SYMPOSIUM B

Materials Science of Microelectromechanical Systems (MEMS) Devices IV

November 25 – 28, 2001

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TUTORIAL

FT B: : MICROELECTROMECHANICAL SYSTEMS (MEMS): FABRICATION AND HOT TOPICS

Sunday, November 25, 2001
9:00 a.m. - 5:00 p.m.
Room 206 (Hynes)

The first part of the tutorial will provide an overview of the technologies and processes available for creating MEMS using surface and bulk micromachining. In the second part, special "hot topic" discussions on RF-MEMS and Micro-Chemical-Reactors will be presented. The morning section on fabrication will cover photolithography, thin film deposition, wet and dry etching, including extensive discussions of deep reactive ion etching and wafer bonding. Best practices in each of these areas will be highlighted. After lunch, the RF-MEMS section will highlight new research areas and market trends including wireless communications. The Micro-Chemical-Reactors section will emphasize trends and future prospects in this area. Micro-Chemical-Reactors device characteristics and system architectures will be discussed.

Instructors:
Carol Livermore, Massachusetts Institute of Technology
Darrin Young, Case Western Reserve University
Alexander Franz, Massachusetts Institute of Technology
Arturo A. Ayon, Sony Semiconductor

SESSION B1: APPLICATIONS/METROLOGY I

Chair: S. Mark Spearing
Monday Morning, November 26, 2001
Room 309 (Hynes)

8:30 AM ***B1.1**

MICROSENSORS FOR AUTOMOTIVE APPLICATIONS. G. Flik, H. Eisenschmid, C. Raudzis, H.-P. Trah Robert Bosch GmbH, Stuttgart, GERMANY.

According to market surveys automotive microsensors will evolve into a multi billion dollar business by 2005. Key roles play inertial sensors for passenger safety systems and mass flow and pressure sensors for engine and emission control systems. Thin film techniques together with silicon bulk or surface micromachining have been established as preferential production processes to achieve reduction of sensor size, weight and cost along with improvements of sensor functionality and reliability. Enhanced sensor performance often pushes the limits of process technology and therefore the need arises very early in the MEMS design process to identify materials and geometry related parameters which are critical with respect to their tolerance band specifications. In order to control these critical parameters, automated testprocedures need to be developed (based preferentially on electrical quantities) and additionally accounted for in the sensor design phase (design for test). In analogy to microelectronics, 2D wafermaps of critical parameters may give hints on how to improve process stability or how to adapt the sensor design in order to optimize yield. Examples of critical material parameter variations include stress (gradients), thermal conductivity and microstructure related filmresistance.

9:00 AM B1.2

AUTOMATED INTERFEROMETRIC MEASUREMENT OF MATERIAL PROPERTIES IN SURFACE MICROMACHINING. M.P. de Boer[a], N.F. Smith[b], M.B. Sinclair[c] and M.S. Baker[a], Sandia National Laboratories. [a]MEMS & Novel Si S&T Dept; [b]Radiation & Reliability Physics Dept; [c]Information Discovery, Extraction & Analysis Dept.

Knowledge of material properties in surface micromachining is critical for design, modeling, characterization and fabrication process control. Properties can vary across a wafer and from lot-to-lot. To address this issue, a new metrology system to accurately, rapidly, non-destructively and verifiably measure mechanical properties across an entire wafer has been developed. We first re-engineered the optics of a conventional microelectronics probe station. This has enabled speckle-free three-dimensional imaging while maintaining a one inch objective working distance, and standard electrical probes or probe cards can now be used for actuation. We have proceeded to map out mechanical properties of polycrystalline silicon (polysilicon) along a wafer column by the Interferometry for Material Property Measurement (IMaP) methodology. From the resulting interferograms of actuated beams, nanometer scale deflection profiles are obtained. These are analyzed by integrated software routines that extract basic mechanical properties such as cantilever curvature, Young's modulus and residual stress to precision levels of $\pm$ 0.1 m^{-1}, $\pm$ 5% and $\pm$ 0.25 MPa, respectively. Non-idealities such as support post compliance and beam takeoff angle are simultaneously quantified. Although deflections of cantilevers for an individual polysilicon layer varied across the wafer, Youngs modulus (E=161 GPa) is independent of wafer position as expected. This result is achieved because the non-idealities have been taken into account. However, stress gradient and residual stress were found to correlate with wafer position. In this work, we will further report material properties for three different layers of polysilicon across a wafer fabricated in our MEMS process. 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.

9:15 AM B1.3

THREE DIMENSIONAL THERMAL IMAGING IN MEMS DEVICES. Edward Van Keuren, John Currie, Makarand Paranjape, Georgetown University, Dept. of Physics, Washington, DC; Robert White, Thomas Schneider, SAIC, McLean, VA.

A three dimensional thermal imaging system is being developed for measuring temperature profiles in MEMS-biomedical devices. These devices rely on a thermal microablation of the dead-skin layer in order to sample transdermal fluids. This is accomplished using microheaters embedded into a PDMS microchannel device. In order to determine the proper functioning as well as long-term safety of the devices, a temperature profile of the device and the skin in contact with the heaters is needed. Using a three-dimensional chemical imagining microscope and temperature-dependent fluorophores, the temperature profile in a sample can be quantitatively determined. We demonstrate the technique on a number of model samples, and discuss extension to other applications such as thermal imaging in biological systems.

9:30 AM B1.4

PIEZOELECTRIC SHEAR MODE INKJET ACTUATOR. Jürgen Brünahl[a,b], Alex M. Grishin[a], Sergey I. Khartsev[a], and Carl Österberg[a]. [a]Division of Condensed Matter Physics, Department of Microelectronics and Information Technology, Royal Institute of Technology, Stockholm, SWEDEN; [b]Department of Advanced Manufacturing Technologies, XaarJet AB, Jarfalla, SWEDEN.

This paper provides an overview of different inkjet technologies such as continuous jet and drop-on-demand systems, whereat main attention is turned on piezoelectric systems particularly shear mode inkjet printheads. Their complex structures micromachined in bulk PZT ceramics with ink channel widths of 75 μm and depths of 360 μm make them a prominent example for microelectromechanical systems (MEMS). Detailed information about manufacturing and principles of operation are given. Several techniques to control manufacturing processes and to characterize properties of the piezoelectric materials are reviewed. Among them dielectric spectroscopy to obtain dielectric permittivity and Curie temperature, ferroelectric hysteresis P-E loop tracing to deduce remnant polarization and coercive field, and a novel pulsed technique[1] to quantify functional properties of the PZT actuator such as acoustic resonant frequencies and electromechanical coupling coefficient. We discuss how inkjet actuator performance is affected by heat and high voltage treatment.

[1]'Dielectric and pulsed spectrosocpy of shear mode PZT microactuator', Jürgen Brünahl, Alex Grishin, Sergey Khartsev, in Materials Science of Microelectromechanical Systems (MEMS) Devices III, edited by M. deBoer, M. Judy, H. Kahn, S.M. Spearing, Mat. Res. Soc. Proc. 657, EE4.6 (2000).

9:45 AM B1.5

MECHANICAL STIFFNESS ANALYSIS OF NICKEL AND SILICON SPRING SUPPORTED MEMS STRUCTURES ACROSS WAFER LOTS AND AFTER THERMAL CYCLING AND HUMIDITY EXPOSURE. Wade Babcock[a], Kevin Cochran[b], Michael Deeds[b], Gabriel Smith[b], Lawrence Fan[b]. [a]IIT Research Institute, Rome, NY, under contract to Naval Surface Warfare Center Indian Head Division (NSWC/IHD); [b]Naval Surface Warfare Center Indian Head Div, Indian Head, MD.

The Naval Surface Warfare Center Indian Head Division is executing a development program to deploy a MEMS-technology-based Fuze/Safety & Arming device in the 6.25-inch Countermeasure Anti-Torpedo (CAT) torpedo. The torpedo will provide the WSQ-11 Torpedo Defense System hardkill capability against aggressor torpedos. This program requires quality control and process assurance procedures of fabricated MEMS components. A method to measure the mechanical response of LIGA-nickel and DRIE single crystal silicon MEMS spring-supported structures has been refined and applied in these quality and process control efforts. Examination included structures within single fabrication runs as well as additional structures which were put through heat and humidity exposure and thermal cycle testing. Mechanical stiffness was seen to vary from wafer to wafer within single fabrication runs of LIGA-nickel structures, and these structures stiffness was also affected by thermal and humidity exposure. Stiffness of single crystal silicon structures is also characterized with temperature and humidity exposure.

SESSION B2: MECHANICAL PROPERTIES I
Chair: Maarten P. de Boer
Monday Morning, November 26, 2001
Room 309 (Hynes)

10:30 AM *B2.1
SIZE EFFECTS IN MATERIALS FOR MEMS DUE TO MICROSTRUCTURAL AND DIMENSIONAL CONSTRAINTS. Oliver Kraft and Eduard Arzt, Max-Planck Institut, Stuttgart, GERMANY.

Effects of size on predominantly mechanical properties of materials are reviewed at a first order level. Microstructural constraints, e.g. due to grain boundaries and dimensional constraints in small scale structures such as microfabricated thin films are distinguished. Phenomena addressed include the yield and fatigue response of thin films in microfabricated structures. Important aspects can be understood from the point-of-view of the intereaction of a characteristic length (such as the dislocation radius of curvature) with a size parameter (grain size or film thickness). It is demonstrated that such an approach can reveal interesting analogies between otherwise very different properties of materials.

11:00 AM B2.2
MICROSTRUCTURE, HARDNESS, AND CONTACT RESISTANCE OF THIN GOLD FILMS. Beth L. Pruitt, Thomas Kenny, Stanford University, Dept of Mechanical Engineering, Stanford, CA; Dae-Han Choi, William Nix, Stanford University, Dept of Materials Science and Engineering, Stanford, CA.

The continued miniaturization of IC's, interconnects, relays, and packaging depends critically on the electrical and mechanical properties of the thin film metallization used in low-force electrical contacts. To evaluate contact resistance at forces in the nN to mN range, we have designed, fabricated, and characterized a micromechanical force sensor integrated with 4-wire electrical contact characterization capability. The force sensors are piezoresistive single crystal silicon cantilevers with contact leads and metallization patterned to the tip of the cantilever. With these sensors, electrical contact resistance can be measured simultaneously with displacement and contact force. The other half of the contact pair is a spherical tip with identical metallization coating its surface. The spheres are glass calibration spheres, reflowed gold wirebond wire, or reflowed resist to provide varied substrate properties. These data are useful when combined with measurements and analysis of the morphology, microstructure, and materials properties through nanoindentation, scanning electron microscopy (SEM), atomic force microscopy (AFM), and focused ion beam (FIB) sections. Gold films are deposited by sputtering, evaporating, or plating directly onto the cantilevers using standard IC fabrication processes. The gold thickness varies from about 100 nanometers to 4 microns and is isolated by 250 Angstroms titanium and 250 Angstroms platinum acting as an adhesion and barrier layer. These manufacturing methods provide variation in microstructure, surface morphology, and materials properties which can then be correlated with contact resistance behavior. Initial results indicate sizable differences in behavior for different methods of manufacture and varying film thickness. Contact resistance will be characterized as a function of these observed differences in materials properties.

11:15 AM B2.3
MECHANISMS OF FATIGUE IN POLYSILICON MEMS STRUCTURES. P. Shrotriya, S. Allameh, J. Lou, Princeton University, The Princeton Materials Institute and The Department of Mechanical and Aerospace Engineering, Princeton, NJ; A. Butterwick, University of Michigan, Department of Applied Physics, Ann Arbor, MI; S. Brown, Exponent Failure Analysis and Associates, Natick, MA; and W.O. Soboyejo, Princeton University, The Princeton Materials Institute and The Department of Mechanical and Aerospace Engineering, Princeton, NJ.

