficiency of bacteria-surface interactions delivers the new fundamental knowledge about the biofilm development. This fundamental issue is extremely important for the biological warfare defense, biosecurity and food safety, since it allows developing the new approach to the diseases prevention and pathogens inactivation.

## A5.84

Formation of a BaTe Surface on GaAs. Kevin A Boulais, Francisco Santiago and Karen Long; Systems Research and Technology Department, Dahlgren Division Naval Surface Warfare Center, Dahlgren, Virginia.

The formation of a BaTe surface on GaAs has been investigated. The surface was created using molecular beam epitaxy. A GaAs (100) surface was first exposed to Te and was characterized using x-ray photoelectron spectroscopy (XPS), reflective high energy electron diffraction (RHEED) and low energy electron diffraction (LEED). The Te-reacted surface was then exposed to BaF2 where a second reaction occurred. In this reaction, the BaF2 molecule was broken leaving Ba at the surface. No fluorine has ever been observed on the surface following the Te treatment as determined by XPS. This is in contrast to the clean GaAs (100) surface in which BaF2 has been shown to grow epitaxially. Although high order exists in the early stage of the growth, further exposure to the BaF2 flux eventually gives way to a

polycrystalline form of a BaTe surface. It has been suggested that a chalcogenated GaAs surface can be made stable to air by exposure to a metal that forms a low solubility chalcogenide. The results of our work will be presented.

## A5.85

Characterization of Encapsulated PC12 Neurosecretory Cells and Mass Transfer Studies of Neurotransmitter/Neurotrophic Factor Transport on Silicon Nanoporous Membranes. Carlos Alejandro Lopez and Tejal Ashwin Desai; Biomedical Engineering, Boston University, Boston, Massachusetts.

Current therapies for neuronal pathologies are based mainly on pharmaceutical treatment with drugs. With the use of micro and nanotechnology, it has been possible to create devices which may provide a more permanent and successful treatment method for diseases resulting from the loss of specific hormonal and biochemical expression. These technologies have provided us the means to build structures which can promote cellular attachment, proliferation, differentiation, and long-term survival. Also, the physical characteristics of these devices can be controlled to allow effective immunoisolation and the incorporation of activating components for the controlled release of desired cellular compounds. Silicon-based devices are easily manufactured in bulk, can be easily modified, and are known to be biocompatible because of the unique properties of these materials. Research is being conducted on the integration of neuronal cell lines and photolithographically-created silicon nanoporous biocapsules. Neuronal attachment, differentiation, and functionality are all being investigated on these silicon capsules through various biocompatibility studies. The release rate of neurotransmitters from the biocapsules loaded with PC12 cells and the concentrations obtainable immediately outside the nanoporous membrane are important parameters being investigated in these novel devices. Characterization of release from the biocapsules will be presented with respect to the cell line and the cell number/concentration loaded, as well as the pore size of the nanoporous membrane. The ability of biocapsules to have adequate influx of necessary cytokines and growth factors to stimulate cellular response will also be discussed. Peptide concentrations will be controlled and modeled at physiological levels to maintain biocapsule functionality as in in vivo implantation. Long-term studies will include in vivo implantation and monitoring of biocapsules loaded with PC12 cells in an animal model as well as in vitro studies of the effects of encapsulated cell stimulation of extra-capsular neuronal cultures.

### A5.86

Pull-In Voltage and Frequency Analysis on MEMS Parallel Plate Electrostatic Actuators. Emmanuel Saucedo-Flores, Ruben Ruelas and Martin Flores-Martinez; Ingenieria de Proyectos, Universidad de Guadalajara, Zapopan, Jalisco, Mexico.

Key words: MEMS, modeling, actuators, Simulink. Abstract. This paper describes a modeling tool developed to make fast parameter design analysis on MEMS parallel plate electrostatic actuators. A user friendly Matlab-Simulink interface was implemented to easily introduce material parameter values and observe their influence on the electro-mechanical system behavior. The tool is based on solving the differential equation describing the free electrode displacement for different voltage source loads. It takes into account the stiffening spring and the resonant frequency modification effects. The nowadays classical pull-in voltage or electrostatic instability and the resonant frequency shifting phenomenon were modeled for various electrode plate and gap materials and geometries.

## A5.87

Electrodeposited GMR Multilayer Thin Films, Nanowires and Micro-Posts. Elizabeth J Podlaha<sup>1</sup>, Yutong Li<sup>1</sup>, Qiang Huang<sup>1</sup>, Erick Lawson<sup>2</sup>, Julia Y Chan<sup>2</sup>, Jianqi Zhang<sup>1</sup>, Monica Moldovan<sup>3</sup>, David Young<sup>3</sup>, Dinakar Palaparti<sup>4</sup> and Michael C Murphy<sup>4</sup>; <sup>1</sup>Chemical Engineering, Louisiana State University, Baton Rouge, Louisiana; <sup>2</sup>Chemistry, Louisiana State University, Baton Rouge, Louisiana; <sup>3</sup>Physics, Louisiana State University, Baton Rouge, Louisiana; <sup>4</sup>Mechanical Engineering, Louisiana State University, Baton Rouge, Louisiana, Louisi

Iron-group(Co, Ni, Fe)/Cu nanoscaled multilayer alloys were electrodeposited with different layer thicknesses and electrolyte constituents to map out the governing features affecting magnetoresistance in electrodeposited thin films. Giant magnetoresistance (GMR) in excess of 20% was achieved in CoNiFe/Cu and Co/Cu multilayers, from a single electrolyte, containing both the iron-group elements and copper with pulsed current deposition. The GMR value was dependent on the deposit grain size, intensity ratio of the (200)/(111) XRD reflection peaks, layer sizes and purity of the magnetic layer. The advantage of the electrodeposition process over other vapor techniques is that deposits can be readily fabricated into recessed geometries. Examples of alloy

deposition into nanowires and micrometer-size deep recesses are presented. NiFe/Cu multilayers electrodeposited into 500 mm deep recessed posts, were additionally examined for MEMS micro-molds. The coefficient of thermal expansion was lowered in deposits with Cu nanolayers compared to an Invar-type, unlayered NiFe deposit.

### A5.88

Carbon nanotube coatings for thermal control.

Jennifer L Sample, Keith Rebello and Robert Osiander; Johns

Hopkins University Applied Physics Laboratory, Laurel, Maryland.

The high thermal conductivity combined with nanoscale dimensions and high aspect ratios of carbon nanotubes allow generation of new materials with thermal transport features controlled at the nanoscale. Carbon nanotubes provide innovative options for thermal control applications such as improved thermal interfaces, allowing development of thermal switches with low power and weight. We report growth and coating of carbon nanotube arrays onto compliant, flexible surfaces, and improved thermal transport across coated interfaces. We investigate CVD grown single and multiwall nanotube arrays as well as coatings employing self-assembly for patterned deposition.

## A5.89

**Development of a MEMS Temperature Sensor.** <u>Houri Johari</u> and Ryszard J. Pryputniewicz; Mechanical Engineering, Worcester Polytechnic Institute, Worcester, Massachusetts.

Advances in process control technology depend on ability to measure local temperature. In a number of applications, this ability increases as the size of temperature sensors decreases. Recent breakthroughs in microelectromechanical systems (MEMS) fabrication methodologies have led to development of very small temperature sensors, size of which depends on materials used and their properties. This paper presents development of a MEMS temperature sensor to be made of polysilicon. Polysilicon is a material with grains having a wide distribution of sizes and irregular shapes. Relationships between grain boundaries and electrical properties of doped polysilicon are governed by: 1) dopant-segregation model and 2) carrier-trapping model, used in this paper. Grain structure, size, and orientation of polysilicon films depend on deposition parameters, subsequent doping, and process conditions. High deposition temperatures and high annealing temperatures can result in large grains. Also, electrical resistivity of polysilicon decreases with increasing doping concentrations. For doping doses from  $10^{13}$  atoms/cm $^2$  to  $10^{14}$  atoms/cm $^2$ , large negative thermal coefficients of resistance (TCR) are obtained. In this paper, a computer code was specially developed to determine TCR and resistivity as functions of doping concentrations and grain size. A good sensor should have high sensitivity (related to high TCR), good linearity, resistivity on the order of a  $k\Omega$ , and more than 50 grains. If the deposition and annealing temperatures are in the ranges 700°C to 800°C and 950°C to 1100°C, respectively, then grain size of about  $200 \text{\AA}$  can be achieved. Also, resistivity of 14  $\Omega$ -cm and TCR of 1.4%/°C can be obtained for the temperature sensor characterized by the length, width, and thickness of 1.5  $\mu$ m, 1.5  $\mu$ m, and 0.5  $\mu$ m, respectively. Procedures for determination of parameters controlling functional operation of a MEMS temperature sensor are discussed and illustrated with specific examples.

## A5.90

Electrochemical nitric oxide sensor using carbon nanotubes.

<u>Cuiying Wang</u>, Shaoming Huang, Jie Liu, Claude Piantadosi and

Barry Allen; Chemistry Department, Duke University, Durham, North

Carolina

Electrochemical nitric oxide sensor using carbon nanotubes Cuiying Wang, Shaoming Huang, Jie Liu Department of Chemistry, Duke University, Durham, North Carolina Claude Piantadosi, Barry Allen Duke University Medical Center, Durham, North Carolina A novel electrochemical sensor was fabricated for detecting the biologically-important molecule nitric oxide (NO). The new sensor combines the very large surface area of aligned multi-walled carbon nanotubes (CNTs) with the ability of ruthenium (Ru) to catalyze the oxidation of NO. The aligned carbon nanotubes were grown on silica and then suspended in 5% Nafion(NAF) solution and coated on Pt Chemical vapor deposition (CVD) was used to deposit Ru on the surface of the CNT-coated Pt wire. The new electrode has the following characteristics: 1. The Pt/NAF/CNT/Ru electrode is much more sensitive for NO oxidation at + 675 mV vs. Ag/AgCl than bare Pt and than Pt coated only with carbon nanotubes. We hypothesize that the CNT increase the specific surface area of the electrode and that Ru facilitates NO oxidation by forming nitrosyl groups(M-NO). 2. The current density of the new sensor is 10 times that of bare Pt at 1.5 mM NO. At 0.15 mM NO, the current density is approximately 50 times that of bare Pt, likely due to the fact that the half-life of NO increases as its concentration decreases. 3. The new electrode also detected NO at potentials well below + 675 mV. This is very useful in biological studies, since less extreme potentials result in the electrolysis of fewer interfering substances, whose oxidation signals could be confounded with that of NO.

#### A5.91

Resist Poisoning-Free Advanced PECVD-Based Antireflective Coating(ARC) for 90nm Technology and Beyond. Sang H. Ahn, Sudha S Rathi, Jean Liu, Heraldo Botelho, Wendy Yeh, Martin J Seamons and Hichem M'Saad; Dielectric Systems and Modules, Applied Materials Inc., Santa Clara, California.

A nitrogen-free (N-free) dielectric anti-reflective coating (DARC®) was cost-effectively developed in a plasma-enhanced chemical vapor deposition (PECVD) reactor to eliminate the 193nm resist poisoning interaction caused when N2O is used as a precursor [1]. Although it was found that even a N-free ARC could poison sensitive 193nm resists with -OH radicals [2], which either exist inherently in the ARC or result from H<sub>2</sub>O absorption by the ARC surface, the current investigation has revealed that it was possible to eliminate resist poisoning. The ARC surface acidification circumvents the neutralization of photo-generated H<sup>+</sup> in 193nm resist by -OH radicals inherently in the ARC while the dense ARC film makes it difficult for -OH radicals to form on the ARC surface due to  $H_2O$ absorption. Compressive film stress directly correlates to H2O resistance. It was therefore possible to greatly improve the ARC resistance to  ${\rm H}_2{\rm O}$  absorption by creating and maintaining a process regime that makes the ARC film dense. This dense ARC with an acidified surface demonstrated promising lithography performance with minimal resist poisoning as well as excellent shelf life and O<sub>2</sub>-ashing resistance. This paper explores the N-free DARC material, its development, lithographic integration results and implementation in a production environment to eliminate 193nm resist poisoning.