This paper presents the results of recent experimental and theoretical studies of fatigue in polysilicon MEMS structures. Fatigue crack initiation is shown to be associated with the stress-assisted evolution of a surface silica layers that forms during the normal exposure of unpassivated polysilicon surfaces to lab air (relative humidity of $\sim$70%). In-situ atomic force microscopy techniques are used to reveal the evolution of grain boundary triple points (micro-groove geometries) and overall surface topography during incremental cyclic deformation to failure. The conditions for unstable micro-groove evolution are also predicted using linear perturbation analyses that predict the growth or decay of topographical wavelengths. The predictions from the perturbation analyses are shown to be consistent with Fourier descriptions of the measured surface topographies obtained via atomic force microscopy. Finally, the measured fatigue crack growth rates in polysilicon are compared with those obtained from compression/compression and tension/tension fatigue tests on bulk silica glass.

11:30 AM B2.4
SIZE EFFECTS DETERMINED FROM TENSILE TESTS OF PERFORATED MEMS SCALE SPECIMENS. Ioannis Chasiotis, University of Virginia, Department of Mechanical and Aerospace Engineering, Charlottesville, VA; Wolfgang G. Knauss, California Institute of Technology, Graduate Aeronautical Laboratories, Pasadena, CA.

A systematic study of small-scale specimen size effects has been performed on elliptically perforated specimens with minimum radius of curvature of 1 micron. This study aimed at assessing the dependence of failure stress at the tip of a notch on: (a) varying stress concentration for constant radius of curvature, (b) decreasing radius of curvature of micro-notches relative to the material grain size. The experimental results demonstrate a strong influence of the decreasing notch radii on the failure strength of MEMS scale specimens, while the effect of variation of the stress concentration factor is rather insignificant. The local failure strength at the tip of a notch increases when the radius of curvature becomes smaller, which is in accordance with the probabilistic nature of failure. When the notch radius becomes as small as 1 micron (three times larger than the grain size) then a strong size effect is observed. This effect becomes moderate for larger radii of curvature, up to 8 microns (25 times the grain size), when the failure stress at the notch tip almost reaches the value of the tensile strength of the material as this has been recorded in tensile tests of microfabricated specimens.

11:45 AM B2.5
PLASTICITY LENGTH SCALE AND DISLOCATION SUBSTRUCTURES IN LIGA NICKEL MEMS STUCTURES. J. Lou, P. Shrotriya, S. Allameh, Princeton University, The Princeton Materials Institute and The Department of Mechanical and Aerospace Engineering, Princeton, NJ; N. Yao, Princeton University, The Princeton Materials Institute, Princeton, NJ; T. Bucheit, Sandia National Laboratories, Mechanical Reliability and Modeling Dept., Albuquerque, NM; W.O. Soboyejo, Princeton University, The Princeton Materials Institute and The Department of Mechanical and Aerospace Engineering, Princeton, NJ.

The plasticity length scale parameters associated with rotational and stretch gradients in LIGA nickel MEMS structures are presented in this paper. These are obtained from micro-bend experiments on LIGA nickel foils with different thicknesses. The underlying dislocation substructures associated with known strain gradients are then elucidated by comparing the remnant dislocation substructures in plastically bent foils (extracted using focused ion beam techniques) with those in the gauge sections of tensile specimens deformed continuously to failure under pure axial loading. The implications of the results are discussed for the modeling of plasticity in LIGA nickel structures.

SESSION B3: MICROSTRUCTURE AND PROCESSING
Chair: Harold Kahn
Monday Afternoon, November 26, 2001
Room 309 (Hynes)

1:30 PM *B3.1
FROM MEMS TO NEMS IN THE NEXT 15 YEARS. Marc J. Madou, Nanogen, Inc., Advanced Technology, San Diego, CA.

The combination of natural polymers such as proteins and nucleic acids with MEMS and NEMS promises the advent of a totally new class of devices such as sensors and actuators with applications in diagnostics, responsive drug delivery, biocompatibility, self-assembly etc. Proteins and nucleic acid are information rich molecules with structural and electrical properties making their incorporation in the human manufacturing arsenal an attractive proposition. This combination has become possible today as both top-down traditional manufacturing (e.g., MEMS and NEMS) and novel bottom-up manufacturing can realize components overlapping in size. Examples to illustrate the tremendous potential of merging top-down and bottom-up manufacturing techniques will be presented. These examples are culled from the fields of molecular diagnostics, responsive drug delivery systems, protein and DNA structural elements and sensors and actuators and molecular self-assembly. In molecular diagnostics, we conclude that an important avenue to success is the merging of DNA arrays with microfluidics to achieve

sample to answer systems. Widespread introduction of molecular diagnostics is not about if but when.
Future responsive drug delivery systems are seen as a culmination of results from genomics and proteomics coupled with implantable telemetric devices. This and other developments will cause a renewed interest in $in-vivo$ diagnostics. Natural polymers will become part of our manufacturing arsenal and, when using building blocs in the nanometer range, a fundamental understanding and the use of molecular self-assembly is a must for future progress. While biomimetics in the macrodomain has often led to failure in the past (airplanes do not flap their wings as birds do, see Icarus legend), we believe that biomimetics in the nanodomain will succeed. Nature indeed has worked much longer on arriving at a biological cell than it did at making birds, trees or humans: nature excels at engineering in the nanodomain.
While top-down manufacturing approaches will continue to prevail over the next two decades, we will start seeing hybrid solutions, such as the use of flexible materials (e.g., hydrogels) rather than stiff building materials (e.g., steel and Si). There will be more emphasis on non-Si, modular and "beyond batch" techniques such as pick and place, drop delivery, lamination, etc. The next big breakthrough will be continuous manufacturing, perhaps rendered possible through molecular self-assembly (bath fabrication rather than batch fabrication).

2:00 PM B3.2
BONDING OF BULK PIEZOELECTRIC MATERIAL TO SILICON USING A GOLD-TIN SOLDER BOND. Kevin T. Turner, Massachusetts Institute of Technology, Cambridge, MA; Richard Mlcak, Boston Microsystems, Woburn, MA; David C. Roberts, S. Mark Spearing, Massachusetts Institute of Technology, Cambridge, MA.

A class of MEMS devices, which utilizes microfabrication technology and bulk piezoelectric material, is currently being developed to produce high power density transducers. A thin-film gold-tin eutectic solder bond has been developed to bond bulk piezoelectric elements to microfabricated silicon structures in these devices. A 4.3 μm thick multilayer film structure, which consists of a titanium adhesion layer, platinum diffusion barrier, a gold-tin alloy (80 wt%Au - 20 wt% Sn) solder layer and a pure gold capping layer, was sputter deposited on the piezoelectric components to be bonded. Bonding was accomplished by mating the piezoelectric components with metallized silicon components and heating to approximately 300°C in a reducing atmosphere. The bonding technology allows thin, electrically conductive bonds to be formed between dissimilar materials with minimal amounts of applied pressure during bonding. Successful bonding has been achieved between single crystal silicon and polycrystalline lead-zirconate-titanate (PZT-5H) as well as silicon and single crystal lead zinc niobate-lead titanate (PZN-PT). The process was optimized to produce mechanically robust, void-free bonds. The absence of voids was verified through scanning electron microscope examinations of bond cross- sections. Tensile tests conducted on representative structures indicated that fracture of the piezoelectric material occurred prior to bond failure. In addition, preliminary results from operational test devices suggest the bonds are mechanically sound. Details of the bonding process and bond characterization will be presented.

2:15 PM B3.3
AN EMPIRICAL COMPOSITION MAP FOR DESIGNING Ni-Mn-Ga FERROMAGNETIC SHAPE MEMORY ALLOYS. Xue-Jun Jin, Robert C. O'Handley, and Samuel M. Allen, Massachusetts Institute of Technology, Dept of Materials Sciencs and Engineering, Cambridge, MA; Xu Zuyao, Shanghai Jiao Tong University, Shanghai, CHINA.

Ni-Mn-Ga ferromagnetic shape memory alloys (FSMA) are a potential new class of actuator materials that deform under a magnetic field by the motion of twin boundaries in the ferromagnetic martensite phase. Compared with a traditional thermally-actuated shape memory alloy, the significance of Ni-Mn-Ga FSMA that it is able to respond at higher frequencies (at least 300 Hz) with comparable strains (up to 6%) in a moderate field (below 1 T). The fundamental parameters: saturation magnetization, Ms, Curie temperature, Tc, and martensitic temperature, Tmart are critical to acquire the ferromagnetic martensite phase so that large magnetic-field-induced strains can be achieved in such alloys. It is well known that these parameters are strongly dependent on the composition of the alloy. In this report, the composition dependence of Curie temperature, Tc, saturation magnetization, Ms, and martensitic transformation temperature, Tmart, for Ni-Mn-Ga ferromagnetic shape memory alloys has been empirically outlined in the composition range of Mn: 20-35 at % and Ga: 18-27 at %. The results reveal that Ga has the strongest impact on Tmart, which is also the most sensitive parameter to contents among three critical parameters. Finally, a concise composition map for designing Ni-Mn-Ga FSMAs is introduced and a composition range of alloys with Ms higher than 60 emu/gm for room operation temperature (300 K) has been recommended.

2:30 PM B3.4
CHARACTERIZATION OF ELECTRODEPOSITED Ni-Fe FOR LIGA MEMS COMPONENTS. T.E Buchheit, P.T. Kotula, P.F. Hlava, R.P. Janek and T.R. Christenson, Sandia National Laboratories, Albuquerque, NM.

Electrodeposition remains the primary method for depositing material into PMMA molds fabricated via the LIGA process. As a result, materials such as pure nickel and copper with successful electroplating histories have become primary candidates as structural material for LIGA components. Comparatively few alloys have successful electroplating histories since alloy deposits, which require simultaneous deposition of at least two materials, are often highly stressed. Among binary alloys that have been successfully electroformed for LIGA applications are those from the Ni-Fe system. This paper discusses the characterization and corresponding mechanical properties of pulsed plated and direct current plated Ni-Fe alloys. Although little difference was observed in grain structure and crystallographic texture between pulsed plated and DC plated samples, significant compositional modulation across each pulse was isolated with x-ray spectral image analysis. Through SEM microprobe analyses and small scale mechanical testing, electroformed samples were found to embrittle due to co-deposited sulfur migrating to grain boundaries when subjected to heat treatments between 300°C and 700°C. Conversely, samples heat treated at temperatures above 1000°C were found to have recovered tensile ductility to values greater than the as deposited samples. The effects of sulfur migration on the embrittlement of these electroformed alloys will be discussed in light of literature data on the behavior of bulk polycrystalline and electroplated nickel alloys which contained sulphur. *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*

SESSION B4: APPLICATIONS
Chair: David F. Moore
Monday Afternoon, November 26, 2001
Room 309 (Hynes)

3:15 PM *B4.1
LEVERAGING MEMS MANUFACTURING EXPERIENCE TO BUILD THE NEXT GENERATION. Jack Martin, Analog Devices, Inc., Micromachined Products Division, Cambridge, MA.

Recent market changes have been dramatic. MEMS accelerometers now dominate the automobile air bag sensor market. Digital image projection based on MEMS mirror arrays is also well established. Volume production of solid state gyros, MEMS components for optical communication networks, MEMS relays and MEMS electrical isolators are also imminent. The span of this near term growth is impressive. It extends from unpackaged MEMS die suitable for direct assembly on circuit boards to devices molded in small plastic SO packages to large die (several square centimeters) assembled in hermetic packages with optical windows. Each product class, as well as each package type has unique reliability and durability requirements. The current MEMS growth spurt is the natural result of a maturing industry that has learned to build durable products at high volumes and competitive prices. Success is largely based on standard IC design tools and processes, a fundamental understanding of materials/stresses, unusual manufacturing diligence and process innovations that solve MEMS-specific problems. Durability requires control of many factors. Particulates are an obvious example. However, MEMS devices have high surface/volume ratios so stiction, surface charges and ambient gas interactions are equally important, although less predictable. In practice, durable MEMS products must have surfaces that are passivated or otherwise controlled. This is a particular challenge for temperature sensitive devices because few low temperature treatment processes are production-worthy. This presentation will examine some of the durability challenges in MEMS products with a primary focus on surface related issues.

3:45 PM B4.2
THE EFFECTS OF ION IMPLANTATION ON DISSIPATION IN SILICON NANOMECHANICAL RESONANT STRUCTURES. L. Sekaric, M. Zalalutdinov, J.M. Parpia, H.G. Craighead, Cornell Center for Materials Research, Cornell University, Ithaca, NY.

The interest in the effects of ion-implantation on dissipation in MEMS and NEMS is mainly application driven. The cost advantage of MEMS and NEMS is their small size and integrability with electronic devices. The integration and access to the NEMS high-frequency range requires desired conductivity of the structure and/or the substrate which is usually achieved by either metallization or ion doping. The major drawback to successful applications of NEMS to date has been relatively low mechanical quality factor (Q) values even in single-crystal silicon structures. The Q has been found to be on the order of 10^3 at room temperature to 10^4 at low temperature. It has also been found that amorphous metal layers lower the Q significantly thus dopant implantation is a viable tool for customizing the electronic properties of the substrate provided that the implantation-induced damage can be repaired and the highest possible Q restored. We have studied the effects of boron implantation on the mechanical Q factor in single-crystal silicon nanomechanical resonant structures. The devices studied are $2 \times 2\,\mu$m single paddles with supporting beams 1.5–5 μm long and 100–300nm wide and 200nm thick. The paddles have resonant frequencies of 4–25MHz excited via a piezo drive (to avoid any differences in the resistive losses). Upon implantation we find a decrease in the Q factor which shows a strong dependence on the amount of damage of the bulk. For example, a 70 keV implantation with the range the entire thickness of the device results in a six-fold decrease of Q. Annealing is used to repair the damage induced. Anneals in a furnace, at 800°C, 900°C and 1000°C were done in nitrogen for 35 min. The 800°C anneal restores the Q factor completely in all cases whereas the 900°C and 1000°C anneals do not result in a further improvement of the Q factor. In addition, an HF dip following an anneal improves the Q even further, which gives us a Q factor of about 10^4. Currently we are studying the dose dependence and different thermal treatments. Low-temperature studies should enable us to identify lattice processes and their contribution to the internal friction while probing at relatively high resonant frequencies available by working with devices in this size range.

4:00 PM B4.3
ELECTRO-MECHANICAL COUPLING AND POWER GENERATION IN A PZT MICROENGINE. D.F. Bahr, K.R. Bruce, B.W. Olson, L.M. Randall, C.D. Richards, and R.F. Richards, Mechanical and Materials Engineering, Washington State University, Pullman, WA.

A MEMS power generation system, the P3 microengine, has been developed using a lead – zirconate – titanate (PZT) film on a bulk micromachined silicon wafer. Electrical power generation using this flexing piezoelectric membrane of PZT has been demonstrated using PZT films with a variety of thicknesses and electrode structures. A method for extracting piezoelectric constants and coupling coefficients while characterizing the mechanical response of the membranes from a bulge testing system at large deflections (in the non-linear pressure deflection regime) is developed. As the grain size and orientation (and dielectric constant) are altered by the underlying materials, the efficiency and power density for converting mechanical work to electrical work is dependant on these parameters. In the current study, the effects of processing variables such as interfacial roughness and thermal treatments are correlated to the power generated using mechanical bulge testing of a bulk micromachined membrane coated with between 300 and 900 nm of PZT using a solution deposition process. These processing parameters are also correlated to the resulting structure of the film. Demonstration of membranes which generate 750 mV and 0.1 mW will show that by controlling the roughness, chemistry, and grain size of the PZT significant power generation can be achieved using standard batch processing methods.

4:15 PM B4.4
ASSEMBLY AND INTEGRATION OF RF-RANGE NANO-ELECTROMECHANICAL SYSTEMS. Stephane Evoy, Ben Hailer, Martin Duemling, and Sanjay Raman, Bradley Department of Electrical and Computer Engineering, Virginia Tech, Blacksburg, VA; Benjamin R. Martin and Thomas E. Mallouk, Department of Chemistry, The Pennsylvania State University, University Park, PA; Irena Kratochvilova and Theresa S. Mayer, Department of Electrical Engineering, The Pennsylvania State University, University Park, PA.

Recent advances in surface nanomachining have allowed the fabrication of mechanical structures with dimensions reaching 20 nm, and resonant frequencies in the 100s of MHz. Such nano-electromechanical systems (NEMS) are of interest for the development of highly sensitive sensors, and the integration of low power RF-range nanomechanical signal processors in RF microsystems. Carr, Evoy et al. have already reported Si NEMS with record resonant frequencies of 380 MHz. [1] However, we have also established that native oxide and/or other damaged layer severely degrades the performance of these devices. [2] These nanostructural issues and their related dissipative processes prevent the "top-down" surface machining of GHz range nanomechanical resonators. Gigahertz-range systems are rather to be bestowed by "bottom-up" nanomanufacturing technologies.
We report the surface assembly of RF-range NEMS. Using an electrostatic technique developed by Smith et al., [3] we have successfully positioned Rh mechanical beams onto specific sites of a silicon electronic circuit. With diameters as small as 250 nm and lengths varying from 2 to 3 um, preliminary results show mechanical resonances ranging from 5 MHz to 80 MHz, and quality factors as high as 1000.
Compatible with established CMOS processes, this technique bridges the gap between bottom-up nanotechnology and microsystems. First and foremost, it lifts the materials limitations inherent to surface nanomachining, enabling the integration of resonators with engineered nanostructural properties. We will therefore report the development and assembly of mechanically optimized NEMS, and present strategies for their integration in RF microsystems. [1] D.W. Carr, S. Evoy, L. Sekaric, J.M. Parpia, and H.G. Craighead, APL. 75, 920 (1999). [2] S. Evoy, D.W. Carr, A. Olkhovets, L. Sekaric, J.M. Parpia, and H.G. Craighead, APL. 77, 2397 (2000). [3] P.A. Smith, C.D. Nordquist CD, T.N. Jackson, T.S. Mayer, B.R. Martin, J. Mbindyo, and T.E. Mallouk. APL, 77, 1399 (2000).

4:30 PM B4.5
MICROMACHINED NANOPARTICULATE CERAMIC GAS SENSOR ARRAY ON MEMS SUBSTRATES. M. Heule, L.J. Gauckler, ETH Zurich, Dept of Materials, Zurich, SWITZERLAND.

In most MEMS applications, dust and particles are avoided with considerable endeavour. However, for many applications such as gas sensors, powders of functional ceramics would often provide better performance than corresponding thin film layers. There are abundant ways to synthesize powders with well defined chemical composition, phase and size distribution, whereas the processing parameters for thin-film preparation often are limited. Specifically where the functionality is based on a chemical reaction on surfaces, nanoscaled powders with a high specific surface area have proven useful. This is the case for tin oxide gas sensors that exhibit a change in electrical resistivity induced by the catalytic combustion of the analyte gas. Soft lithography as well as conventional photolithography were used for the fabrication of mesoscaled powder-based ceramic structures. In this paper, we present the integration of small tin oxide micro-structures (10 by 30 μm^2) on a micro-hotplate substrate. Such a substrate can heat the ceramic sensor to operating temperatures quickly with low power consumption. A whole array of sensors can be integrated on one micro-hotplate. Processing steps to prepare the sensor array on the micro hot plate are presented and discussed concerning processing sequence, sensitivity towards the analyte gas and power consumption. Also, problems arising from the introduction of ceramic powders in a MEMS fabrication process are addressed.

4:45 PM B4.6
STRENGTH ANALYSIS OF A MICRO-ROCKET COMBUSTION CHAMBER. Erin E. Noonan, Christopher S. Protz, Yoav P. Peles, S. Mark Spearing, Massachusetts Institute of Technology, Department of Aeronautics and Astronautics, Cambridge, MA.

The micro-rocket is one of several MEMS power generation devices currently under development at MIT. The performance of the micro-rocket is currently limited by structural failures of the combustion chamber at pressures below the design pressure of 125 atmospheres. The maximum pressure achieved in a cold test is 40 atmospheres. Hot tests have yielded a maximum pressure of 12.5 atmospheres. The cold test failures suggest that even with improvements to the combustion chamber cooling, the device will continue to fail unless changes are made to the structure itself. The strength of silicon structures is critically dependent on the flaw population of the material. These strength-controlling flaws are most commonly introduced during processing and handling. Flaws that commonly introduce stress concentrations include insufficient fillet radii, surface roughness, wafer misalignment, and bad wafer bonds. To identify the most significant weakness in the structure, strength data must be collected from a statistically significant sampling of devices.[1] This paper will present the fabrication, testing and analysis of the micro-rocket chamber structure. The structure is a rocket chamber with a closed throat and pressure feeds fabricated with processes from the full micro-rocket builds. Mechanical test results will be correlated with inspection of failed devices for flaws and finite element modeling of the test condition. This analysis will guide recommendations for modifications to improve the strength of the micro-rocket chamber. These recommendations will be discussed in the context of the current status of the micro-rocket. [1]. K.-S. Chen, A. Ayon, and S.M. Spearing, "Controlling and Testing the Fracture Strength of Silicon on the Mesoscale", J. Am. Ceram. Soc., vol. 20, no. 6, pp. 1476-84, 2000.

SESSION B5: POSTER SESSION
Chairs: Arturo Ayon, Thomas E. Buchheit, Harold Kahn and S. Mark Spearing
Monday Evening, November 26, 2001
8:00 PM
Exhibition Hall D (Hynes)

B5.1
SURFACE MICROMACHINED POLYSILICON COMPONENTS CONTAINING CONTINUOUS HINGES AND MICRORIVETS USED TO REALIZE TRUE THREE-DIMENSIONAL MEMS STRUCTURES. Edward Kolesar, Matthew Ruff, William Odom, Simon Ko, Jeffrey Howard, Peter Allen, Richard Wilks, Josh Wilken, Jorge Bosch, Noah Boydston, Texas Christian University, Dept of Engineering, Fort Worth, TX.

A new polysilicon surface micromachining technique for realizing three-dimensional structures has been developed. Single-layer polysilicon elements and laminated polysilicon panels with trapped-glass reinforcement ribs have been successfully attached to a silicon substrate with robust and continuous hinges that can be rotated out-of-plane and assembled. One of the elevatable panels are terminated with an array of open windows, and a matching set of microrivets with flexible barbs protrudes from the other rotatable element. Because the microrivet barb tip-to-barb tip separation is larger than the opening in the matching window, the barbs flex as they pass through the open window and result in a permanent latch condition and a three-dimensional structure. Three novel microrivet designs have been micromachined to facilitate the latching process, including a simple arrowhead design, a high-aspect ratio shank-to-barb length variant, and a rivet-like structure with a split shank and a hemispherical shaped cap. To minimize panel breakage after the sacrificial glass release etch process and facilitate alignment during assembly, a network of sacrificial electrothermally-actuated mechanical links ('fuses') have been integrated into the design to promote robust and stable three-dimensional MEMS structures.

B5.2
A TWO INPUT PARTS' RECTANGULAR-BAR-SHAPED MULTILAYER PIEZOELECTRIC TRANSFORMER OPERATING IN THE THIRD SYMMETRIC EXTENSIONAL MODE. Yu Zhang, Jian Sun, Jun HuiHu, Helen Lai Wah Chan, Department of Applied Physics, Material Research Center, The Hong Kong Polytechnic University, HungHum, Kowloon, Hong Kong, CHINA.

In this study, a two input parts' rectangular-bar-shaped multilayer piezoelectric transformer operating in the third symmetric extensional vibration mode is proposed, and its characteristics are investigated. The transformer consists of a two input parts' multilayer piezoelectric (lead zirconate titanate-PZT) ceramic bar with a high mechanical quality factor which has a length of 31 mm, a width of 6 mm and a thickness of 1.6 mm. The transformer is poled along the thickness direction, but at the input section, due to its multilayer structure, each neighboring ceramic layer was poled conversely. The surfaces of the transformer's up and down sides are divided to three parts individually. On the one hand, the central part is painted by a pair of rectangular external electrodes, and serve as the output part electrodes. On the other hand, the two edges of the ceramic bar are also painted by the rectangular shaped external electrodes of the same size on its up and down surfaces. These external electrodes are connected to the internal electrodes of the input parts of the transformer with a designed structure. The external electrodes of the input part and the output part are separated by narrow gaps. For a temperature rise of 20 Celsius degree, the transformer has an output power of 3.48 W and a power density of 11.7W/cm^3. With a matching load resistance of 3.42KW, its maximum efficiency is 87.2% the maximum voltage gain is 7.6. Compare with single layer's length vibration mode piezoelectric transformer, the voltage gain has magnificently increased, and output power density has also increased very much. However, the power transmission efficiency of this transformer is still slightly less than the commonly used Rosen type single layer piezoelectric transformer, this means some further detailed adjustment on the fabrication work of this multilayer device still need to be done. Key word: Multilayer piezoelectric transformer; External electrode; Internal electrodes; PZT ceramic bar; The third symmetric extensional vibration.