> SESSION A6: Mechanical Properties I Chair: Arturo Ayon Tuesday Morning, December 2, 2003 Room 200 (Hynes)

## 8:30 AM \*A6.1

Processing Issues and Material Properties in Ni and Ni-Fe plating in High Aspect Ratio Micro Structure Technology (LIGA). Klaus Bade, Achim Janssen and Joachim Schulz; IMT, Forschungszentrum Karlsruhe, Karlsruhe, Germany.

Micro-electroplating is an important process step in the LIGA process for the fabrication of metallic micro components and for molding tools used in replication of polymeric micro parts. The influence of the micro-electroplating conditions on the formation of the material will be discussed in terms of simple models from electrodeposition. With respect to LIGA and LIGA-like applications, several requirements arise concerning for example life time or geometric tolerance aspects. Material strength and hardness are typical parameters used to characterize life time of electroplated mold inserts. Residual stress is an important factor during material deposition which may significantly influence the geometry. Both properties are well known to the electroplating community, but the special aspects of micro technology forces us to review these results. First steps in order to better understand electroplating in micropatterns and to understand the resulting material properties will be presented. The strength of electroformed Nickel-Iron alloys was characterized by hardness as well as in tensile tests in the deposited state and after additional thermal treatment. The latter was studied to understand the behavior and possible failure mechanisms of the material during hot embossing. At annealing temperatures below 620 K a hardness increase was found. In contrast electroplated Nickel shows a hardness loss at temperatures above 473 K. The role of mechanical stresses during plating will be shown for the standard nickel electroforming under conditions for obtaining molding tools by LIGA for the replication of the micro pattern into polymers. For these large area depositions the flatness of the mould insert is influenced in a sensitive way by stress growth. A deflection method can be used for process monitoring to maintain a sensible process with the stress value being kept within the required

## 9:00 AM <u>A6.2</u>

Thermophysical Properties of Ni Films for LIGA

Microsystems. Rajesh Nimbalkar<sup>1</sup>, <u>Theodorian Borca-Tasciuc</u><sup>1</sup>, Samuel Graham<sup>2</sup>, Diana-Andra Borca-Tasciuc<sup>1,3</sup> and Gang Chen<sup>4</sup>; <sup>1</sup>Mechanical, Aerospace, and Nuclear Engineering, Rensselaer Polytechnic Institute, Troy, New York; <sup>2</sup>Microsystems and Materials Mechanics, Sandia National Laboratories, Livermore, California; <sup>3</sup>Mechanical and Aerospace Engineering, University of California at Los Angeles, Los Angeles, California; <sup>4</sup>Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

This work reports temperature dependent thermophysical properties

characterization of electrodeposited nickel sulfamate, nickel with saccharin, and nickel with manganese additions. These materials are intended to use in LIGA microsystems. Anisotropic thermal diffusivity measurements are performed using a photothermoelectric technique. A steady state method is used to determine the in-plane thermal conductivity. Thermal properties of as grown samples are compared with those of temperature annealed specimens. The results are correlated with scanning electron microscopy studies of the grain structure and measurements of the electrical transport properties.

## 9:15 AM A6.3

Mechanical Properties of Metallic Thin Films: Tensile Tests vs. Indentation Tests. Wei Tong, Nian Zhang and Changjin Xie; Yale University, New Haven, Connecticut.

Metallic thin films are widely used in many microelectronic and MEMS applications. The mechanical properties of these thin films are commonly evaluated via various instrumented indentation techniques, as direct tensile testing is rather difficult if not impossible for thin films. The existing interpretations of indentation test data (either theoretical or numerical approaches) have been largely based on isotropic polycrystalline material models while most of the metallic thin films are strongly textured with a few grains or a single grain running through the thickness of the film. The multicrystalline nature of the thin films on their indentation and tensile properties is the focus of our investigation. Using multicrystalline aluminum and copper alloys as model material systems, both tensile tests and indentation tests are performed and the testing results are compared based on a crystal plasticity finite element analysis. The conventional correlation between the indentation data and the tensile test data (at an effective or equivalent strain) is critically examined for these two multicrystalline materials.

## 9:30 AM <u>A6.4</u>

SiGe Relaxation on Silicon-on-Insulator Substrates.

Michelle M Roberts, B Yang, D E Savage and M G Lagally;
University of Wisconsin-Madison, Madison, Wisconsin.

We have observed the early stages of strain relaxation of SiGe films on silicon-on-insulator (SOI) substrates in real time using low-energy electron microscopy (LEEM). SiGe films on SOI are becoming increasingly important for high-speed low-power devices. For this film system to be useful, it is necessary to control the strain and dislocation driven strain relaxation. While misfit dislocations at the film interface are necessary for relaxation, threads through the SiGe film destroy device performance. Understanding how the buried oxide influences dislocation motion in the system is key in controlling relaxation of the SiGe film. We have grown SiGe alloys on bulk Si(100) and on SOI with different Ge compositions and Si template layer thickness by chemical vapor deposition. The SiGe film thickness was chosen to be below the critical thickness for the same SiGe alloy grown on bulk Si. Recent models suggest that a reduction in the line energy of the dislocation at the Si/oxide interface allows the system to relax with a reduced threading dislocation density.1,2 If there is a reduction in line energy at the Si/oxide interface, the dislocations at the Si/oxide interface should be able to extend for thinner films than if there was no reduction in line energy at the thermodynamic limit. We will show LEEM observation of dislocations extending for the SiGe on SOI system for SiGe film thicknesses that could not occur without a reduction in the dislocation line energy. We observe no dislocations for the same film grown on bulk substrates. By dislocation counting, the resulting relaxation is calculated and compared to a force balance model, with good qualitative agreement. (1) Kästner, G. and U. Gösele, Appl. Phys. Lett., 2003. 82(19):3209. (2) Rehder, E.M. and T.F. Kuech, submitted to J. Appl. Phys. Research supported by DARPA and NSF

## 9:45 AM $\underline{A6.5}$

Low Friction / Flexibel Material. Yasufumi Shibata<sup>1</sup>, Katsutoshi Noda<sup>1</sup>, Takeshi Hamada<sup>2</sup> and Noriko Yamada<sup>2</sup>; <sup>1</sup>Material Engineering Division, Toyota Motor Corporation, Susono, Shizuoka, Japan; <sup>2</sup>Technical Development Bureau, Nippon Steel Corporation, Futtsu, Chiba, Japan.

We have been developing the Low friction / Flexible material that has flexibility, lypophilic, high mechanical strength and high thermal conductivity, and also developing the fundamental applying technologies for a piston ring. The objective is reduction of friction force of piston ring to less than 80% of the cast iron, keeping the property of thermal conductivity. The basic material is Inorganic / Organic hybrid. In this paper, by controlling the inorganic component, we studied to improve lypophilic and mechanical strength, and evaluated friction property of developed material. In the evaluation of friction property, stribeck model was used to analyze, and it was revealed that the fluid lubrication area of Low friction / Flexible material is much wider than other materials such as metal and ceramics.

SESSION A7: Biotechnology and Nanotechnology Chair: David LaVan Tuesday Morning, December 2, 2003 Room 200 (Hynes)

## 10:30 AM A7.1

Protein Transport and Separation on a Glass Microchip.

Chang Lu<sup>1</sup>, Tina Jeoh<sup>2</sup>, Larry P. Walker<sup>2</sup> and Harold G. Craighead<sup>1</sup>;

Applied and Engineering Physics, Cornell University, Ithaca, New York; <sup>2</sup>Biological and Environmental Engineering, Cornell University, Ithaca, New York.

Techniques for high-resolution separation of proteins are important for a variety of uses ranging from proteomics to the development of industrial enzymes. One of the most widely used methods for protein separation and analysis is capillary electrophoresis. We have been investigating the transport and separation of several common proteins and a family of key industrial enzymes (cellulases) in glass microchips. Micro or nanofluidic channels are fabricated in pyrex wafers by reactive ion etching and thermal bonding. Plugs of model proteins or enzymes are electrokinetically injected into a separation channel by a gated virtual valve. Laser-induced fluorescence detection is conducted in the separation channel to monitor the labeled protein bands. We have realized separation of some model proteins: conalbumin (80 kDa), bovine serum albumin (67 kDa), ovalbumin (40 kDa) and α-Lactalbumin (14.2 kDa) in their native state. We are also exploring the behavior of denatured proteins in the microchannels. We have been investigating how the electrical double layer thickness in very small channels affects the transport of proteins. The electrostatic effects of double layers are varied with different channel depths and buffer concentrations. We are also exploring using channel geometry with side wells attached to the main channel for possible separation, based on molecular sizes.

## 10:45 AM A7.2

Electrical Porous Silicon Microarray for DNA Hybridization Detection. Marie J Archer<sup>2</sup>, <u>Deoram Persaud</u><sup>1</sup>, Karl Hirschman<sup>1</sup>, Marc Christophersen<sup>2</sup> and Philippe M Fauchet<sup>2</sup>; <sup>1</sup>Departments of Microelectronic Engineering and Material Science & Engineering, Rochester Institute of Technology, Rochester, New York; <sup>2</sup>Center for Future Health and Departments of Biomedical Engineering and Electrical and Computer Engineering, University of Rochester, Rochester, New York.

The sensitivity of Porous Silicon (PSi) to the presence of charged molecules and its large internal surface area represent two important properties that make this material an ideal candidate for electrical biosensor development. We have demonstrated the use of a porous silicon electrical sensor for label-free detection of DNA hybridization and identification of different organic solvents in aqueous phase. Binding of DNA inside the porous silicon matrix induces a change in capacitance and conductance that can be measured in real time. Having addressed the suitability of macroporous silicon layers for real time detection of DNA hybridization on a single element device we have extended our findings to the fabrication of a microarray with individual device electrical addressing capabilities. On a crystalline p-type silicon wafer, process steps such as KOH etching and electrochemical etching are employed in selective regions to create a free standing porous membrane for sensing applications. Electrical contacts for each device are made on crystalline silicon regions with the biological species being infiltrated from the backside. This allows for complete exposure of the surface to the molecule of interest and reduces the generation of ionic currents through the porous matrix. Under these conditions, the most attractive features of porous silicon are fully exploited, while avoiding exposure of the sensing species to foreign materials without jeopardizing the sensitivity, selectivity and response speed. The device was fabricated and fully characterized using these performance parameters. We will report on the design and fabrication of this novel approach for an electrical-readout microarray technology as well as the results obtained from the individually addressable elements in which DNA binding assays are performed.

## 11:00 AM <u>A7.3</u>

Molecular imaging of micro- and nano- patterned proteins on gold/silicon substrates. Mandana Veiseh<sup>1</sup>, Bronwyn T. Wickes<sup>3,4</sup>, David G. Castner<sup>2,3,4</sup> and Miqin Zhang<sup>1</sup>; <sup>1</sup>Materials Science and Engineering, University of Washington, Seattle, Washington; <sup>2</sup>Bioengineering, University of Washington, Seattle, Washington; <sup>3</sup>Chemical Engineering, University of Washington, Seattle, Washington; <sup>4</sup>National ESCA and Surface Analysis Center for Biomedical Problems, University of Washington, Seattle, Washington.