B5.3
FERROMAGNETIC NiMnGa AND CoNiGa SHAPE MEMORY FILMS. Manfred Wuttig, Corneliu Craciunescu, J. Cullen, L.V. Saraf and R. Ramesh, Dept. of MS&E, U. of Maryland, College Park, MD.

The paper presents recent work on ferromagnetic NiMnGa and CoNiGa films sputter and/or pulsed laser deposited on silicon cantilever substrates as well as GaAs. NiMnGa films deposited at RT and subsequently annealed and films deposited on substrates held above 385°C show a transformation comparable to NiTi shape memory films. Measurements of the temperature dependence of the change of the elastic modulus as a function of the magnetic field of a nominally $Ni_{50}Mn_{25}Ga_{25}$ film suggest that the magnetic anisotropy field increases proportional to the transformation strain, (c/a-1), from 0.3 T at 50°C to 0.8 T at -150°C. Ferromagnetic Co_2NiGa Heusler alloys pulsed laser deposited at 500°C grow epitaxially on (001) GaAs substrates. Structural measurements reveal the films to be highly [001] oriented with a considerable degree of in-plane cube-on-cube orientation. The magnetization of the films at constant field displays a sharp drop at a critical temperature that depends on the film thickness occurring at 125 K and 225 K for a 3000 Å and 7000 Å thick films respectively. This anomaly represents the martensitic transformation in the films, which is increasingly suppressed by coherency strains as their thickness decreases.

B5.4
FILM ELECTROSTATIC MICRORELAY ON THE BASE OF INORGANIC ELASTIC DIELECTRIC MATERIALS. Ivan L. Grigorishin, Ludmila G. Polevskaya, Michael E. Stelmakh, Vacuum Microelectronic Laboratory, Institute of Electronics, Belarus Academy of Science, Minsk, BELARUS.

In this article methods of creation of electrostaticly control microcommutative film devices on the base of inorganic elastic dielectric materials are proposed. Two types of electromechanical microdevices are discussed. The first is film electrostatic microrelay with the cantilever switch element formed on thin (10-20 micro-meters) elastic substrate of amorphous alumina with operate and commutative electrodes. Unmovable element of microrelay with operate and commutative electrodes is formed on the microrelief alumina substrate. In central part of the latter microrelay there is movable element formed on the base torsion suspensions. Two independent groups of operate and commutative electrodes are located on unmovable and movable elements. This microrelay operates on polarized principle. If operate electrodes are separated by dielectric of movable element, this microrelay is characterized by two stable states during its work. Presence of deep traps in dielectric leads to that injected charge keeps movable element in the attracted condition long time which can be from some months to some years in the case of vacuum operation of microrelay. Switching of the microrelay in other state is carried out by the applying of voltage at the other operate electrodes. Devices are formed by batch technology. Power consumption is units of microwatt and operate voltage is 30-80 V in depending on design of microrelay.

B5.5
ALUMINUM - SILICON AND GOLD - SILICON EUTECTICS: NEW OPPORTUNITIES FOR MEMS TECHNOLOGIES. Ciprian Iliescu, Poenar Daniel Puiu, Miao Jianmin, Nanyang Technological University, SINGAPORE.

It is known that aluminum and gold can be used as thin adhesion layers in wafer-to-wafer bonding. Generally, the main advantage of this technology lies in the lower temperature which can be used for bonding (compared to the typical range of silicon fusion bonding: 800-1100°C). In this case, the bonding temperatures are between 600 and 650°C for aluminum and around 450°C for gold, respectively. The bonding mechanism relies on the formation of Al-Si or Au-Si eutectics at the interface of the wafers, which are developed at 577°C for Al-Si, or about 363°C for Au-Si, respectively. Other advantages of these eutectic-based bonding are their very good hermeticity and reliability. Our practical experiments also highlighted that Al-Si and Au-Si eutectics present good resistance to anisotropic wet etchants of Si (e.g. KOH and TMAH) and to the isotropic one (HF/HNO_3 solution), too. Therefore, the resulting interfacial eutectics could be used as etch-stop layers in wet etching-based bulk micromachining. The paper also presents how Al-Si and Au-Si eutectic layers can be used for such applications, as well as for the fabrication of a high-pressure sensor.

B5.6
NOVEL, SENSITIVE, AND INEXPENSIVE POROUS SILICON GAS SENSOR. Lenward Seals, Laam Angela Tse[a], Peter J. Hesketh and James L. Gole, School of Physics, Georgia Institute of Technology, Atlanta, GA. [a]School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, GA.

The development of a sensitive porous silicon (PS) gas sensor, which utilizes electroless metallization as a means to obtaining a highly efficient electrical contact has been demonstrated for the detection of HCl, NH_3, and NO at the 10 ppm level. Preliminary measurements have also been made on Glucose. The problem of spreading resistance (kΩ - MΩ) normally associated with PS is overcome as low surface and contact resistance contacts ~40Ω are made to the mesoporous PS surface structure through electroless gold plating. This technique is based on photoreduction at the PS surface precipitated by a "long-lived" surface bound fluorophor. The resulting sensor response is rapid and sensitive, and the device has an extremely rapid recovery time. It might be easily integrated into an electronic measurement system. The created sensor relies on our ability to stabilize the "long-lived" PS photoluminescence which is subsequently used to catalyze the formation of a low resistance connection to the surface.

B5.7
ELEVATED-TEMPERATURE TENSILE, CREEP AND

LONG-TERM FATIGUE BEHAVIOR OF LIGA Ni MEMS STRUCTURES. H.S. Cho, K.J. Hemker, The Johns Hopkins University, Departments of Mechanical Engineering and Materials Science and Engineering, Baltimore, MD; Y. Desta, K. Lian, J. Goettert, Louisiana State University, Center for Advanced Microstructures & Devices (CAMD), Baton Rouge, LA.

Room temperature indentation and micro-tensile measurements of LIGA Ni MEMS scale structures have been reported, but a full balance of mechanical properties is still lacking for this class of materials. The present study was undertaken to characterize the elevated temperature tensile and creep strengths and the long-term fatigue performance of electroplated LIGA Ni structures. Special attention has been focused on the importance of microsample geometry, electroplating conditions and the influence of elevated temperature exposure and cyclic loading. Free-standing LIGA Ni microsamples have been fabricated at CAMD and tested using the microsample test methodology developed at JHU. Preliminary results suggest that the underlying creep and fatigue behavior of LIGA Ni is similar to what is observed in coarse grained Ni, but that the importance of specimen geometry cannot be overlooked. A measurable decrease in strength has been evidence at widths of less than 100μm, and the fatigue resistance was found to be lowered by the presence of a shoulder in one set of specimens. These findings are expected to have profound consequence on the design and reliability of MEMS-based structures, and the present paper will expand on these observations.

B5.8
ON THE UNIFICATION OF MATERIAL STRENGTH TESTING FOR MEMS APPLICATIONS. Kuo-Shen Chen, National Cheng-Kung Univ, Dept of Mechanical Engineering, Tainan, TAIWAN.

The information of material strength is critical for supporting microelectromechanical system (MEMS) design. Due to strong length-scale effect and process dependence, material strength data tested at conventional scale usually could not be directly applied for MEMS applications. As a result, considerable amount of research activities have been conducted in material strength characterization for MEMS during the past decade. It is still a major research topic in the future development of MEMS. However, unlike material testing at conventional scale, which has established standard procedures, the MEMS material strength characterization is not unified at this moment. The lack of standard testing procedures makes it difficult to form a material database; as a result, considerable effort has been wasted in performing properties and strength testing for same material by different test methods. To make things even worse, different testing procedures usually report considerable different material data. This makes it difficult to choose the material properties for engineering design. These facts impose a significant obstacle for MEMS development. Although establishing standard test procedures would be the best approach to solve the problem, it is difficult to achieve due to lack of similarity in scale, geometry, and loading among most MEMS devices. It takes time to achieve agreements and to finalize the optimal testing procedures. However, at this moment, it is possible to develop a method to correlate strength data from different testing procedures and apply the experimental data to MEMS structural design. The focus of this article is to address the importance and to develop such a conversion methodology. Since most MEMS materials are brittle; their strength is controlled by the local behavior of dominated flaws, which depend on structural size, fabrication processes, and loading condition. Strength of these MEMS material is, therefore, a stochastic variable. The reported material strength is only meaningful for MEMS structures with same processing routes, same loading condition, and similar geometry and size with the testing coupon. Strictly speaking, the traditional deterministic safety factor approach should not be used for brittle MEMS structural design. In order to have a better representation of material behavior, instead of material strength, failure probability has been proposed for dealing with MEMS material strength and as the design criterion against structural failure. An "equal failure probability" method is proposed to correlate different material strength test results and to apply these data to MEMS structural design. The basic idea is to analyze the failure probability for different testing specimens and procedures by analytical and finite element models. A dimensionless scaling parameter, called stress ratio, is introduced to modify the test results from different testing procedures. With the assistance of this parameter, it is possible to interrelate testing results between different testing procedures by shaping their tested strength to reach an "equal failure probability" state. In addition, by doing this way, it is also possible to modify these coupon testing data to fit the requirement for MEMS structural design. As a result, a material database can be gradually established. This will make a significant contribution for the future development of MEMS industry.

B5.9
MATERIALS ISSUES OF CERAMICS MICROPARTS. Alfredo M. Morales, Linda A. Domeier, Shawn Allan, Carlos Chang, Sandia National Laboratories, Livermore, CA; Terry Garino, Kevin Ewsuk, Thomas Buchheit, Sandia National Laboratories, Albuquerque, NM.

Modern macroscopic devices achieve novel functionalities by combining components made from a variety of materials. In contrast, currently available fabrication processes limit material choices in microelectromechanical systems (MEMS) devices. For example, leading MEMS technologies such as surface micromachining and LIGA rely primarily on polysilicon or on a few metals which can be readily electroplated as the structural materials. This paper discusses a new a low cost technique for the fabrication of ceramic microparts with typical features ranging between 10 to 1000 mm. Parts are fabricated by sintering powder preforms using ceramic nanoparticles shaped with sacrificial plastic molds generated using LIGA or high aspect ratio UV lithography. For sintered ceramic microparts to be used in MEMS applications, processing issues such as dimensional control, shrinkage, warpage, and material density/porosity must be critically controlled. Properties related to material microstructure and surface roughness must also be thoroughly investigated. This paper will present our baseline preform microfabrication technique, methods for evaluating the properties of ceramic microparts and will discuss the effects of nanoparticle size, fabrication parameters, and sintering schedule on properties of ceramic micropart materials.

B5.10
PIEZOELECTRIC CERAMIC THICK FILMS DEPOSITED ON Si SUBSTRATES BY SCREEN-PRINTING. Kui Yao, Xujiang He, Yuan Xu, Xuanxiong Zhang, and Francis Eng Hock Tay, Institute of Materials Research and Engineering, SINGAPORE.

The attractive piezoelectric properties of lead zirconate titanate (PZT) ceramics are leading to the increasing interests to use them in diverse micro electromechanical systems. In comparison with thin film deposition and ceramic-bonding methods to realise piezoelectric structures, the screen-printing process offers the advantages in producing directly patterned and integrated piezoelectric elements, and filling an important technological gap between thin-film and bulk ceramics. However, several existing problems associated with printed piezoelectric films, such as the low density and the required high sintering temperature, are limiting their performance and applications. In this presentation, a new chemical method was developed to prepare PZT based ceramic pastes. Piezoelectric thick films were deposited with the pastes on silicon substrates by the screen-printing process. Microscopic observations showed that the thick films could be sintered at a greatly lowered temperature, and the structures at different firing temperatures were examined with XRD. Electrical measurements were carried out to investigate the dielectric properties. To evaluate the piezoelectric performance of the films, we fabricated piezoelectric membranes of various sizes with the PZT thick films through micromachining. Mechanical vibrations of the membranes under electrical driving were characterised with a laser scanning vibrometer. The results indicated that the screen-printed piezoelectric thick films would be very promising for many applications in micro electromechanical systems.

B5.11
CHARGE TRANSPORT IN LOW STRESS SILICON-RICH SILICON NITRIDE THIN FILMS. S. Habermehl, C. Carmignani, Sandia National Laboratories, Microelectronics Development Lab, Albuquerque, NM.

Low stress silicon nitride thin films exhibit a useful combination of chemical, elastic and dielectric properties. As a result they are frequently used in the production of MEMS components such as actuators and sensors. However, under an applied electric field thin films of Si-rich silicon nitride can exhibit relatively high leakage currents that may be detrimental in certain applications. In order to develop an understanding of this phenomena field dependent charge transport is studied in correlation to the local atomic Si-N bond strain. Across a range of film compositions varying from fully stoichiometric Si_3N_4 to Si-rich $SiN_{0.54}$, Poole-Frenkel emission is determined to be the dominant charge transport mechanism with the Poole-Frenkel barrier height found to decrease concomitantly from 1.10 to 0.52 eV. Across the same composition range the local residual Si-N bond strain, as measured by FTIR spectroscopy, is observed to vary from 0.006 to -0.0026. Comparison of the barrier height to the residual strain reveals a direct correlation between the two quantities. It is concluded that reductions in the Poole-Frenkel barrier height are a manifestation of compositionally induced strain relief at the molecular level. Reductions in the barrier height result in increased Poole-Frenkel emission detrapping rates and consequently higher leakage currents in Si-rich films.

B5.12
UNIDIRECTIONAL SOLIDIFICATION OF TERBIUM - IRON

ALLOY USING MAGNETIC FIELD IN MICROGRAVITY. Hideki Minagawa, Hideaki Nagai, Yoshinori Nakata, Masataka Sasamori, and Takeshi Okutani, AIST, Microgravity Materials Lab., Sapporo, JAPAN; Keiji Kamada, JSUP, JAPAN.

$TbFe_2$ and $Tb_{0.3}Dy_{0.7}Fe_2$ have drawn much attention due to its excellent magnetostrictive properties at room temperature. These materials have found many applications such as linear actuators, and magnetic sensor system. To obtain excellent magnetostrictive property, the $TbFe_2$ and $Tb_{0.3}Dy_{0.7}Fe_2$ having $< 111 >$ oriented and large crystal are needed. However, oriented and large crystals of $TbFe_2$ or $Tb_{0.3}Dy_{0.7}Fe_2$ are very difficult to synthesize in normal gravity environment because of heterogeneous nucleation and subsequent randomly oriented polycrystalline structure derived from thermal convection in the melt. In microgravity environment, thermal convection, mass transfer, and heat transfer are suppressed. In our previous study of InSb, unidirectional solidification by rapid cooling in microgravity was recently evidenced to produce single crystal. Therefore, we applied magnetic field during solidification in microgravity to produce the oriented structure and large crystal. Usually, magnetic field with several T applied to a melt is known to suppress the convection during the solidification. But, in this study we applied magnetic field less than 0.1T to arrange the structure. In case of $TbFe_2$, when the magnetic field strength was increased from 0 T to 0.045 T during unidirectional solidification in microgravity, a [111] crystallographic alignment dominated, oriented column-shaped crystal became larger and the maximum magnetostriction constant increased from 1000 ppm to 4000 ppm. For unidirectional solidification in normal gravity, the maximum magnetostriction constant remained at 2000 ppm with increasing of magnetic field. These results show that unidirectional solidification using a static magnetic field in micro-gravity has advantage in synthesizing magnetostrictive materials.

B5.13
FABRICATION OF 3D FEED HORN SHAPE MEMS ANTENNA ARRAY USING MRPBI (MIRROR REFLECTED PARALLEL BEAM ILLUMINATOR) WITH AN ULTRA-SLOW-ROTATED AND INCLINED X-Y-Z STAGE. Jong-yeon Park, Kuntae Kim, Sung Moon, Korea Institute of Science and Technology, Microsystem Research Center, Seoul, KOREA; James Jungho Pak, Korea Univ, Dept of Electrical Engineering, Seoul, KOREA.

A 3-Dimensional Feed Horn shape MEMS antenna has some attractive features for array application, which can be used to improve microbolometer performance. Since MEMS technology have been faced many difficulties to fabrication of 3-Dimensional Feed Horn shape MEMS antenna array itself. The purpose of this paper is to propose a new fabrication method to realize a 3D Feed Horn shape MEMS antenna array using a MRPBI (Mirror Reflected Parallel Beam Illuminator) with an ultra-slow-rotated and inclined x-y-z stage. A High-aspect-ratio 300 micro meter sidewalls had been fabricated using SU-8 negative photo resist and 100 micro meter sidewalls had been fabricated using PMER negative photo resist. It can be demonstrate to feasibility of realize 3-Dimensional Feed Horn shape MEMS antenna array fabrication. In order to study the effect of this new technique, the 3-Dimensional Feed Horn shape MEMS antenna array had been simulated with HFSS (High Frequency Structure Simulator) tools and then compared with traditional 3-Dimensional theoretical antenna models. As a result, it seems possible to use a 3-Dimensional Feed Horn shape MEMS antenna at the Tera hertz band to improve microbolometer performance and optical MEMS device fabrication

B5.14
VICKERS INDENTATION FRACTURE TESTS ON ZnO THIN FILMS DEPOSITED ON SILICON WAFERS. Bin Huang, Ming-Hao Zhao, and Tong-Yi Zhang, Hong Kong University of Science and Technology, Department of Mechanical Engineering, Clear Water Bay, Kowloon, HONG KONG.

In the present work, the RF sputtering was adopted to deposit ZnO thin films with various thicknesses on (100) Si wafers at room temperature. Then, we used the Vickers indentation fracture test to assess the residual stresses of the ZnO films and the interface toughness between the ZnO thin film and the Si wafer. The experimental results indicate that there exists a critical value of about $0.7\mu m$ in the film thickness. When the film was thinner than the critical thickness, the damage mode under the indentation test was the radical cracking. In this damage mode, we estimate the residual stress of the films by measuring the crack lengths under various loads. The residual stress in the ZnO thin film is about 350 MPa in compression. When the film thickness was around the critical value, another damage mode of a circular interface cracking occurred together with the radical cracking. If the film thickness was larger than the critical value, the radial cracking was depressed and only the circular interface cracking occurred. From the radius of the interface crack, we estimated the interface toughness of the film/substrate to be about 0.55 J/m^2.

B5.15
MICROSTRUCTURE FABRICATION BY ANODIZATION OF P^+-TYPE Si SUBSTRATE. Shingo Uehara, Junpei Sugimoto, Yoichi Eto and Tadashi Matsubara, Seikei Univ, Dept of Electrical Engineering and Electronics, Tokyo, JAPAN.

Microstructures were fabricated by selective anodization of Si substrate. n-type layers were used as anodization resistive regions which were used either as the microstructure components or as the current restricting areas to form specific etched structure. The idea of this kind of process was first reported by Higa et al. [1]. They applied porous mode anodization on p-type substrate and used the resulted porous layer as a sacrificial layer. In this work, we applied polishing mode anodization on p^+-type substrates. The polishing mode anodization made the fabrication process simpler and the p^+-type substrates allowed us to form microstructures which were not obtained by using p-type substrates. Boron doped (100) oriented Si wafers with a resistivity of 0.01-0.02 $\Omega\cdot$cm were used and n-type layers were formed by phosphorous diffusion. Anodization was made under the current density of $200mA/cm^2$ using 1:1 HF-ethanol electrolyte. After a proper time of anodization, the part of the substrate beneath the narrow n-type region is etched off leaving an air suspended n-type Si stripe which is used as a cantilever arm. The cantilever is actuated by applying reverse bias voltage on the pn-junction formed between the wider n-type region at the end of the arm and the substrate. The restricted current flow under the n-type region leaves specific silicon structures on Si substrate reflecting the flow pattern. By using circular or square shaped n-type regions, and by stopping anodization just at the time when etch fronts meet each other at the center of the bottom of the n-type region we can form micro pins with submicron radius tips extruded out from the substrate. 10x10μm spaced pin array was fabricated by the process. [1] K. Hiaga and T. Asano, Jpn. J. Appl. Phys. Vol. 35 (1996) pp. 6648-6651

B5.16
FABRICATION OF PIEZOELECTRIC DIAPHRAGM USING LEAD ZIRCONATE TITANATE (PZT) FILMS. Eunki Hong[a], S.V. Krishnaswamy[a,b], C.B. Freidhoff[b] and S. Trolier-Mckinstry[a]; [a]Materials Research Institute, The Pennsylvania State University, University Park PA; [b]Northrop Grumman Electronics, Sensors and System Sector, Baltimore, MD.

Piezoelectric films can provide both sensing and actuation mechanisms in many MEMS structures. For this reason, these films have been studied for many micro-sensor and actuator applications. Here, the fabrication of piezoelectric diaphragm using lead zirconate titanate is presented. Two kinds of diaphragms were fabricated: d_{31} and d_{33} mode. For d_{31} mode devices, $Pt/Ti/SiO_2/Si$ substrates were used, on which $\sim 1\mu m$ sol-gel PZT layer was deposited. Evaporated Cr/Au formed the top electrode. Each layer was patterned using photolithographic technique. To release diaphragms from the substrate, deep reactive ion etching was used. A description of fabrication process flow will be presented. Over 4$''$ wafer, the thickness variations of the PZT and Cr/Au were below 5%. The dielectric constant and loss of the piezoelectric layer at 1 kHz were >800 and <2%, respectively. The remanent polarization was $\sim 20\mu C/cm^2$ and the coercive field was $\sim$61kV/cm. For d_{33} mode devices, ZrO_2 was used as a barrier layer between the PZT and the substrate. The top electrode layer was patterned into interdigital transducers (IDT). Ferroelectric measurements showed well-developed hysteresis loops for the d_{33} PZT films. The sizes of d_{31} and d_{33} mode diaphragms were from $100\mu m$ to $1000\mu m$. To estimate their performance, charge output from diaphragms was collected with various pressure oscillations. $1000\mu m$ d_{31} diaphragms generated $\sim$25 pC with 0.2 kPa pressure oscillation. Resonance characteristics of these diaphragms were evaluated using an impedance/phase analyzer (HP4194A). The deflection of the diaphragms was measured using an interferometeric technique. Details of the resonance and deflection measurements along with ANSYS finite element analysis modeling will be presented.

B5.17
NEUTRON IRRADIATION-INDUCED DIMENSIONAL CHANGES IN MEMS GLASS SUBSTRATES. Clark L. Allred[a,b,c], Jeffrey T. Borenstein[a], Linn W. Hobbs[b], [a]The Charles Stark Draper Laboratory, Inc., Cambridge, MA; [b]Massachusetts Institute of Technology, Cambridge, MA; [c]Air Force Institute of Technology, Wright-Patterson AFB, OH.

A study is made of radiation-induced expansion/compaction in Pyrex and Hoya SD-2 glasses, which are used as substrates for MEMS devices. Glass samples were irradiated with a neutron fluence composed primarily of thermal neutrons, and a floatation technique was employed to measure the resulting density changes in the glass. Transport of Ions in Matter (TRIM) calculations were performed to relate fast ($\sim$1 MeV) neutron atomic displacement damage to that of boron thermal neutron capture events, and measured density changes in the glass samples were thus proportionally attributed to thermal and fast neutron fluences. The trend for strain with thermal neutron fluence (n/cm^2) was found to be a linear compaction of -2.8 × 10^{-20} for Pyrex and -1.0 × 10^{-21} for Hoya SD-2. For fast neutron fluence, the trend for strain was also linear: -6.1 × 10^{-21} for Pyrex and -7.9 × 10^{-22} for Hoya SD-2. The measured radiation-induced compaction of Pyrex is found to agree with that of previous studies. To our knowledge, this work represents the first study of compaction in Hoya SD-2 with neutron fluence. Hoya SD-2 is of considerable importance to MEMS, owing to its close thermal expansivity match to silicon from 25-500°C.