Recently, patterning protein on solid substrates has drawn considerable attention because of its important role in developing biosensors, molecular electronics, bioreactors, and tissue engineering. In our work, various proteins are patterned on gold-silicon substrates with high precision, selectivity, bioactivity, and reproducibility. The

patterning is accomplished by an approach that combines the photolithography and surface molecular engineering. A micro- and nano- array of gold squares are fabricated on a silicon substrate by microfabrication technology. To pattern proteins in the gold regions only, the silicon background is immobilized with polyethylene glycol (PEG) to resist protein adsorption, and the gold regions are subsequently bound with proteins. Fourier transform infrared (FTIR) reflectance spectroscopy is used to confirm the immobilization of proteins on substrates. Proteins are tagged with Rhodamine fluorescent probes to visualize the pattern formed on the substrate by fluorescence microscopy. We also use the time-of-flight secondary ion mass spectroscopy (TOF-SIMS) to examine each step of our surface modification scheme to optimize the processing conditions. The images are constructed for each secondary ion species corresponding to each mass detected. Fragment signals corresponding to amino acids can be identified in the analysis of protein molecules.

## 11:15 AM A7.4

Interfacing Biological Systems with MEMS by Means of Poly(ethylene glycol) Photolithography. Alex Revzin<sup>1</sup>, Michael V Pishko<sup>2</sup> and Mehmet Toner<sup>1</sup>; <sup>1</sup>Center for Engineering in Medicine, Massachusetts General Hospital / Harvard Medical School, Charlestown, Massachusetts; <sup>2</sup>Chemical Engineering, Materials Science and Engineering, Pennsylvania State University, University Park, Pennsylvania.

Successful integration of biological systems with microfabricated devices requires methods for rendering surfaces of these devices resistant to non-specific cell and protein attachment. This paper describes a surface modification procedure whereby poly(ethylene glycol)-diacrylate (PEG-DA) is photopatterned to manufacture micron-scale hydrogel microstructures on glass or silicon. This approach mimics traditional photoresist lithography in that a polymer solution is spin-coated on the surface and exposed to UV light through a photomask. However, rather than form a barrier to penetration of etching agents, fabricated PEG micropatterns resist protein or cell adhesion. Because of its similarity to a "top-down" photolithography process, this surface patterning method could be combined with registration steps to form cell-repellent PEG microstructures on top of existing microfabricated layers. In general, biocompatibility of silicon or glass substrata modified with PEG was greatly enhanced as very limited non-specific cellular or protein adsorption was observed.

### 11:30 AM A7.5

Controlling the Diffusion of Single Organic Molecules by Means of STM Manipulation. Roberto Otero Martin, Frauke Hummelink, Federico Rosei, Michael Schunack, Peter Thostrup, Erik Laegsgaard, Ivan Stensgaard and Flemming Besenbacher; Department of Physics and Astronomy, University of Aarhus, Aarhus, Denmark.

The diffusion of large and complex organic molecules on surfaces is a topic with important implications for very different fields, like the growth of well-ordered organic ultra-thin films for optical or electronic devices, or the dynamics of biological molecules on surfaces. Although a very detailed knowledge of the diffusion processes of atoms and small molecules on surfaces exists, the concepts developed in the course of this research indicate that traditional parameters such as energy barriers and prefactors are insufficient to describe the kinetic behaviour of larger and more complex molecules. Due to their complexity, these molecules present often a rich variety of adsorption geometries and conformations, but little is known about the role of these effects on their diffusion properties. In this contribution we will show that the diffusion coefficient of a molecule known as Violet Lander (VL, C108H104) on Cu(110) can be decisively modified by forcing new adsorption geometries by means of STM manipulations. This molecule, a model for a molecular wire, aligns parallel to the close-packed surface rows, and the molecules are observed not to diffuse at RT. By means of STM manipulations at low temperatures (< 200 K), the VL can be forced to adopt a new adsorption geometry in which the axis of the molecule is 70 degrees off the close-packed direction of Cu(110). No other adsorption geometry can be forced. STM movies prove that those molecules with the board rotated diffuse on the surface along [1-10] even at 150 K, whereas the molecules that are aligned along the close-packed direction are not observed to move even at RT. Moreover, the diffusing molecules can be stopped at any time simply by flipping their orientation back to the [1-10] direction, allowing us to discard the possible influence of pinning defects of the substrate. Therefore, the diffusion of the molecules can be activated or deactivated simply by rotating the board of the molecule with the STM tip. In conclusion, we have proven that the diffusion coefficient of the VL molecule on Cu(110) depends on their adsorption geometry. This effect can be explained by the geometrical constrain imposed to the point-contacts by the molecular architecture, that forces them to diffuse in a correlated manner that differs depending on the adsorption geometry. Therefore, the diffusion of the VL molecules can be turned on and off simply by inducing a rotation of the molecule with the aid of the STM tip.

11:45 AM <u>A7.6</u>

Potentiometric Detection Of DNA Molecules Hybridization Using Genetic Field Effect Transistor And Intercalator.

Toshiya Sakata, Hidenori Otsuka and Yuji Miyahara; Biomaterials Research Center, National Institute for Materials Science, Ibaraki, Japan.

Several types of DNA chips and DNA microarrays have been developed and some of them are used in the field of molecular biology. Although most of the current DNA chips and DNA microarrays are based on the fluorescent detection method, amperometric detection methods have been developed in combination with redox reagents. In this work, the novel approach to realize an electrochemical and a potentiometric detection for DNA chips was investigated using a genetic field effect transistor (FET) and intercalator. The surface of genetic FET was covered with a Si3N4 as insulated gate. Reactive amino groups were generated on the Si3N4 surface based on treatment with aminosilane. Amino-modified oligonucleotide probes were attached following surface treatment with glutaraldehyde. Free aldehyde groups were blocked with glycine. Double stranded DNA molecules were heat denatured and annealed to the genetic FET. Although DNA molecules are negatively charged in an aqueous solution, it is in general difficult to detect them with field effect devices. One of the reasons for difficulty is that the charge density change as a result of hybridization is small. Therefore, some intercalators such as ethidium bromide, Hoechst 33258, and so on were introduced to the genetic FET gate surface with hybridized DNA molecules and the potential difference before and after the introduction of intercalator was measured. The signal based on the introduction of intercalator to the genetic FET gate surface with hybridized DNA molecules was significantly obtained. Most of intercalators have ionized characteristic in an aqueous solution, while they are used as a fluorescent reagent in the conventional method. Intercalators are ionized and positively charged, and introduced into double stranded oligonucleotide probes on the genetic FET surface gate, which leads to increase of the surface charge density. In this way, the signal of DNA molecules hybridization was enhanced and detected by the use of genetic FET with intercalator. We developed a new method for detecting DNA molecules using genetic FETs and intercalators. The result could be valid for single nucleotide polymorphisms (SNPs) detection.

> SESSION A8: Mechanical Properties II Chair: Mark McNie Tuesday Afternoon, December 2, 2003 Room 200 (Hynes)

## 1:30 PM <u>A8.1</u>

A Bicrystal Microcantilever Method for Evaluating the Effects of Segregants and Misorientation Angle on Interfacial Fracture Toughness. Deanna L Phillips  $^{1,3}$ , Lukmaan Bawazer $^{1}$ ,

Khevna Shastry<sup>2</sup>, Peter Anderson<sup>1</sup>, Ken Sandhage<sup>1</sup>, Pirouz Pirouz<sup>2</sup> and Ron Kerans<sup>3</sup>; <sup>1</sup>Materials Science and Engineering, The Ohio State University, Columbus, Ohio; <sup>2</sup>Materials Science and Engineering, Case Western Reserve University, Cleveland, Ohio; <sup>3</sup>Air Force Research Labs, Wright Patterson Air Force Base, Ohio.

The work reports on the design, processing, and evaluation of a microcantilever test method designed to accelerate the measurement of fracture toughness in pure and doped samples at scales much smaller than traditional test methods. The approach involves micromachining of numerous cantilevers from polished, diffusion-bonded wafers and subsequent mechanical testing using a nanoindenter or fiber push-out apparatus. To demonstrate feasibility, cantilevers 500 microns thick and 500 microns wide composed of sapphire doped with yttrium were fabricated to study both the effect of yttrium dopant level and grain boundary twist orientation on fracture toughness. Important scaling relations were identified to favor grain boundary fracture rather than cantilever snap-off as the dominant mode. Fracture toughness results for both determinants-segregant level and grain boundary misorientation-are reported here for sapphire/sapphire interfaces. Pre and post fracture characterization of the chemical and structural features of the grain boundaries and fracture surfaces are correlated with trends in fracture toughness. The presentation will conclude with a critical assessment of the benefits and drawbacks of this test method to determine mechanical properties at the micron scale.

## 1:45 PM A8.2

Characterization of Microsystems with Depth-Sensing Instrumented Indentation. Nuwong Chollacoop, Ming Dao and Subra Suresh; Dept of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

A methodology for interpreting instrumented sharp indentation with

dual sharp indenters of different tip apex angles is presented by recourse to finite element computational modeling. The forward problem predicts an indentation response from a given set of elasto-plastic properties. On the other hand, the reverse analysis seeks to extract elasto-plastic properties from depth-sensing indentation response by developing algorithms derived from computational simulations. Finite element computations were carried out for a wide range of material properties and various tip apex angles (i.e. 50, 60 and 80 degrees) in addition to the apex angle of 70.3 degrees for Berkovich/Vickers indenters. Using dimensional analysis, additional closed-form dimensionless functions were constructed to relate indentation response to elasto-plastic properties for different indenter tip geometries. Incorporating the results from 60-degree tip to the single-indenter algorithms, the forward and reverse algorithms for dual indentation can be established. Comprehensive sensitivity analyses showed much improvement of the dual-indenter algorithms over the single-indenter results. Experimental verifications of these dual-indenter algorithms were also carried out. The proposed methodology is examined in the context of the uniqueness of mechanical properties extracted from instrumented indentation. Possible extensions of the present results to studies involving multiple indenters are also suggested.

## 2:00 PM <u>A8.3</u>

Elastic and Plastic Behavior in Nanoscale Materials.
Tahir Cagin and William A Goddard; Caltech, Pasadena, California.

Properties (electronic, optical, dynamic, mechanical) of various materials show dramatic changes as the dimensions of the systems reaches sub-micron levels. In this presentation, we will particularly deal with the mechanical properties. In macroscopic materials the surface to volume ratio (which tends to zero) of materials enable us to treat them free from the surface effects. As it is commonly known, the behavior beyond the elastic limit, each material type (metal, ceramic, polymeric) has a specific way of accommodating the plastic deformation energy, i.e. accommodating through the creation/healing and dynamics of extended defects (dislocations, cracks, void growth and coalescence). The nanoscale materials, due to their increasing surface to volume ratio (i.e. S/V  $\sim$  1/d, where d is the smallest dimension of the sample), reveal a rich novel set of events to accommodate the deformations induced by external loads. For instance, in carbon nanotubes these may be various modes of surface undulations (these are not defects in the usual sense). The deformations may even be accommodated by surface events with chemical rearrangements. Indeed, the nanoscale mechanics present nanoscale simulation and modeling both new opportunities and challenges to attack this area through discrete particle-based simulations. Over the years, we have studied the mechanical properties of several low dimensional systems at the atomic scale: nanodots (metallic clusters, dendrimers), nanowires (metallic wires), nanoshells (thin films of metals, oxides, and self assembled monolayers of organic films), carbon nanotubes (isolated, bundled single and multiple walled). We not only have determined the elastic properties of these systems, but also identified and analyzed various failure modes, the yield behavior, and new modes of deformation arising due to nanoscale size of some or all dimensions. Using various level molecular level modeling techniques (from electronic structure methods to molecular dynamics) we will describe examples from nanomechanical studies we have conducted on nanoscale materials.