B5.18
ISSUES WITH GOLD ELECTROPLATING FOR MICRO-ELECTROMECHANICAL SYSTEMS (MEMS) APPLICATIONS.
Caroline A. Kondoleon, Thomas F. Marinis, and Todd A. Hamilton, The Charles Stark Draper Laboratory, Electronics Packaging and Prototyping Division, Cambridge, MA.

Electro-plated gold films are used extensively in packaging of Microelectromechanical (MEM) sensors to make connections to signal conditioning electronics. Over the past two years, production lots of gold plated substrates, procured from various vendors, failed an accelerated aging qualification test. In this test, aluminum wires (diameter = 0.0025 mm) were ultrasonically welded to the film, followed by an aging process at 120°C for 48 hours, and then pulled to destruction. The criterion for passing this test was that the wires should break both before and after aging. In the defective lots, the wires lifted off of the gold film after aging. Analysis of these defective films by Scanning Electron Microscopy (SEM), Auger Electron Spectroscopy (AES), and Time-of-flight Secondary Ion Mass Spectroscopy (TOF-SIMS) suggested that cyanide residues, deposited from the plating bath, concentrated beneath the bond as the gold and aluminum reacted to form an inter-metallic compound during aging. An etch and cleaning treatment combination was developed for the defective substrates, which removed an amount of cyanide residues from the gold sufficient enough to pass the qualification test.

B5.19
THE FABRICATION OF STAINLESS STEEL PARTS FOR MEMS.
Terry Garino, Alfredo Morales, Kevin Ewsuk, Thomas Buchheit, Sandia National Laboratories, Albuquerque, NM.

Stainless steel is one of the most widely used structural materials due to it's unique mechanical and chemical properties. Applications exist in MEMS where stainless steel is ideally suited but it is not possible to use the standard MEMS processes to fabricate stainless parts. We have developed an economical, powder metallurgy-based process for fabricating parts in the 0.1 to 1 mm size range with features less than 10 μm using a stainless steel nanopowder and sacrificial polymeric micromolds. Several techniques have been developed for filling the mold cavities with the powder and then removing the formed parts from the molds. The sintering behavior of stainless steel parts was studied in situ using video observation up to 1350°C. The microstructure of sintered parts was characterized using SEM. Surface roughness, dimensional tolerance and part camber were also investigated. The mechanical properties including modulus and yield stress were measured using millimeter-size parts made by this process. 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.

B5.20
THERMAL LOSSES AND TEMPERATURE MEASUREMENT IN SOI MEMS HEATER.
Mark McNeal, James T. Daly, Anton C. Greenwald, Edward A. Johnson, Nicholas Moelders, Martin Pralle, Ion Optics, Inc. Waltham, MA; Thomas George and Daniel S. Choi, Jet Propulsion Laboratories, Pasadena, CA.

A sensor chip has been designed and tested that uses a MEMS strip heater as both a source and detector of infrared radiation. A power supply stable to one pert per million was developed so that, in the absence of signal, the temperature of the heater is reproducible from pulse to pulse to better than 0.01%. An optical cavity reflects infrared radiation back onto the source filament. Changes in reflected light intensity modify heater temperature, and the measured signal is a change in resistance. To maximize this signal, we used single crystal silicon for the heater filament because it has a very high temperature coefficient for a change in resistance (TCR). Starting material for MEMS fabrication was SOI fabricated by wafer bonding and etch back. The single crystal silicon heater was about 10 microns thick, 0.3 mm wide and 3mm long. To minimize thermal losses by conduction, a series of 6 filaments connected by a bus bar is supported only by very thin electrical connections to the substrate. Substrate material underneath the heaters was completely etched away. Thermal losses due to convection were determined as a function of pressure. Vacuum packaging is required. Measured (delta)R/(RT) was about 0.01, or a one percent change in resistance per degree centigrade change in temperature. The use of SOI is more expensive than polycrystalline silicon deposited on top of silicon oxide, but increased sensitivity by about a factor of ten.

B5.21
MODELING OF MICROCANTILEVER BEAMS SUBJECT TO COMBINED ELECTROSTATIC AND ADHESIVE FORCES.
J.A. Knapp and M.P. de Boer, Sandia National Laboratories, Albuquerque, NM.

One of the most important issues facing the continued development and application of microelectromechanical systems (MEMS) is that of adhesion and friction between microstructures intended to transfer force. In this talk, we develop modeling approaches for studying adhesion (i.e., stiction) using the observed shape of microcantilever beams under electrostatic loading. Exact analytical models for an idealized configuration are first presented. The exact solutions show the regimes over which the beam deflections are sensitive to adhesion versus applied loading, as well as revealing the existence of an applied pressure where the beam contacts the underlying surface. Further, the beam adhesion value is shown to be independent of the elastic energy that is expended to bend the beam into its adhered shape. Secondly, we present the finite-element modeling approach that is required to model the non-uniform electrostatic loading force used in our experiments, as well as other non-idealities such as support post compliance. We use the finite-element modeling to address the question of sensitivity to the surface force law. An important result is that the beam deflections are insensitive to the details of the surface force law, so adhesion is well quantified in our experiments. We conclude by showing how interferometric data from the beam deflections is used with the finite element calculations of deflections to obtain adhesion values continuously along the length of a cantilever beam. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, supported by the US Department of Energy under contract DE-AC04-94AL85000.

B5.22
MEMS TONPILZ TRANSDUCERS.
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Tonpilz transducers consist of a heavy tail mass, a piezoelectric spring, and a light head mass. Miniaturized tonpilz transducers are potentially interesting for the 10-100 MHz frequency range in imaging transducers, since the mass loading enables the piezoelectric film to be comparatively thin (4-10 μm). In this work, fabrication and characterization of lead zirconate titanate (PZT) thick films on conductive oxide $LaNiO_3$ (LNO) coated silicon on insulator (SOI) substrates will be reported for this application. First, conductive LNO thin films approximately 300 nm in thickness were grown on SOI substrates by a metal-organic decomposition (MOD) method. The room temperature resistivity of the LNO was 6.5×10^{-4} Ω·cm. Randomly oriented PZT (52/48) films up to 7 μm thick were then deposited using a sol-gel process on the LNO coated SOI substrates. 20 mol.% excess lead was added to the solutions to compensate for lead volatilization during film heat treatments. PZT films with LNO as the bottom electrode showed good dielectric and ferroelectric properties. The dielectric permittivity (at 1 kHz) was over 1000, and the dielectric loss was less than 0.03. The remanent polarization of PZT films was larger than 25 $\mu C/cm^2$. The $e_{31(eff)}$ coefficient of PZT thick films was larger than -4.5 C/m^2 when poled at 70 kV/cm for 15 minutes. Silver layers approximately 40 μm thick were screen-printed onto the PZT films. Elements were diced and the bulk silicon was removed by a wet-etching method. Fabrication of MEMS tonpilz microstructures will also be presented.

B5.23
INFLUENCE OF FABRICATION PROCESS ON THE PROPERTIES d33 MODE OF PZT CANTILEVERS.
Q. Zhang[a,b], S.J. Gross[a], F.T. Djuth[b], S. Tadigadapa[a], T. Jackson[a], and S. Trolier-McKinstry[a]. [a]The Pennsylvania State University, University Park, PA; [b]Geopace Research, Inc. El Segundo, CA.

$Pb(Zr_{0.52}Ti_{0.48})O_3$ (PZT 52/48) cantilevers with surface interdigitated (IDT) electrodes which can be actuated using a d_{33} mode were fabricated using surface micromahing techniques. ZrO_2 thin film, prepared using a sol-gel technique, was used as the buffer layer between the Si_3 N_4 support and the PZT. PZT films exhibited a random perovskite structure. The influences of the fabrication processes on the properties of PZT were investigated. Domain pinning was observed after Au/Cr metals were evaporated on the PZT surface followed by lift off to form IDT electrodes. The pinning can be removed by annealing the sample at 450°C on a hot plate. The

cantilever was patterned by ion milling PZT and ZrO_2. Voltage shifts and remanent polarization reduction in the transverse polarization-voltage loop of the patterned PZT were observed. To investigate the influence of ion milling on the piezoelectric and dielectric properties PZT samples with Pt bottom and top electrodes were used. It was found that the piezoelectric coefficient d_{31}, and the dielectric constant decreased after ion milling while the dielectric loss increased. Ion milling also induced an internal electric field directed toward to bottom electrode which was manifested in asymmetry in the piezoelectric coefficient d_{31} as a function of poling direction. All of the damage induced by ion milling could be removed annealing the film at 450°C for 15 min.

B5.24
NEW MEMS TECHNOLOGY USING MULTI-LAYER NILC POLY-Si AND NiSi FILMS.
N. Zhan, C.F. Cheng, M.C. Poon, M. Qin, C.Y. Yuen, M. Chan, Dept. of Electrical & Electronic Engineering, Hong Kong University of Science & Technology, Sai Kung, HONG KONG.

A novel MEMS technology using multi-layer poly-silicon (poly-Si) and nickel silicide (NiSi) films is proposed. The poly-Si film is formed from the new Nickel-Induced-Lateral Crystallization (NILC) method and has very large grain (>10um) and near crystal quality. 700nm thermal oxide was grown on Si wafer. 100nm LPCVD amorphous Si (a-Si) was deposited and followed by 5nm Ni. The a-Si was crystallized at 550°C/65hr and subsequent 800°C/1hr to form the first (lower) NILC poly-Si. The process was repeated and a second (upper) poly-Si was formed on top of the first poly-Si. The lower poly-Si has slightly larger grains and better material quality. Thin-film-transistors (TFT) fabricated on the 3-dimensional (3-D) poly-Si layers have I-V characteristics similar to (>40%) silicon-on-insulator TFTs. While TFTs on lower layer have better mobility and device properties, TFTs on upper layer have better uniformity. The accumulated heating and other effects have also been studied. Besides aluminum, NiSi film formed by rapid-thermal-annealing (RTA) has also been attempted as common contact and electrode layer for the 3-D MEMS structures. The silicide consumes less Si, has simple formation process, low resistivity (~15 uohm-cm) and is stable beyond 800°C. The NiSi also has good elasticity (Young's modulus ~132 GPa) and has been used to form low stress micron-size micro-bridges, cantilevers and other MEMS structures. The new NILC poly-Si and NiSi MEMS technology can have promising applications in novel materials, structures, devices and circuits.

B5.25
MICROSTRUCTURING OF SILICA AND POLYMETHYL-METHACRYLATE GLASSES BY FEMTOSECOND IRRADIATION FOR MEMS APPLICATIONS.
Saulius Juodkazis, Kazuhiko Yamasaki, Andrius Marcinkevičius, Vygantas Mizeikis, Shigeki Matsuo, Hiroaki Misawa, Tokushima Univ., JAPAN; Thomas K. Lippert, Paul Scherrer Institut, SWITZERLAND.

We report on the microstructuring of silica and polymethyl-methacrylate (PMMA) glass by tightly focused femtosecond (fs) laser pulses (the numerical aperture of the objective lens we used was NA = 0.8-1.35, pulse duration was 140 fs, and the wavelength 795 nm). The typical pulse duration at the irradiation point was less than 400 fs at the pulse energy of 2-20 nJ for the different materials and the depth of focusing. Chemical wet etching of irradiated regions allows the fabrication of complex three-dimensional (3D) structures inside the bulk of those materials. The interconnected channels were etched out in silica and crystalline sapphire. The achievable smallest diameter was about 10 μm for a 200 μm long channels. Selectivity of etching (the etching rate of irradiated regions vs. that of unexposed material) depends strongly on the etchant and was typically between 20-60 for the HF-based etchants of silica. Pentazadiene doping of PMMA makes possible to control the light-induced damage threshold (LIDT) of material. 8 wt% doping of PMMA lowered the LIDT to 35% of its value in the undoped PMMA. The potential of the 3D fs-microstructuring of transparent materials for the fields of micro-fluidics and photonic crystals is discussed. For comparison, the preliminary results on the LIDT and microstructuring of rubber materials by fs pulses will be demonstrated.