## 2:15 PM <u>A8.4</u>

Contact Stresses in Nanoscale Contacts: Comparison of Simulations to Continuum Predictions. Sangil Hyun 1,2, Binquan Luan 1, Judith A Harrison 3 and Mark O Robbins 1,2; 1 Physics and Astronomy, Johns Hopkins Univ., Baltimore, Maryland; 2 Mechanical Engineering, Johns Hopkins Univ., Baltimore, Maryland; 3 Chemistry, Unites States Naval Academy, Annapolis, Maryland.

Contact mechanics is traditionally based on continuum approaches that assume homogeneous bulk properties and that the contacting surfaces are smooth on sufficiently small scales. As contact dimensions move below the micron scale, these approximations become increasingly questionable, yet analytic results from continuum mechanics are still routinely applied. The common assumptions of isotropy and homogeneity also become less valid as smaller regions are sampled. This talk will describe tests of continuum theory against molecular dynamics simulations in a variety of geometries. Examples include atomically flat surfaces and self-affine surfaces whose overall geometry is either flat or cylindrical. Both crystalline and amorphous solids have been studied. While some correspondence between continuum and molecular results can be found in all cases, there are usually substantial discrepancies and anisotropy is almost always relevant. Atomic scale roughness leads to large fluctuations in local surface stresses, and the finite range of molecular interactions complicates comparison of contact areas. These effects lead to local plasticity at lower stresses than anticipated and change the effect of adhesive forces.

## 2:30 PM A8.5

# ${\bf Control\ of\ Stress\ in\ a\ Metal-Nitride-Metal\ Sandwich\ for\ CMOS-Compatible\ Surface\ Micromachining.}$

Rhodri Rhys Davies, David J Combes, Mark E McNie and Kevin M Brunson; Optronics, QinetiQ Ltd, Worcestershire, United Kingdom.

Further to previous work which demonstrated the ability to engineer the in- and out-of-plane stress components in PECVD silicon nitride, this paper reports on the control of stress in a metal-nitride-metal sandwich. The addition of a symmetric metalisation to a core nitride layer gives additional functionality by enabling electrical connectivity and electrostatic transduction to provide a widely applicable structural layer for micromachining. A variety of process conditions and layer combinations have been investigated. Detailed analysis of the in- and out-of-plane stress components within the sandwich using dedicated test structures has been used to optimise the structural layer properties. As the elastic modulus of aluminium is approximately three times lower than nitride, the effective strain components of the sandwich would be dominated by the metal stress unless compensated for by making the nitride layer significantly thicker. However, the stress relaxation of aluminium as a consequence of post-deposition thermal processes must also be considered. The relationships between stress components of the sandwich as a function of the nitride deposition parameters are reported. The measured effective elastic modulus of the sandwich was determined to be 115GPa and a value of 195GPa was extracted for the nitride by modelling. The optimised sandwich is repeatably controlled to realise low tensile in-plane stress (<100MPa) and low out-of-plane stress gradient (0±10MPa/micron). These parameters are used in CAD to improve simulation accuracy and maximise the chance of first-pass success of a design in the process. Work to further reduce the in-plane stress is on-going by tailoring the strain in each metal layer. The metal-nitride-metal sandwich described in this paper may also be used to post-process MEMS on CMOS as it is a low temperature process based on standard CMOS materials. Examples of successful monolithical integration of devices utilising metal-nitride structural layers include capacitive micro-ultrasonic transducers and microphones.

## 2:45 PM A8.6

Microtribological Properties of Silicon and Titanium Carbide. Imad Ahmed, Giuseppe Bregliozzi and Henry Haefke; Micro and Nanomaterials, CSEM S.A., Neuchatel, Switzerland.

The stiction, high friction and wear problems associated with the most popular MEMS material: silicon is well-documented in several studies in literature. Present solutions consist of either coating the silicon surface to reduce its poor tribological properties or by abandoning silicon and using alternative materials. One material with favorable tribological properties that has been identified is silicon carbide. Another possible alternative could be titanium carbide, which has shown favorable properties in specialty systems such as high precision ball bearings in space applications. Both materials can also be deposited on silicon surfaces. This paper examines the microtribological properties of both surfaces sliding against a 1-2 mm diameter titanium carbide, silicon and silicon nitride counterbody. A key difficulty in making comparative material studies in the microtribological regime is obtaining identical surface topographies. Adhesion properties examined by measuring the pull-off force between a smooth titanium carbide ball and the two carbides indicate a low pull-off force for titanium carbide against titanium carbide. The pull-off force could be reduced an order of magnitude by using a rougher counterbody made of the same material. The friction-load curves measured under low-loads (up to 3 mN) show titanium carbide as the surface with the lowest friction. No velocity dependence on friction was observed for both materials, but this is believed to be the result of the sample surface roughness. The least humidity dependence from both materials was observed for titanium carbide. Friction maps of both surfaces were generated to identify application regimes of the two materials and wear of sample and counterbody was examined by scanning electron microscopy and atomic force microscopy. This study shows that carbide surfaces are well-suited for microtribological applications.

> SESSION A9: Nanotechnology II Chair: Prasad Somuri Tuesday Afternoon, December 2, 2003 Room 200 (Hynes)

## 3:30 PM A9.1

Mechanical Behavior of Helical Nanosprings and Nanorods under Monotonic and Fatigue Loading Conditions. Deli Liu $^1$ , Fazeel Khan $^1$ , Dexian Ye $^1$ , Catalin Picu $^1$ , Gwo-Ching Wang $^1$ , Toh-Ming Lu $^1$  and B.-K. Lim $^2$ ;  $^1$ Rensselaer Polytechnic Institute, Troy, New York;  $^2$ Nanyang Technological University, Singapore, Singapore.

The emerging field of nanotechnology requires basic understanding of the physical, electrical, and optical properties of smaller and smaller volumes of materials. In particular, knowledge of the mechanical behavior of materials in the nanoscale regime is essential for building useful nano-machines. However, both the fabrication of suitable nanosize test structures and the measurement of their mechanical properties are challenging. In the present work, we show that it is possible to measure the elastic behavior of individual (isolated) helical nanosprings and nanorods (fabricated by the oblique angle deposition with substrate rotation) using a tip-cantilever assembly attached to a conventional atomic force microscope (AFM). The height of the helical spring structures is about 4 microns, while the wire diameter is about 200 nm. The nanorods are 1.5 microns long and 50 to 90 nm in diameter. The elastic response and the behavior under repeated loading conditions (fatigue loading) of the individual nanostructures, was measured. The response of the structures was modeled using finite elements and it was shown that the conventional formulae for the spring constant obtained by axial loading requires modifications before it could be used for the direct loading scheme employed in the present experiment.

### 3:45 PM A9.2

In-Plane Deformation of Single Walled Carbon Nanotubes and its Effect on Electron Transport. Jan M. Smits <sup>1,5</sup>, Buzz Wincheski<sup>2</sup>, JoAnne Ingram<sup>3</sup>, Neal Watkins<sup>2</sup> and Jeffrey Jordan<sup>2</sup>; <sup>1</sup>Lockheed Martin Space Operations, Hampton, Virginia; <sup>2</sup>NASA Langley Research Center, Hampton, Virginia; <sup>3</sup>Swales Aerospace, Hampton, Virginia; <sup>4</sup>National Research Counsel, Hampton, Virginia; <sup>5</sup>Joint Institute for the Advancement of Flight Sciences, George Washington University, Hampton, Virginia.

Incorporating the use of electric field alignment and various lithography techniques we have developed a single wall carbon nanotube (SWNT) test bed for measurement of conductivity/strain relationships. Nanotubes are deposited at specified locations through AC electrophoresis. Contact electrodes are then deposited over the aligned tubes to provide low resistance junctions for conductivity measurements of the SWNTs. The circuit is designed such that the central, current carrying section of the nanotube is exposed to enable atomic force microscopy and manipulation in-situ while the transport properties of the junction are monitored. Studies of in-plane strains in the SWNTs produced through the use of the Nanomanipulator haptic feedback atomic force microscope (AFM) system, are discussed. Applications include the ability to alter the transport properties of single walled carbon nanotubes by applying mechanical strain using an AFM and Nanomanipulator system.

## 4:00 PM <u>A9.3</u>

Carbon nanotube network as a chemical gas sensor. Youngsik Song and Jaewu Choi; Dept. of Electrical and Computer Engineering, Detroit, Michigan.

We have developed a highly sensitive chemical gas sensor by employing direct horizontal growth of carbon nanotube between two electrodes. The significant conductivity change of the carbon nanotube was observed with extremely small amount of gases. Carbon nanotubes were grown directly from metal electrode with the present of electric field.  $\bar{\mathbf{T}}$ he electrodes had three-layer structure, where catalyst layer was embedded between two metallic layers. The diameter of carbon nanotube was controlled by the thickness of catalyst layer, and the directional growth was also controlled by electric field. Semiconducting carbon nanotube only circuit was obtained from mixed phases of carbon nanotubes by breaking out metallic carbon nanotubes to achieve high gas sensitivity. In addition to the transport measurement, we employed SEM and SPM to study growth pattern, and atomic and electronic structure of carbon nanotubes. This study indicates that the high sensitivity of the sensor is attributed to its structure consisted of single-phase -semiconducting carbon nanotubes, and low contact resistance by direct growth from metal electrodes.

## 4:15 PM <u>A9.4</u>

Ferroelectric Nanoshell Tubes. Yun Luo<sup>1</sup>, Izabela Szafraniak<sup>1</sup>, Valanoor Nagarajan<sup>3</sup>, Ralf B. Wehrspohn<sup>1</sup>, Martin Steinhart<sup>1,2</sup>, Ming-Wen Chu<sup>1</sup>, Joachim H. Wendorff<sup>2</sup>, Nikolai Zakharov<sup>1</sup>, Ramamoorthy Ramesh<sup>3</sup> and Marin Alexe<sup>1</sup>; <sup>1</sup>MPI of Microstructure Physics, Halle/S, Germany; <sup>2</sup>Institute of Physical Chemistry, Philipps-University, Marburg, Germany; <sup>3</sup>University of Maryland, College Park, Maryland.

Tubular nanostructures have attracted increasing interest as building blocks for miniaturized devices [1]. In particular, oxide nanotubes exhibiting ferroelectric or piezoelectric properties can serve as components for nano-electromechanical systems (NEMS). Here, we report on the ferroelectric switching of lead zirconate titanate (PbZr0.52Ti0.48O3, PZT) and barium titanate (BaTiO3, BTO)

nanotubes by atomic force microscopy in the piezoresponse mode. Rectangular ferroelectric hysteresis loops of individual PZT nanotubes were obtained, showing a sharp switching at the coercive voltage of about 2 V and an effective remnant piezoelectric coefficient of about 90 pm/V. The BTO and PZT nanotubes were fabricated by wetting of ordered porous templates [2], such as porous alumina and macroporous silicon, by polymeric precursors. After pyrolysis and crystallization, the as-obtained ferroelectric oxide nanotubes show perovskite phase. The nanotubes were straight, smooth and had a very high aspect ratio of more than 50. Depending on the templates, their outer diameter ranges from 50 nm up to several micrometers and they had lengths up to more than 100 micrometers. Template-wetting and a subsequent selective etching step allows fabricating extended ordered arrays of aligned free-standing ferroelectric tubes with one end embedded in silicon template. Composite systems consisting of ferroelectric oxides and metals obtained by consecutive wetting of the porous structures may act as capacitor with an enhanced specific surface or as tunable photonic crystals. [1] C. R. Martin, Science 1994, 266, 1961 [2] M. Steinhart, J.H. Wendorff, A. Greiner, R.B. Wehrspohn, K. Nielsch, J. Schilling, J. Choi and U. Goesele, Science 2002, 296, 1997.

## 4:30 PM A9.5

Gas adsorption evidence of single-wall and multi-wall carbon nanotube opening. Moulay Rachid Babaa<sup>1</sup>, Nicole Dupont-Pavlovsky<sup>1</sup>, Fabrice Valsaque<sup>1</sup>, Edward McRae<sup>1</sup>, Sandrine Delpeux<sup>2</sup>, Francois Beguin<sup>2</sup>, Ingrid Ingrid Stepanek<sup>3</sup> and Patrick Bernier<sup>3</sup>; <sup>1</sup>UMR CNRS 7555 - UHP Nancy I, Laboratoire de Chimie du Solide Mineral, Vandoeuvre les Nancy, France; <sup>2</sup>CNRS - Universite Orleans, Centre de Recherche sur la Matiere Divisee, Orleans, France; <sup>3</sup>UMR CNRS 5581 - Universite Montpellier II, Groupe de Dynamique des Phases Condensees, Montpellier, France.

Carbon nanotubes offer a surface very similar to that of graphite, a reference substrate in physisorption experiments aimed at studying substrate-adsorbate interactions with simple or more complex molecules. The curvature, however, introduces new questions about the this interaction. What are the effects of this curvature on condensation pressures or heats of adsorption? Can one experimentally distinguish between different adsorption sites? In the current study, some of these questions are examined through comparing adsorption isotherms with several simple gases (Kr, Xe, CCl4) on both single-wall (SWNTs) and multi-wall carbon nanotubes (MWNTs), before and after opening. For mechanically opened SWNTs, prepared by the electric arc technique, the accessibility of the adsorption sites and the molecular arrangements of the adsorbed gases are discussed as a function of the molecule size and the isosteric heats of adsorption are calculated. The much bigger, well-defined MWNTs were synthesized by decomposition of acetylene at 900°C over a 10% Co silica catalyst. For these tubes, the "cutting method" called upon a nitric acid treatment. TEM investigations confirmed the tube opening and physisorption studies further clearly showed that the inner channels thus became accessible to some gas molecules.

## 4:45 PM <u>A9.6</u>

Structure, Formation Mechanisms and Properties of Carbon Nanotube Y- Junctions. Leonid Alex Chernozatonskii<sup>1</sup>, I. V. Ponomareva<sup>1</sup>, A. N. Andriotis<sup>2</sup> and M. Menon<sup>3</sup>; <sup>1</sup>Material Research, Institute of Biochemical Physics of RAS, Moscow, Russian Federation; <sup>2</sup>Institute of Electronic Structure and Laser Foundation for Research and Technology-Hellas, Heraklio, Crete, Greece; <sup>3</sup>Dept of Physics and Astronomy, University of Kentucky, Lexington, Kentucky.

Carbon nanotube multi-terminal junctions have recently emerged as excellent candidates for use as building blocks in the formation electronic devices. Between them high-yield fabrication of Y junctions have been obtained by using template-based chemical vapor deposition, and pyrolis of organometallic precursor, and electron beem welding. The conductance measurements of these Y junctions have shown intrinsic nonlinear and asymmetric I-V behavior at room temperature. Here we consider: - the formation mechanism of Y-junctions in developing template-based CVD and pyrolis techniques ; - energy optimization different types of Y and T junctions (metal or semiconductor branches and stem, symmetric or asymmetric branches) using the molecular-dynamic methods; - coalescence of two or three nanotubes into T- and Y- junctions comparison with experiments; - topological classifications of planar Y- ("fork", "slingshot"-, "bough"- types), and T- junctions which require the presence of topological defects in the form of pentagons, heptagons and octagons, the relationship between the number of defect rings and number of branches for an arbitrary nanotube junction; - theoretical calculations of I-V characteristics and formation conditions of T- and Y- junctions; - molecular-dynamic calculations of Y- junctions behavior under their branch loading. This work was supported by NSF (ITR-0221916), Russian programs "Topic directions in condensed matter physics" and "Low-dimensional quantum structures".

SESSION A10: Applied Micro and Nanotechnology Chair: Marc Madou Wednesday Morning, December 3, 2003 Room 200 (Hynes)

## NOTE EARLY START

### 8:15 AM A10.1

Fabrication and RF Properties of Titanium Carbide/Gold Coplanar Waveguides for RF MEMS Applications.

<u>Gouri Radhakrishnan</u>, Samuel S Osofsky, Ruby E Robertson, James S Swenson, Paul M Adams and Keven S MacGowan; The Aerospace Corporation, Los Angeles, California.

The application of MEMS technology to RF microwave systems is being actively pursued because of the high potential benefits of this technology for better system performance and reduced cost. In the area of active RF switching for antenna applications, low loss, low cost, small size, and low weight are a necessity. Major questions remain about the performance and long-term reliability of these MEMS switches in a space environment. Typically, gold (Au) is the metal of choice for planar RF transmission lines because of its high conductivity. However, Au is soft and tends to stick to itself and other materials under continuous switching action when used in a MEMS switch configuration. One method for mitigating the undesirable mechanical properties of the Au is to deposit a hard coating, such as titanium carbide (TiC), on top of the Au in the switching region. RF switches built of metal film stacks may have substantially higher losses than the bulk metal properties would predict. Hence when implementing a new design, such as a Au-TiC stack, it is critical to measure the additional loss due to the deposited coating, i.e. TiC. Here we report on a first set of measurements for RF loss due to pulsed laser deposited (PLD) TiC on Au using coplanar waveguide (CPW) structures. These structures comprise film combinations involving Au only, TiC only, and TiC on Au. The effect of varying TiC/Au thickness ratios on these structures has also been investigated. Multiple-line Through-Reflect-Line (TRL) CPW calibration sets were fabricated with different Au and TiC film combinations. The response of the TRL calibration sets is measured by means of microwave probes. The measured results are used to determine the loss of these films and film combinations by comparison of the waveguide attenuation constant and thus evaluate their performance.

## 8:30 AM A10.2

Characterization and Modeling of Wafer- and Die-Level Uniformity in Deep Reactive Ion Etching(DRIE). Hongwei Sun, Martin Schmidt and Duane Boning; Microsystems Technology Laboratories, Massachusetts Institute of Technology, Cambridge, Massachusetts

Study of etching non-uniformity in DRIE is urgently needed since this non-uniformity strongly influences the performance of MEMS devices such as high speed rotating devices. This paper focuses on experimental methodology and modeling to quantitatively characterize etching uniformity during DRIE etching. Unlike previous work focused on feature scale effects in deep and narrow silicon etching (involving RIE lag and ARDE effects), we investigate waferand die-level variations in etching large feature MEMS structures. Using new test masks, a two-level DRIE etch model has been developed and successfully applied to predict observed etch non-uniformity in high-speed rotating microstructures. Understanding the separation of wafer level from die level is achieved by sequentially etching uniformly distributed holes with wafer level loadings of 0.03%, 1.1%, 4.4% and 17.6%. The loading is adjusted by varying the sizes and spacing of holes. A series of wafer level etching maps obtained based on this method reflect clearly the transition from ion-limited region to neutrals transport limited region. The etching results for single die was successfully predicted by these wafer-level etching maps in the absence of so called 'die-to-die' interaction. In the specific etching example we conducted, the long-range die-level etch interactions are found to contribute non-uniformity at a comparable magnitude to that of wafer-level. This conclusion is proved by rotating wafers to eliminate the wafer level non-uniformity. The die-level etching variation is proposed to result from a distance-dependent depletion of neutrals and product species around the etching regions. To characterize this effect, annulus dies were etched with uniformly distributed monitoring points. The normalized etch rate after subtraction of the wafer-scale dependence shows clearly its effect. A model considering both the diffusion of neutrals and reaction rate of neutral with silicon was built to predict the etching of up to 21 dies. Good agreement between measurement and prediction prove that the depletion of radicals is the main contributors to die level etching variation. In summary, we show results that allow prediction and modeling of die-to-die and wafer level etch variations. The modeling tool we build can capture pattern dependence in Deep Reactive Ion Etching.

### 8:45 AM A10.3

Process for high aspect ratio carbon nanotube atomic force microscopy probes. Yusuf N Emirov<sup>1</sup>, Joshua D Schumacher<sup>1</sup>, Martin M Beerbom<sup>1</sup>, Z F Ren<sup>2</sup>, Z P Huang<sup>3</sup>, B B Rossie<sup>4</sup> and Rudy Schlaf<sup>1</sup>; <sup>1</sup> Electrical Engineering, University of S. Florida, Tampa, Florida; <sup>2</sup>Boston College, Boston, Massachusetts; <sup>3</sup>NanoLab, Inc., Brighton, Massachusetts; <sup>4</sup>Center for Ocean Technology, University of S. Florida, St. Petersburg, Florida.

Due to their great mechanical strength, carbon nanotubes (CNT) are promising candidates for high aspect ratio atomic force microscopy (AFM). Applications in sub 100 nm critical dimension metrology are among the envisioned uses for such probes. The challenge for the preparation of CNT AFM probes lies in the precise placement of one well-defined CNT at the end of a regular Si cantilever tip fitting commercially available AFM equipment. This CNT needs to have a well-defined diameter, length and orientation. We report about our recent progress in developing a manufacturing process for such CNT probes. Our process is based on CNT growth by plasma enhanced chemical vapor deposition (PECVD), which uses a catalyst to induce CNT growth. The need for a catalyst enables the specified placement and the definition of the diameter of the grown CNTs. Our method uses the focused ion beam (FIB) technique in combination with thin film catalyst deposition techniques to define the catalyst patterns.

### 9:00 AM <u>A10.4</u>

Nanomechanical resonators can act as RF bandpass filters at specific center frequencies and bandwidths, and they promise several advantages in high-frequency RF communications. For many RF applications, the center frequency of the RF carrier wave is very high relative to the signal bandwidth. For this reason, RF filters require high Q (very accurate center frequency resolution) and high sensitivity (very low noise figure devices). One of the barriers for practical RF applications of mechanical resonators is the intrinsic energy loss, which lower the mechanical quality factor of the resonator and thus the sensitivity of the devices. To date, several factors have been shown to influence the loss factor or Q degradation in resonators including the character of the vibration (compressional, torsional, or flexural), the nature of the attachment to the substrate, the mode shape of the oscillation and hence the resonator geometry, and finally the material properties. Based on these findings, we are developing high Q nano-mechanical resonators by optimizing the design and materials of resonators via an iterative process of experimental verification followed by model refinement. We are focusing our efforts on torsional motion in order to take advantage of a novel transduction method that utilizes an electret located on the resonator. We fabricate resonators using Si, AlN, GaN and nanocrystalline diamond that promise high-Q performance through a combination of superior bulk and surface properties. Resonators are fabricated using optical and electron beam lithography and various Reactive Ion Etching (RIE) methods developed for each material. Evaluation of resonator designs is carried out using scanning Laser Doppler Vibrometry (LDV), which enables imaging of the dynamic behavior of the resonators by generating both "modal maps" as well as determining the energy flow into the attachments. Modeling of resonator performance is performed using the finite element-based structural dynamics numerical codes.