B5.26
ELECTROMECHANICAL PROPERTIES OF PZT SOL-GEL THIN FILMS BY NANOINDENTATION.
P. Hvizdos[a], M. Alguero[b], A.J. Bushby[a], M.J. Reece[a], R.W. Whatmore[c] and Q. Zhang[c]. [a]Department of Materials, Queen Mary, University of London, London, UNITED KINGDOM; [b]ICMM, CSIC, Madrid, SPAIN; [c]School of Industrial and Manufacturing Science, Cranfield University, UNITED KINGDOM.

The potential commercialisation of MEMS rests on addressing design and reliability issues. Their design requires knowledge of the elastic coefficients of the ferroelectric film, as well as the other materials in the system. Understanding their reliability requires the determination of stress thresholds for depolarisation. Lead zirconate titanate (PZT) compositions are preferred for actuation applications. We present here the mechanical properties of PZT films with different Zr/Ti ratios, prepared by sol-gel processing. Nanoindentation with spherical tipped indenters was used to obtain the elastic stiffness coefficient of the films. Electromechanical measurements were also performed with the nanoindentation system. The spontaneous polarisation and stress induced depolarisation of films was investgated by measuring the current intensity transients during indentation.

B5.27
INDUCTIVE COUPLED PLASMA ETCHING OF HYDRO-GENATED AMORPHOUS SILICON CARBIDE FILMS.
J. Shi, W.K. Choi, E.F. Chor, National University of Singapore, Department of Electrical and Computer Engineering, SINGAPORE.

Amorphous silicon carbide (a-SiC) films have been suggested as passivation material for electrical devices, or as masking material for Microelectromechanical systems. In these applications, etching of a-SiC is an essential step. There are only a few reports on the wet etching of a-SiC. In this paper, we report the inductive coupled plasma (ICP) etching of plasma enhanced chemical vapour deposited (PECVD) hydrogenated amorphous silicon carbide (a-SiC:H) films using the CF_4/O_2 chemistry. The carbon content in the films, determined by the flow rates of silane and acetylene, was varied from 20 to 80%. The etching process was conducted by varying the RIE power from 50W to 500W and the ICP power from 50 W to 750W. The etch rate of the as-prepared a-SiC:H films increased only slightly with an increase in the carbon concentration in the films under our present etching conditions. This suggests that the etch rate of our films was rather insensitive to the carbon concentration in the films. However, the furnace annealed a-SiC:H films showed a large reduction in the etch rate under the same etching condition. The IR spectra of the annealed films showed a significant reduction in the Si-H and C-H intensity, i.e. hydrogen effusion from the films. Note that we have also shown that annealing caused a great reduction in Si- and C- dangling bonds. Therefore, we suggest that hydrogenation and the elimination of dangling bonds could be responsible for the decrease in the etch rate of our samples. We are currently carrying out the XPS and ESR measurements on our samples to determine the density of the Si-Si, C-C, Si- and C- dangling bonds. The IR, XPS and ESR results will provide us information on the distribution of the various bonds in our films. This will enable us to examine the etching behaviour and mechanism of a-SiC:H films with different carbon or hydrogen contents.

B5.28
Transferred to B9.5

B5.29
HIGH TEMPERATURE RESISITOR MATERIAL FOR MICRO HOT PLATES.
Jacek Baborowski, Nicolas Ledermann, Arnaud Pollien, Paul Muralt, Ceramics Laboratory, Swiss Federal Institute of Technology EPFL, Lausanne, SWITZERLAND.

Ta_5Si_3, a new material for the resistive thin film of micro hot plates was investigated and tested. The material was obtained by dynamic co-sputtering of Ta and Si, and crystallized in the finished device by resistive heating to 900°C. The silicidation temperature is 650°C, the melting temperature 2400°C. Resting on a Si_3N_4/SiO_2 membrane, and topped with a SiO_2 protection film, this material allows for operation temperatures of up to 1000°C. The resistance increases linearly with temperature. The TCR amounts to (5.5 ± 0.6)*10-4 °K-1, the specific resistivity to 90 μ*cm at 20°C. The material was investigated by means of Transmission Electron Microscopy and X-ray diffraction as a function of temperature. After deposition at 500°C, the material is mostly amorphous with grains nucleated on the top suface. These grow through the film during silicidation. The resistivity drops and the mechanical stress decreases to zero. The use as infrared source is one of the promising applications of the micro hot plate. A large amount of heat is radiated as infrared light (around 10% into 7 to 14 μm).

B5.30
PIEZORESISTIVE CHARACTERISATION OF 4H-SiC ON INSULATOR (SICOI) USED AS A HIGH TEMPERATURE PRESSURE SENSOR.
Emmanuel Defay, Vincent Mandrillon, Patrice Rey, Fabrice Letertre, Bernard Diem, CEA-LETI, Silicon Technologies Department, Microsystem Technologies Division, Grenoble, FRANCE.

SiC is a very promising material for high temperature applications in the micro-sensors field. In this paper, the piezoresistive properties of 4H-SiC are demonstrated for the first time. A 4H-SiC thin film (1 μm) reported onto an oxidized Si wafer using the Smart Cut method is used as the sensing element of a piezoresistive pressure sensor. As the substrate is silicon, the classical microelectronic tools are used in order to process the micro-sensor. The isolation of the SiC sensing element from the Si-substrate avoids leakage current, especially at high temperature. The sensor was characterized between room temperature and 380°C. The sensitivity of the device goes from 2.4 mV/V bar at room temperature to 2.0 mV/V bar at 380°C. The average temperature coefficient of sensitivity (TCS) is -0.05%/°K.

B5.31
HIGH ASPECT RATIO MICROSTRUCTURES BY NEGATIVE PHOTOSENSITIVE GLASS PROCESS. Sooje Cho, Seongil Chang, Sanghoon Kim, MEMSware, Seoul, KOREA (SOUTH); Yangdo Kim, Jaewoong Choi, Sunggoon Kang, Hanyang Univ, Seoul, KOREA (SOUTH).

High aspect ratio microstructures (HARMSTs) are being used in many fields such as mechanical parts, bio devices, sensors, actuators and so on. A number of technologies have been developed to fabricate HARMSTs. Even conventional mechanical machinings as well as LIGA and silicon anisotropic etching have advantages for the fabrication of microstructures. One of the traditional method of HARMSTs is photosensitive glass process which was discovered by Corning's Stookey about fifty years ago. The microstructures formed by this method have been already used for many devices like vacuum spacer, micro mixer, lab on a chip, ink jet head and so on. But because some microsystems need much finer microstructuring, it can not meet this need due to its low etching selectivity. In 1998, the first author also discovered a photosensitive glass process which is opposite phenomenon of the conventional photosensitive glass process. In the case of conventional photosensitive glass process, the exposed area is crystallized by heat treatment, and the crystallized area has high etch rate in hydrofluoric acid solutions. But in negative photosensitive glass process, the unexposed area is crystallized and etched in acid solution. It has etching selectivity of about 70 in static hydrofluoric acid solution. This is about 3 times higher than that of conventional process. The glass exposed through photomask and crystallized was dipped into the etching solution and the holes with the diameter of 70 micrometer were formed at 1mm thick plate. At the same etching condition, microgears fabricated by this method showed much finer structures than that of conventional photosensitive glass process. So much more microsystems can be realized by negative photosensitive glass process.

B5.32
TRANSFER OF HIGH-TEMPERATURE-ANNEALED CERAMIC MICROSTRUCTURES ONTO HEAT-SENSITIVE SUBSTRATES. M. Heule, L.J. Gauckler, ETH Zurich, Dept of Materials, Zurich, SWITZERLAND.

Novel microfabrication techniques based on Soft Lithography allow to produce powder-based ceramic microstructures with feature sizes in the order of 10 μm. Since there are abundant ways to synthesize powders with well defined chemical composition, phase and size distribution, integrating ceramic powders into microfabrication processes may offer additional functionality in MEMS devices, e.g. micromachined gas sensors. In contrast to thin-film preparation methods predominantly used for MEMS fabrication, the processes employed here resemble the classic route to produce structural ceramic parts. This includes the preparation of a suspension, forming, drying and sintering. However, heating to high temperatures is required in the sintering step (>1073 K) which almost all standard MEMS devices would not stand. In order to avoid this incompatibility, an approach to transfer ceramic microstructures after the sintering on a heat resistant substrate to another heat sensitive substrate will be demonstrated. After embedding the microstructures into a polymer, they are released from the original substrate and placed on a new one. The process will be evaluated in terms of the abilities to handle the fragile micro-ceramics without damage and to preserve the relative positions initially defined by the microfabrication process.

B5.33
THE EFFECTS OF TENSION AND COMPRESSION ON FATIGUE OF POLYSILICON MEMS DEVICES. H. Kahn, R. Ballarini, A.H. Heuer, Case Western Reserve University, Dept of Materials Science and Engineering, Dept of Civil Engineering, Cleveland, OH.

Polysilicon films deposited via LPCVD are the current mainstay structural material for MEMS. As a result, it is important to understand and quantify the processes leading to fracture and fatigue of polysilicon MEMS, in order to design future devices and predict their reliabilities. It has long been assumed that brittle materials such as polysilicon are immune to fatigue effects. However, it has been reported (by Brown, et al. in 1997 and 2000, and by us in 1999) that polysilicon devices with stress concentrations (notches) exhibit delayed cyclic fatigue fracture under resonance loading. In all of these tests, the site of stress concentration, where the fatigue fracture occurred, was subjected to cyclic tension-compression loading, with the magnitude of the maximum tensile stress equal to that of the maximum compressive stress in the cycle. We have previously developed an on-chip electrostatic actuator which can generate sufficient forces (up to -0.8 mN) to bias the MEMS fracture mechanics specimen with either a tensile or compressive stress, before resonance is initiated. We can therefore determine the effects of the compressive and tensile portions of the loading cycle on the fatigue behavior. It has also been reported (by Connally and Brown in 1992 and Van Arsdell and Brown in 1999) that relative humidity has a strong influence on crack growth in resonating silicon devices. Both of these effects in notched specimens are being studied and will be reported on.

B5.34
DEVELOPMENT OF ULTRA-THIN RESONATING MICRO CANTILEVERS FOR HEAVY VAPOR DETECTION.
Parshuram Zantye, Rajnish K. Sharma, Ashok Kumar and J. Mark Anthony, Center for Microelectronics Research, University of South Florida, Tampa, FL.

The development of nano-technology and Microelectromechanical Systems MEMS has made it possible to develop very sensitive micro sensors and detectors for multifunctional applications. The increasing use of Silicon On Insulator {SOI} substrates in micro machining has made the fabrication of the ultra thin components of Micro-electromechanical Systems {MEMS} like cantilevers, actuators, micro mirrors very convenient. The ultra thin silicon cantilevers {thickness <0.25 μm} can be put to use for detection of vapors having density more than air {heavy vapors}. In this paper we describe a device made of gold-coated ultra thin silicon resonating cantilever {50 μm x 10 μm x 0.24 μm} fabricated over SOI substrate to detect mercury vapors. The device is based upon the principle of measurement of the change in resonant frequency of the ultra thin resonator due to surface adsorption of mercury molecules on the resonator. The change in resonant frequency $\Delta f = (\{1.875\}^2/4\ \pi\}$ x $\{EI/12\}^{0.5}\}$x $\{\{m\ load\}/\{m\ c\}^{1.5}\}$, where {m load} is the mass of the cantilever after loading due to mercury molecules, {m c} is the initial mass of the cantilever, E is the modulus of elasticity of the cantilever and I is the moment of inertia of the cantilever. The selectivity of the device is achieved by sputter deposition of gold on the surface of the resonating cantilever. These cantilevers are fabricated using an easy two-step fabrication process. They are subsequently released using supercritical drying method. This system has the following distinct advantages: a) ease of fabrication b) high reproducibility c) capability to achieve sub micron dimensions in thickness d) high selectivity due to specific vapor selective thin film e) high sensitivity due to relatively low system size. The technology can be extended for detection of substances such as ethanol, methanol and acetone in gaseous form by choosing suitable thin film coating to achieve the desired selectivity.

B5.35
INTERFEROMETRIC CHARACTERIZATION OF CANTILEVER DYNAMICS FOR MEGAHERTZ FREQUENCY (ULTRASONIC) VIBRATIONS. Bryan D. Huey, Oleg V. Kolosov, Oxford University, Dept. of Materials, Oxford, UNITED KINGDOM; Robert Szoszkiewicz, Andrzej Kulik, EPFL, Deparment of Physics, Lausanne, SWITZERLAND; Andrew Briggs, Oxford University, Dept. of Materials, Oxford, UNITED KINGDOM.