## 9:15 AM A10.5

Non-impact electrostatic micromanipulation by voltage sequence for time. Shigeki Saito<sup>1, 2</sup>, Kunio Takahashi<sup>2</sup> and

Masataka Urago<sup>2</sup>; <sup>1</sup>Department of Material Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; <sup>2</sup>Department of International Development Engineering, Tokyo Institute of Technology, Meguro-ku, Tokyo, Japan.

Micromanipulation techniques have been in demand to realize highly functional microdevices. During micromanipulation, the influence of gravitational force becomes extremely small. The adhesional force is more significant for smaller objects. Electrostatic force is generally known to be effective for the detachment of a microparticle as the repulsive force. Our previous study experimentally clarifies the voltage for detachment of a microparticle; however, it also suggests simultaneously that the excessive kinetic energy after detachment might cause the excessive impact that prevents the soft landing of a particle. Therefore, the present study proposes how to theoretically determine voltage sequence for time to realize the kinetic control of a particle for a non-impact electrostatic micromanipulation. The system consists of three conductive objects: a manipulation probe, a spherical particle, and a substrate plate. The particle, which is initially

adhering to the probe tip, is detached due to the applied voltage. The electrostatic force on the particle is evaluated through a boundary element method. Although the numerical method is used, all the parameters are normalized. We clarify the condition of voltage and time for the non-impact method by considering the total work to the particle, which also reveals the feasibility of the method from the viewpoint of the through-rate of a power-source.

## 9:30 AM <u>A10.6</u>

 $\begin{array}{c} \textbf{Evaluating The} & \textbf{Mechanical Properties of MEMS by} \\ \textbf{Combining The Resonance Frequency and Microtensile} \\ \textbf{Methods.} & \underline{\textbf{Dongil Son}}, \underline{\textbf{Jong-jin Kim}}, \underline{\textbf{Dong-Won Kim}}, \underline{\textbf{Tae Won Lim}} \\ \textbf{and Dongil Kwon; School of Materials Science and Engineering, Seoul National University, Seoul, South Korea.} \\ \end{array}$ 

Tensile, fracture and fatigue properties of LIGA-Ni, Si and poly-Si were evaluated by the resonance frequency and microtensile methods and a new method for evaluating the fracture toughness that combines these two methods was proposed. A pre-crack was generated in an electrostatically driven test specimen and a load was applied by piezoelectrically driven microtensile equipment. Before the microtensile test, a new surface micromachining technique including two-step sacrificial layer removal was used. The pre-cracked specimen was attached to microtensile equipment by a UV-adhesive glass grip. The fatigue pre-crack was successfully introduced and the fracture toughness could be derived on the basis of fracture mechanics. The fracture toughness of the pre-cracked specimen was relatively low compared with that of the notched specimen, so that we were able to determine the effect of the notch tip radius. The dependence of the fatigue properties of LIGA-Ni film on annealing time and temperature was also evaluated. A tensile-tensile fatigue load was applied by a piezoelectric actuator, and real-time load-displacement curves were displayed via computer. The dependence of S-N curves, crack propagation rates and fatigue-notch factor on the applied load for Ni film of thickness  $10\mu m$  was analyzed; microstructural effects and applications of this test method were also discussed.

### 9:45 AM A10.7

Characterizing Thin Film Morphology And Sam Structure Using Cantilever Sensors. Mike Godin<sup>1</sup>, Olivier Laroche<sup>1</sup>, Vincent Tabard-Cossa<sup>1</sup>, Luc Y Beaulieu<sup>1</sup>, Peter Grutter<sup>1</sup>, Bruce Lennox<sup>2</sup> and P J Williams<sup>3</sup>; <sup>1</sup>Physics, McGill University, Montreal, Quebec, Canada; <sup>2</sup>Chemistry, McGill University, Monteal, Quebec, Canada; <sup>3</sup>Physics, Acadia University, Wolfville, Nova Scotia, Canada.

Cantilever sensors are an important example of micromechanical devices where an input stimulus (i.e. mass, heat, magnetic moment, electric field, surface stress, etc.) translates into a mechanical response in the sensor material (silicon, silicon nitride, etc.). If a micromachined cantilever is derivatized to become sensitized to specific stimuli, it can be capable of detection at the nanogram ( $10^{-9}$  g), picoliter ( $10^{-12}$  l), femtojoule ( $10^{-15}$  J), or attomolar ( $10^{-18}$  M) levels. In this talk we will show how cantilever sensors can be used to characterize the morphology of thin films ex situ. Moreover, we will also show how cantilever sensors can be used to determine the structure of self-assembled monolayers in situ. The results presented have direct applications in the coating industry.

SESSION A11: Alternative Fabrication Techniques II Chair: Arturo Ayon Wednesday Morning, December 3, 2003 Room 200 (Hynes)

## 10:30 AM \*A11.1

Thin Layer Transfer: From Material To New Structures.

Bernard Aspar<sup>1</sup>, Chrystelle Lagahe-Blanchard<sup>1</sup>, Marc Zussy<sup>2</sup> and

Hubert Moriceau<sup>2</sup>; <sup>1</sup>Tracit Technologies, Grenoble, France;

<sup>2</sup>CEA-DRT - LETI/DTS, CEA/GRE, Grenoble, France.

Layer transfer technologies based on direct wafer bonding and mechanical thinning down is an industrial way to perform stacked structures for microelectronics or microsystems applications. For example, such technologies are well suited to BSOI type wafers manufacturing. In this way, a lot of specific structures can be performed using such a process. For instance, it is worth achieving thick SOI wafers in which the crystalline orientation of the top silicon layer is referred to the base silicon wafer one accurately. A misorientation of less than 0.2  $^{\circ}$  between both film and substrate <100> orientations can be obtained. Furthermore, the control of wafer bonding energy allows achieving a new kind of substrates, which can be subsequently debonded after device manufacturing process. To carry out these "debondable substrates" (D-BSOITM), two approaches based either on cleaning or on roughening of wafer surfaces before bonding have been investigated. The relevance of each approach depends on the targeted applications and more specifically on thermal

budgets they need for device manufacturing process. Experiments conducted to demonstrate this debonding concept will be described. These debondable substrates open the scope to new applications notably in the field of microsystems. This generic technology is also well suited to the integration of various devices, such as integrated circuits, or microsystems. Thereby SOI devices are very attractive for device transfers as buried oxide layer is very efficient etch stop layer. We will present examples of SOI device transfers. Moreover, it will be highlighted that these devices can be transferred onto other substrates or stacked structures in order to create 3D structures. D-BSOITM is a trademark of Tracit Technologies.

## 11:00 AM <u>A11.2</u>

Piezoelectric Polyimide Mems: Fabrication And Processing. Gary Atkinson<sup>2</sup>, Zoubeida Ounaies<sup>1</sup>, Cheol Park<sup>3</sup> and Joycelyn Harrison<sup>4</sup>; <sup>1</sup>Mechanical Engineering, Virginia Commonwealth University, Richmond, Virginia; <sup>2</sup>Electrical Engineering, Virginia Commonwealth University, Richmond, Virginia; <sup>3</sup>National Institute of Aerospace, Hampton, Virginia; <sup>4</sup>NASA Langley Research Center,

We have demonstrated a process for fabricating MEMS cantilever and bridge structures using a novel high-temperature piezoelectric polyimide as the structural material. The process consists of conventional lithography and metallization processes and uses a sacrificial layer of photoresist. Electrodes are fabricated on the upper and lower surfaces of the suspended structures and a self-test electrode is fabricated underlying each suspended component for testing. Particular attention is paid to the baking and curing of the polymer films used for structural, sacrificial and lithography layers in order to maintain the structural integrity of the devices throughout the fabrication process. A key aspect is the polyimide processing (curing, solvent evaporation and poling) and its compatibility to the patterning and MEMS fabrication process. Currently, the sacrificial photoresist limits the upper cure temperature due to possible melting at higher temperature. Curing and in-situ poling are investigated using differential scanning calorimetry (DSC), dynamic dielectric spectroscopy (DDS), ellipsometry and thermally stimulated current (TSC) measurements. These measurements provide the glass transition temperature  $(T_g)$ , the dielectric constant  $(\epsilon)$ , index of refraction (n), and remanent polarization  $(P_r)$ . Monitoring these material parameters will determine two key issues: 1) the effect of cure cycle on solvent content, and 2) the effectiveness of in-situ poling on aligning the dipoles in the polyimide. Our ultimate goal is to develop a MEMS compatible curing and poling process that will result in piezoelectric constants comparable to those reported for bulk piezoelectric films. This process has application to building a variety of microelectromechanical sensors and actuators.

11:15 AM A11.3 Chemical Vapor Deposition of Polyoxymethylene as a Sacrificial Layer for Air-Gap Fabrication. Kelvin Chan and Karen K Gleason; Department of Chemical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

The goal of this research is to devise a novel technique for fabricating air gaps using polyoxymethylene (POM) thin films as sacrificial layers. Air has the lowest dielectric constant of 1.0 and would decrease the parasitic capacitance between metal lines if it replaced current intermetal dielectrics. Also, Air has the lowest refractive index of 1.0, and the replacement of current low-index materials with air would enable high-reflectance optical filters to be made with fewer layers. In the fabrication of microelectromechanical systems (MEMS), the use of POM may resemble that of silicon dioxide. POM decomposes in the absence of oxygen into formaldehyde gas between 160-400 C, leaving behind negligible residue. The actual decomposition temperature is tunable and depends on molecular weight and endcapping. POM is an engineering plastic with high modulus and strength and is resistant to most chemical attacks. These properties make POM a good sacrificial material for fabricating closed-cavity air gaps. The scheme of fabrication includes, at a minimum, four steps-thin-film deposition of POM, patterning of POM film, deposition of overcoat layer, and decomposition of POM. During decomposition, formaldehyde diffuses through the overcoat layer, leaving void spaces behind. Unlike silicon dioxide and other sacrificial materials used for optical MEMS, POM does not require an etchant. Chemical-vapor-deposition techniques have been developed for depositing thin POM films with thicknesses in the order of 1000 A. POM patterning is accomplished through oxygen-plasma etching with organosilicate glasses or photoresist as a mask. The etch selectivity of POM to photoresist is at least 5:1, and the etch rate is in the order of 1000 A/min. Closed-cavity single-level air-gap structures as narrow as 2 um have been fabricated and confirmed using cross-sectional scanning electron microscopy. Smaller air gaps can be fabricated with better lithographic tools, and multilayered structures can be achieved through multiple fabrication cycles.

## 11:30 AM <u>A11.4</u>

 $\textbf{Bulk Micromachined Titanium MEMS.} \ \underline{Marco \ F. \ Aimi, \ M. \ P.}$ Rao and N. C. MacDonald; Materials Dept., UCSB, Santa Barbara,

Recent process developments have allowed for the realization of Microelectromechanical Systems (MEMS) in bulk titanium substrates. Utilizing the MARIO Process (Metal Anisotropic Reactive Ion etching with Oxidation), arbitrarily high aspect ratio structures with straight sidewalls and micron-scale features have been fabricated in titanium substrates of various thicknesses, ranging from 1/2 millimeter plate down to 25 micron free-standing titanium foil. The use of titanium as the structural constituent allows for the exploitation of a drastically different set of material properties relative to traditional semiconductor-based MEMS (e.g. higher toughness, better biocompatibility/bioactivity, etc). Furthermore, the thin foils can be through-etched and bonded to titanium substrates as a way of electrically isolating various structures. Consequently, the MARIO Process enables the fabrication of novel devices that capitalize on these assets to yield enhanced functionalities that would not be otherwise possible using more traditional micromechanical material systems. As an illustration of the unique benefits afforded by bulk micromachining of Ti, a variety of devices that have been fabricated using the MARIO Process will be discussed, including: a micromirror array to emphasize the processing of thin foils and integration with silicon; fluidic channels to demonstrate biocompatible devices & packaging; and micro-tensile testers to enable the measurement of the mechanical properties of bulk titanium at the micron length-scale.

### 11:45 AM A11.5

Direct Writing of 3D Microstructures Using a Scanning Laser System. Hui Yu<sup>1</sup>, Alexander Gruntzig<sup>1</sup>, Yi Zhao<sup>1</sup>, Andre Sharon<sup>1</sup>, Biao Li<sup>2</sup> and Xin Zhang<sup>1</sup>; <sup>1</sup>Department of Manufacturing Engineering, Boston University, Boston, Massachusetts; <sup>2</sup>Fraunhofer USA Center for Manufacturing Innovation, Boston, Massachusetts.

Recently, the interest in microstructures with multidimensional geometries has dramatically increased in microsystems applications. However, such true 3D microstructures are difficult to be realized through conventional microfabrication techniques. In this paper, we demonstrated a simple beam-scanning laser system for rapid prototyping of 3D microstructures on soft templates. This technique facilitates very fast prototyping without the need for masks, resulting in a low-cost, short-turn-around flexible microfabrication process This technique also avoids the errors transferred from mask and/or the errors due to diffraction. A diode-pumped, high repetition rate, nanosecond pulse duration 3rd harmonic Nd:YAG laser operating at a wavelength of 355nm was used as the light source. The laser produced a maximum power of ~400 mW at 25kHz. The output energy was adjusted by varying the pump diode current or by altering the non-linear crystal oven temperature, changing the third harmonic conversion efficiency. An x-y-z- $\theta$  stage was built to translate a sample in four degrees-of-freedom. The laser light was directed through a 10× expander and a 2-axis Scan Head before it reaches the sample. The expander was set up to expand the input laser beam diameter to the Scan Head so as to produce a smaller spot size as well as a larger scan field. The maximum scan field size is 100×100mm<sup>2</sup>; the minimum spot size is  $\sim 10 \,\mu\text{m}$ . A variety of SU-8 3D microstructures were achieved such as a micro-peg array fabricated using a single inclined UV laser beam with an incident angle of  $45^{\circ}$ , a T-plug and embedded channel array using two-step inclined UV laser beam writing with stage rotation angle of  $\pm 60^{\circ}$ . The high resolution, high speed and low cost of the scanning laser direct writing system opens up a variety of applications in micro-channels, micro-nozzles, micro-mixers SU-8 fabrication and could be readily extended to other materials such as Si, glass etc.

> SESSION A12: Alternative Materials and Metrology Chair: David LaVan Wednesday Afternoon, December 3, 2003 Room 200 (Hynes)

Tribological Behavior of Novel Polymer-Derived Ceramics for Harsh-Environment MEMS Applications. Somuri V Prasad Sandeep R. Shah², Tsali Cross² and Rishi Raj²; ¹Sandia National Laboratories, Albuquerque, New Mexico; ²University of Colorado, Boulder, Colorado.

This paper deals with the tribological mechanisms in polymer-derived ceramics (PDCs) for microelectropmechanical systems (MEMS). In view of the large surface area to volume ratio, surface interactions dominate the performance and reliability of MEMS. Coatings (e.g. self-assembled monolayers) are often required to mitigate friction and wear in silicon devices. The presence of a carbon rich phase in PDCs

presents us an opportunity to tailor their microstructure and chemistry for generating self-lubricating surfaces without resorting to coatings. In the current study, we have evaluated the friction and wear behavior of SiCN ceramic. The material was fabricated by casting the polymer precursor into lithographically prepared micro-molds, followed by thermal decomposition<sup>1</sup>. Friction and wear measurements were made against a  $Si_3N_4$  counterface in a ball-on-disk configuration. Measurements were made in dry nitrogen and in humid air. Results indicate that the coefficient of friction for SiCN ranged from 0.1 to 0.2. The morphology and chemistry of wear scars were analyzed by electron microscopy and Raman spectroscopy respectively. The feasibility of fabricating high-temperature polymer-derived ceramics for MEMS applications will be discussed. <sup>1</sup>R. Raj, R. Riedel and G. D. Soraru, J.Am. Ceram. Soc., <u>84</u> (2001) 2158. \* Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000. Work at the University of Colorado was supported by the Air Force Office of Scientific Research.

### 1:45 PM A12.2

High Temperature Micromolding of Metals: Experiments and Modeling. Dong Mei Cao, Wen Jin Meng and Glenn B Sinclair; Mechanical Engineering, Louisiana State University, Baton Rouge, Louisiana.

The LiGA (Lithographie, Galvanoformung, Abformung) technology is uniquely suited for fabricating High Aspect Ratio Microscale structures (HARMs) out of non-silicon-based materials. The high cost of synchrotron X-ray lithography (Lithographie) and the slow speed/restrictiveness of electrodeposition (Galvanoformung) make replication by molding (Abformung) the key to economical mass production of HARMs and incorporation of a broader range of engineering alloys into HARMs based microdevices. Although metallic HARMs are a pre-requisite to the realization of many HARMs based micromechanical devices, especially devices for harsh environments, high temperature micromolding of metals has not been studied in detail to date. We report instrumented microscale compression molding of lead and aluminum with LiGA fabricated Ni mold inserts at ~120-275oC and ~450oC, respectively. The molding process was monitored quantitatively through continuous measurements of insert displacement and molding forces, yielding information such as load-displacement curves, work of molding, and insert displacement rates. Preliminary modeling was carried out to gain an understanding of molding forces and displacement rates, and will be discussed together with experimental results.

## 2:00 PM A12.3

Molecular Dynamics Simulations of Single Asperity Contacts: monotonic and cyclic loading. Pil-Ryung Cha and David J. Srolovitz; Department of Mechanical and Aerospace Engineering, Princeton Materials Institute, princeton, New Jersey.

Many state-of-the-art microelectronic, photonic and MEMS devices are based upon small scale contacts. While the size of contacts in MEMS and NEMS continues to decrease, the early stages of contact in present day systems occur on nanoscale dimensions through asperity contact. Descriptions of such small contacts must account for such effects as elastic and plastic deformation, adhesion and evolution of contact shape. Plastic deformation and morphology evolution on the nanoscale is fundamentally different from those at large scales. In this presentation, we present a molecular dynamics (embedded atom method potential for Au) study of single asperity contacts. We examine both loading and unloading of the contact as well as the frequency effects in cyclic loading. During the deformation process, we monitor the full force-displacement curve, the evolution of the atomic structure and asperity morphology, nucleation and motion of dislocations. The contact area and the force-displacement relation evolve discontinuously because of nucleation and motion of dislocations and structural transformations. Plastic deformation leads to the disapearance of individual atomic layers and corresponding abrupt jumps in the applied force-displacement relations, as seen in earlier nanoindentation experiments. Examination of the atomic structure showed that the dislocations that nucleated on loading tended to be partials. In the initial stages of unloading, the dislocations run out of the asperity, again leaving a perfect crystal. The tensile stresses generated during pull-off produce twin-like structures. The nature of the deformation produced in cyclic loading was extremely sensitive to the deformation rate. The hysteresis observed in repeated loading evolves with the number of cycles. Comparisons of the present results with a wide range of contact and indentation experiments will be presented.

## 2:15 PM A12.4

Measurement of the Work of Adhesion on Wafers for Direct Bonding. K. T. Turner<sup>1</sup> and S. M. Spearing<sup>2</sup>; <sup>1</sup>Mechanical Engineering, Massachusetts Institute of Technology, Cambridge,

Massachusetts;  $^2{\rm Aeronautics}$  and Astronautics, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Direct wafer bonding has emerged as an important technology in the manufacture of silicon-on-insulator substrates and micro electromechanical systems (MEMS). Success in the process relies on maintaining proper tolerances on the wafer geometry (bow nanotopography, roughness, etc.) and achieving a high work of adhesion or surface energy, which serves as the initial driving force in the bonding process. A range of tools and techniques exist to characterize the geometry of the wafers, however there are far fewer options to evaluate the work of adhesion of surfaces. This work looks at the errors associated with the traditional technique used to evaluate work of adhesion, proposes correction factors for this technique, and examines alternative techniques for evaluating work of adhesion. Historically, a displacement loaded double cantilever beam geometry, commonly referred to as the "blade insertion test", has been used to quantify the work of adhesion. The primary sources of error associated with this test technique are analyzed in detail. The effects of wafer bow and surface waviness on the measured work of adhesion are assessed through analytical models. In addition, finite element modeling is used to quantify the effect of performing the test on circular wafers rather than thin beams as the analysis that is commonly used in data reduction assumes. In addition, alternative test structures have been developed to evaluate the work of adhesion. One of these structures uses a residually stressed film combined with a shallow pattern to achieve a measurable bond area that corresponds to the work of adhesion. Experiments have been performed in which the work of adhesion has been systematically modified through the use of different wet chemistries, and then measured using the traditional blade insertion test and alternative test structures. The results from the different techniques are compared and limitations of each are discussed.

## 2:30 PM <u>A12.5</u>

Methanol Partial Pressures Effect on MEMS Cantilever Adhesion. Richard Anton Plass and Maarten P. de, Boer; Radiation and Reliability Physics, Sandia National Laboratories, Albuquerque, New Mexico.

The possibility of permanent sticking failures is one of the major concerns hindering the expected widespread implementation of MEMS technology. To properly address the role of environment on these failures, we have constructed an interferometric UHV- compatible probe station capable of isothermally heating the sample and the chamber, cleaning the sample via gas plasmas, and introducing controlled partial pressures of various solvents. Once plasma cleaned, an Si(100) blank can typically be kept clean for over 24 hrs in purified nitrogen flowing through the probe station (as determined by water contact angle measurements). No evidence of charging after plasma cleaning was observed, although a > 7 Watt oxygen plasma will cause significant silicon oxide growth. Previously [1] we have found that for oxidized polycrystalline surfaces there is an exponential dependence of surface adhesion energy on RH. More recently we have found that for in-situ plasma cleaned surfaces with roughnesses between 1.8 to 4.0 nm, adhesion remains exceedingly low ( $\sim$ 0.01 mJ/m2) to about 95% RH (even for samples manufactured in the same time frame as the results in Ref. [1]). Above  $\sim 95\%$  RH, adhesion increases rapidly with both time and humidity to over  $\sim 70 \text{ mJ/m2}$ . We believe these fortuitously low adhesion results at low and moderate humidities are an anomaly arising from the presence of 20 to 50 nm diameter particles on the surface which seem to be coming from the MEMS release process chemicals. The presence of these particles give these surfaces an effective roughness of the diameter of the larger particles (~20 nm or more) since they prevent the cantilever and landing pad from coming any closer together, a result consistent with continuum modeling. Repeating the experiment at 85 deg. C did not change the key results. Similar results were obtained when methanol was the vapor instead of water. Water partly dissolves silica and likely leaves behind solid bridges between asperities when evaporated away, the solid bridges thus lock the cantilevers in place. Methanol is not expected to dissolve silica and hence, when removed, cantilever beams should release to their previous, low-partial-vapor-pressure positions. We find that they do release, but only to a 3 to 14 mJ/m2 effective adhesion energy with a lot of scatter in the energy value from cantilever to cantilever and die to die. Neither subsequent elevated sample temperatures nor plasma cleaning will make the methanol-exposed cantilevers release further. We will also present results from cantilevers without particles. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energys National Nuclear Security Administration under contract DE-AC04-94AL85000. [1] M. P. de Boer, P. J. Clews, B. K. Smith, and T. A. Michalske, Mat. Res. Soc. Symp. Proc. 518 (1998) 131.

## $\textbf{2:45 PM} \ \underline{\textbf{A12.6}}$

Tunneling Probe as Noncontact Acoustic Emission Sensor for

Monitoring of Nanoscale Surface Modification. Oleg Lysenko, Nikolai Novikov, Alexander Sherbakov and Alexei Pokropivny; Institute for Superhard Materials, Kiev, Ukraine.

The acoustic emission method is effective tool used for monitoring of some nanotechnological procedures, for example in nanoindentation. Unfortunately, the sensitivity of conventional contact acoustic emission sensors is too low for nanotechnological measurements. In addition, all contacting sensors physically coupled with the surface of material may add mechanical loading, which can distort the source signal. The new techniques for noncontact measurements of acoustic waves and vibration parameters, such as acoustic atomic force microscopy and tunneling accelerometer have been developed the last years. We have developed a new highly sensitive technique to measure acoustic emission effect of surface modification. The experimental setup includes two probes with a semiconducting diamond tips. Nanoscale manipulator based on the 3D piezo-controlled device and aimed for nanoindentation, nanoscratching or other surface modification operates the first probe. The second probe is operated by 2D piezo-controlled device and measures the tunneling current. The acoustic emission is determined as change of tunneling current initiated by the transition of surface acoustic wave during surface modification. A theoretical model embracing the most significant features of wave phenomena at nanoscale level is described. Laboratory experiments have displayed results that are in excellent agreement with those obtained from theoretical model.

> SESSION A13: Surface Engineering and Tribology Chair: Mark McNie Wednesday Afternoon, December 3, 2003 Room 200 (Hynes)

## 3:30 PM \*A13.1

Effects of Operation, Processing and Storage Environments on the Performance and Structure of Monolayer Lubricants for MEMS. Michael Dugger<sup>1</sup>, Joshua S. Wiehn<sup>1</sup> and Diane E. Peebles<sup>2</sup>; <sup>1</sup>Microsystem Materials, Tribology and Technology, Sandia National

 $^1\mathrm{Microsystem}$  Materials, Tribology and Technology, Sandia National Laboratories, Albuquerque, New Mexico;  $^2\mathrm{Materials}$  Characterization, Sandia National Laboratories, Albuquerque, New Mexico.

Organic monolayer films have been employed during fabrication of silicon surface micromachined (SMM) devices to improve yield and insure proper device operation. Methyl-terminated monolayers reduce adhesive interactions between contacting surfaces, and lower static and dynamic friction coefficients compared to uncoated surfaces. However, changes in the chemistry of surfaces due to degradation of this film can have significant impact on the performance of SMM devices. Changes in surface chemistry can result from mechanical stress, such as that during operation of devices with contacting surfaces, or from interaction of the monolayer with other species adsorbed on the surface during storage. Improved understanding of the evolution in surface chemistry with time for devices exposed to packaging and storage environments is critical to establishing the required confidence to use SMM devices in high-consequence applications. Diagnostic MEMS structures have been used to investigate the effects of operating conditions, processing, and storage environments on the friction and adhesion performance of chemisorbed monolayer lubricants. Surface analysis, including photoelectron spectroscopy and scanning probe microscopy, has been used to investigate the chemistry and structure of the films. Exposure conditions investigated include back-end-of-line process conditions, such as elevated temperature in the presence of water vapor, outgassing products from die attach materials, temperature soaks and thermal cycling. This paper will discuss correlations between surface chemistry, structure, adhesion, and static and dynamic friction coefficient. The ramifications of changes in the behavior of interfaces on the operation of real SMM devices will also be discussed.

## 4:00 PM A13.2

Electrochemically-assisted Covalent Modification of Ultrananocrystalline Diamond Films for Biosensor Applications. Jian Wang, Xingcheng Xiao, Jennifer E. Gerbi, James Birrell, Orlando Auciello, Millicent A. Firestone and John A. Carlisle; Materials Science Division, Argonne National Laboratory, Argonne, Illinois.

Ultrananocrystalline diamond (UNCD) possesses properties well-suited for biochemical/biomedical applications such as MEMS compatible chemical sensors, chip-based biosensors, implants, and bioelectrodes. For these applications, the control of specific interactions between biomolecules and the UNCD surface is crucial. Surface functionalization provides an efficient way to tailor UNCD surfaces, which enables control of hydrophobicity and surface charges, thereby promoting the selective absorption of biomolecules, and suppressing biofouling. We report here a new strategy for surface

functionalization of conductive (N-doped) UNCD films by electrochemically reducing aryl diazonium cations in a nonaqueous medium. The one-electron transfer reaction leads to the formation of solution-based aryl radicals that couple to the UNCD surface, forming covalent C-C bonds. The modified UNCD surfaces were fully characterized by AFM, XPS, NEXAFS, cyclic voltammetry and impedance measurements. The XPS spectra show chlorine or nitrogen signals after 3, 5-dichloroenyl or 4-nitrophenyl groups were attached to the UNCD surface. The surface coverage, estimated from the electrochemical and XPS measurements, is as high as 70% of a compact monolayer. Facile electron transfer to redox active groups attached to UNCD surfaces via the aryl derivatives was observed by cyclic voltammetry and AC impedance measurements. The attached aryl derivatives also served as a conductive linker for the subsequent attachment of a variety of biomolecules. The integration of biological functions with UNCD will ultimately yield a robust platform for biosensing applications. \* This work was supported by the US Department of Energy, BES-Materials Sciences, under Contract W-13-109-ENG-38.

## 4:15 PM A13.3

Fundamental Studies of Au Contacts in MEMS RF Switches.
Steven Todd Patton<sup>1</sup> and Jeffrey S. Zabinski<sup>2</sup>; <sup>1</sup>Nonmetallic
Materials, University of Dayton Research Institute, Dayton, Ohio;
<sup>2</sup>Materials and Manufacturing Directorate, Air Force Research
Laboratory, Wright-Patterson Air Force Base, Ohio.

Fundamental aspects of the electrical and tribological behavior of Au contact materials used in microelectromechanical systems (MEMS) radio frequency (RF) switches were studied using a micro/nanoadhesion apparatus. MEMS-level contact forces (microN scale) were applied in hot switching experiments that were conducted over a wide range of electric currents. The purpose of the work was to simulate MEMS switch operation while controlling and/or measuring fundamental parameters such as normal load, adhesion, electric current, and contact resistance. An emphasis was placed on determining factors that affect switch reliability and durability. The results of this study provide a totality of information that is not available from studies using actual MEMS switches. Durability experiments were conducted in a rapid switching mode to simulate an oscillating MEMS switch, with an emphasis on failure mechanisms at various normal force and electric current. Interesting results have been obtained connecting fundamental properties to performance. Necking and nanometer scale plastic deformation were observed when separating the surfaces in quasi-static load-displacement experiments. In rapidly oscillating switch contacts, a threshold force of about 50 microN was needed for reliable switch operation. Durability of contacts was found to depend strongly on current, with switches failing after about 1000 cycles at currents at and above 1 mA (high current). The failure mechanism at high current was intermittent short-circuiting (failure to open upon demand). High and unstable contact force was observed just prior to and during switch failure. Scanning electron microscopy revealed that gold nanowires bridged the contact and were responsible for switch failure. At currents of 0.1 mA and below, switches did not fail by shorting, but instead became susceptible to high adhesion/stiction. Aging effects, due to exposure of samples to ambient air, were also observed and primarily affected contact resistance and adhesion.

## 4:30 PM <u>A13.4</u>

Effects of Adhesion on Dynamic and Static Friction in MEMS. Alex D Corwin and Maarten de Boer; Reliability Physics Deptartment (1762), Sandia National Laboratories, Albuquerque, New Mexico.

We present a series of measurements made with a MEMS inchworm device showing the presence of non-zero friction for zero applied normal load under both static and dynamic conditions. We attribute this effect to a surface adhesion, which is important for MEMS devices. The inchworm device is fabricated by polycrystalline silicon surface micromachining and consists of an actuation plate suspended between two friction clamps. With suitable application of phased voltages to the clamps and the plate, it can be made to take  $\sim 40~\mathrm{nm}$ steps. The inchworm device is coated with a monolayer coating that serves to make the surface hydrophobic, thereby preventing capillary condensation. It also reduces the shear stresses between contacting surface asperities. By walking the inchworm out against a calibrated load spring we can determine the static friction coefficient by measuring the clamp force required to overcome the tangential spring load. From these static measurements, we quantitatively determine the size of the adhesive contribution to friction to be 5  $\pm$  0.5 uN (1  $\rm nN/um^2$ ). Also, the static coefficient of friction is 0.33  $\pm$  .04. We also demonstrate a true MEMS dynamic measurement of friction. In this case, we use a "pluck" test where we walk the inchworm out some distance, release both clamps and observe the subsequent damped oscillations. We can follow the motion through the use of an electromechanical strobe technique in which we stop the motion of the inchworm at increasing times in the damping cycle. By applying a

simple model including both viscous damping and dry frictional damping, we extract both the adhesive contribution to friction of 3.9  $\pm$  0.15 uN (0.8 nN/um²), as well as a true dynamic friction coefficient 0.26  $\pm$  0.01. Both these values are approximately 80 percent of the static case. These measurements directly confirm that adhesion plays an important role in MEMS friction under both static and dynamic conditions, and enable MEMS designers to accurately account for its effects. Acknowledgement: Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed-Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

## 4:45 PM <u>A13.5</u>

Semiconductor Surface-Organic Molecule Interactions: A Case Study in the Wet Etching of InP by Alpha-hydroxy Acids. Prabhakar Bandaru<sup>1,2</sup> and Eli Yablonovitch<sup>2</sup>; <sup>1</sup>Materials Science Program, UC, San Diego, La Jolla, California; <sup>2</sup>Electrical Engineering department, UCLA, Los Angeles, California.

Organic acids, whose molecular orbital energy levels (LUMO/HOMO) are matched to semiconductor energy bands, can be used to passivate surface states and impurities. This is especially critical for nanosystems, nanoelectronics, and NEMS devices where high performance is very sensitive to the condition of the surface. In this talk, we present a case study where the concept of energy level matching has been successfully used to modify compound semiconductor surface properties [1]. Controllable etching and surface passivation of InP semiconductors are desirable for removing damaged surfaces and obtaining good electronic properties. We have observed that alpha-hydroxy acids (tartaric, malic, citric and lactic) when used in conjunction with HCl to etch (100) InP surfaces create smoother and defect free surfaces, in comparison to etches based on inorganic acids alone. The chelating action of the organic acids results in efficiently removing In from the surface leading to a very controllable etching. The implications of these chemical treatments in controlling surface band bending and surface carrier recombination velocity will be discussed. [1] P. Bandaru and E. Yablonovitch, J. Electrochem. Soc., 149 (11), G599, 2002. This project was supported in part by DARPA and ARO (MDA972-99-1-001 and DAAD19-00-1-0172).