Microfabricated cantilever structures are commercially applied in systems ranging from MEMS devices to atomic force microscope (AFM) sensors. In certain conditions, a lever can be subjected to excitations from kHz up to the MHz frequencies, for example in the novel variation of AFM called Ultrasonic Force Microscopy (UFM). The dynamics of AFM cantilever vibrations are measured directly in this frequency range using a optical heterodyne interferometer. Three distinct oscillations have been investigated. When ultrasonically exciting the base of a lever with an attached piezoelectric element, an analogue to high frequency system vibrations, the device acts as a waveguide and a frequency dependent standing wave is generated in the lever. For a free lever contacting a vibrating surface with repulsive forces less than 50 nN, encountered in UFM, harmonics of the vibration frequency are generated but the amplitude at the driving frequency is negligible. Finally, the response of a grounded, metallized lever to electrostatic excitations with a nearby biased sample allows high frequency electrostatic tip-sample interactions to be characterized. The spatial resolution of the system is on the order of ten microns and the amplitude resolution of the system is better than 10 pm.

B5.36
ELECTROSTATIC CHARGING OF RESONANT PROBES - EXPLOITING A RELIABILITY PROBLEM. Peter Turmezei, Robert Kazinczi, Jeff Mollinger, Andre Bossche, Delft Univ Technology, Dept of Information Technology and Systems, Delft, THE NETHERLANDS.

Recently more and more micromechanical devices implement mechanical resonators thanks to its extremely high sensitivity and convenient signal processing. The characteristics of micromechanical resonators used in vibration sensors, micromechanical A/D converters etc. are comparable to high-end electronics. One of the key issues is the packaging, which is strongly application dependant, determines the reliability of the system and can take up to 80% of the fabrication costs. We looked into the failure mechanisms of micromechanical resonators in order to determine the minimum requirements for the packaging. Crystalline silicon resonator beams and paddle-beams were studied in different environments. These tests are particularly important for improving the performance and the stability of structures operated in direct contact with the surrounding media. Functionalized probes are such resonant structures. The active area of the resonator adsorbs desired components from the environment. An electrostatic charging-induced failure mechanism was found on high-frequency probes. The resonant beams and paddle-beams collect airborne particles from the environment. Including this extra mass, the probe provides false measurement results. Extra care is required to avoid this “flycatcher effect” on the passive regions of resonant probes. The adsorption rate is calculated from the measured resonance frequency shift, and depends on the driving amplitude and on the environment. The adsorption rate in ambient air was 3.6*10E-13 kg/sm2. The phenomenon can be exploited to detect airborne contamination. The system worked with high sensitivity and fast response time during tests in fine smoke. The measured adsorption rates ranged from 3.8*10E-10 to 2.3*10E-9 kg/sm2 depending on particle content and size. Compared to the active functionalized technique, the electrostatic one has the advantage of easy electronic on/off switching and resetting. The sensor can standby inactively in the measurand media, and can be refreshed with only electrical signal.

B5.37

COMPARATIVE STUDY OF FRICTIONAL BEHAVIOR BETWEEN ALKANETHIOLS ON GOLD AND ALKYLSILANES ON SILICON OXIDE. Byeongwon Park, Mike E. Chandross, Mark J. Stevens and Gary S. Grest, Sandia National Laboratory, Albuquerque, NM.

Molecular dynamics computer simulations were carried out to investigate the frictional behavior of self-assembled monolayers (SAMs) of alkanethiols on gold and alkylsilanes on silicon oxide. Understanding of the lubrication mechanism of SAMs is crucial in applications such as microelectromechanical systems (MEMS), magnetic storage, and nonlinear optics. Alkanethiol films on gold have been extensively investigated because they provide a model for the formation of other molecular organic films. In addition, the huge body of experimental data can be used for validation of computer simulations. Polycrystalline silicon, a common material for MEMS applications, tends to oxidize easily and is prone to problematic adhesion. Organic films such as alkylsilanes are used to minimize adhesion and static-charge by forming low energy and hydrophobic surfaces. In both cases, there are many unanswered questions on the molecular scale and computer simulations can be useful to provide an atomistic view. The structure and frictional behaviors of the two systems are investigated as a function of temperature, chain length and end group. An all-atom Class II force field is used. The details of the structure and frictional behavior of alkanethiols on gold will be presented, and compared with alkylsilanes on silica whenever possible.

B5.38

FRICTIONAL PROPERTIES OF SELF-ASSEMBLED ALKYLSILANE CHAINS ON SILICA. M. Chandross, Byeongwon Park, M.J. Stevens, and G.S. Grest, Sandia National Laboratories, Albuquerque, NM.

We present the results of molecular dynamics simulations of pairs of alkylsilane monolayers on silica surfaces under shear. In particular, we investigate the effects of shear velocity on the friction for chains of 6, 8, 12, and 18 carbon atoms covalently bonded to a crystalline surface. Our studies are performed at loads of 200 MPa and 2 GPa for velocities of 0.1, 1.0, and 10.0 m/s. We find that for perfect (defect-free) monolayers, the effects of chain length and velocity are weak, indicating that the experimentally measured dependence of friction on these properties is primarily due to defects in the monolayer. To further investigate this, we have performed shear simulations in which defects have been added to the monolayers, both through the removal of chains, as well as through the introduction of gauche defects in the chain backbone. We have also investigated possible finite size effects by varying our system dimensions from $40\AA X40\AA$ to $200\AA X200\AA$. We find that increasing the surface area by a factor of N reduces the noise in the shear stress by a factor of $\sqrt{N}$, and has a comparable effect to averaging the smaller system data over bins of $\sqrt{N}$ points. This indicates that finite size effects are negligible in our simulations.

B5.39

DESIGN AND MODELING OF A MICRO-FOURIER SPECTROMETER BASED ON SAMPLING A STANDING WAVE. D. Knipp, M. Rosa, Xerox Palo Alto Research Center, Palo Alto, CA; H. Stiebig, Research Center Jülich, Institute of Photovoltaic, Jülich, GERMANY; H.-J. Büchner, Technical University Ilmenau, Institute of Sensor Technology, Ilemau, GERMANY; R.A. Street, Xerox Palo Alto Research Center, Palo Alto, CA.

A novel concept for an Fourier spectrometer based on thin film technology and surface micro machining will be presented. The spectrometer is based on sampling a standing wave. The standing wave is created in front of an electro statically tunable mirror and sampled by a transmissive detector [1]. The working principle behind this micro-spectrometer reduces the number of components to a minimum and facilitates the realization of 1D and 2D micro-spectrometer arrays. The performance of this is mostly determined by the design of the transmissive optical sensor and the associated materials in the fabrication process. Amorphous silicon and other thin film materials are favorable here, because these layers can be realized at low temperatures on transparent substrates. The performance of the spectrometer in this instance is limited on one hand by the optical design of the sensor taking the interaction between the standing wave and the transmissive sensor into account. On the other hand the device performance is limited by the material properties. Due to the strong wavelength dependent absorption of amorphous silicon within the visible range, the free spectral range is then limited to 50-100nm. Since for shorter wavelengths most of the light is absorbed in the sensor whereas for higher wavelengths the absorption coefficient of amorphous silicon is drastically reduced. We will present the concept for a micro Fourier spectrometer based on thin film technology and surface micro machining. The performance of the spectrometer will be described by an optical model, which takes the optical material properties and the device design into account. The influence of the device design on the free spectral range and the FWHM will be modeled and discussed. [1] H.L. Kung, S.R. Bhalotra, J.D. Mansell, and D.A.B. Miller, “Compact transform spectrometer based on sampling a standing wave,” in International Conference on Optical MEMs 2000, Kauai, HI (Aug 21-24, 2000).

B5.40

DEVELOPMENT OF POLYSILICON MATERIALS FOR IGNITERS AND TEMPERATURE SENSORS IN A MICRO GAS TURBINE ENGINE. Xin Zhang, Amit Mehra, Arturo Ayon, Ian Waitz, Massachusetts Institute of Technology, Cambridge, MA.

We have demonstrated a novel through-wafer interconnect scheme that could greatly facilitate electrical contacts in multi-level MEMS devices by allowing direct electrical access to the backside of a wafer. The technique requires a thin layer of conductive material that is patterned in the shape of a resistor and isolated from the underlying substrate via a thick oxide layer. In this paper, we further report the development and characterization of polysilicon materials as the conductive elements for igniters and temperature sensors in a micro gas turbine engine. In experiment one, samples were progressively exposed to temperatures between 200C and 1000C for four hours. The results show that the room temperature resistance of the igniter remained stable until 400C and then became non-linear after a 600C thermal anneal. The initial increase in resistance is explained by changes in the concentration of electrically active dopant atoms. At higher temperatures, the increase in resistance due to dopant atom segregation is countered by the combined and dominating influence of two additional phenomenon: secondary grain growth and crystallographic relaxation. Experiment two was intended to see whether the sensor could be operated stably after an initial high temperature exposure, and to evaluate the effects of multiple annealing cycles. The results show that the room temperature resistance of polysilicon following isochronal anneals settles into a cyclic pattern. This suggests that the resistance is predominantly determined by a combination of the solid solubility limit and the carrier mobility at each annealing temperature, and is therefore uniquely and reversibly determined by the last annealing temperature. Experiment three was intended to determine a stable operating regime for the sensor. The samples were sequentially annealed in an inert ambient at 650C until the resistance was observed to reach the equilibrium value. The samples were then isochronally re-annealed at temperatures between 300C and 650C to see whether the resistance remained stable thereafter. The results show that even after the sensor has been pre-annealed to equilibrium at 650C, its resistance continues to reversibly change between equilibrium values at 525C and 650C, and is uniquely determined by the last annealing temperature in the 400C-650C range.

B5.41

MICROFABRICATED CREVICE FORMERS WITH SENSOR ARRAY. Xiaoyan Wang, Michael L. Reed, Dept of Electrical and Computer Engineering, University of Virginia, Charlottesville, VA; Robert G. Kelly, Jason S. Lee, Dept of Materials Science and Engineering,University of Virginia, Charlottesville, VA.

Microfabrication of crevice corrosion samples is of importance in developing an accurate, comprehensive, and reliable crevice corrosion model, and real-time acquisition of corrosion information is also essential. Solid-state microsensor arrays have been used for detecting potential, pH, and ion concentrations, and their integration into crevice corrosion testing samples will provide real-time spatial information of crevice corrosion. The crevice corrosion testing sample is constructed by coupling a crevice former to a crevice substrate and has a uniform crevice gap. In this paper we present a crevice former incorporating a potentiometric, ion-selective membrane micro-electrode pH sensor array. The crevice former is built on a silicon wafer via microelectromechanical systems (MEMS) fabrication techniques, and consists of an array of eight independent sensors as well as reference electrodes. The array configuration allows for in-situ spatial pH analysis of crevice corrosion based on information from each sensor. Details of the crevice former and microelectrode sensor fabrications will be elaborated.

B5.42
DEVELOPMENT OF CATALYST MATERIALS FOR A MICRO-SCALE COMBUSTION SYSTEM. Christopher Spadaccini, Xin Zhang, Christopher Cadou, Ian Waitz, Massachusetts Institute of Technology, Cambridge, MA.

This work identified heterogeneous catalysis as a promising concept for hydrocarbon combustion at the micro-scale. Catalysts act to increase the Damkohler number by lowering the activation energy associated with the reaction thereby increasing reaction rate. The increase in reaction rate is typically large and diffusion of the fuel-air mixture to the catalyst is the rate-limiting step. To be effective, the catalyst support were configured to present maximum catalyst surface area to the flow while minimizing total pressure loss. Specifically, total pressure loss must be less that 5% for the thermodynamic cycle. The three-wafer combustor reported by us served as an initial test-bed for catalytic combustors. Because the combustors packaging scheme consists of placing it between two conventionally machined invar plates, inserting catalyst material into the combustion chamber can be done outside of the clean room environment. This makes catalyst insertion a low risk operation and the three-wafer device the ideal initial test-bed. Nickel foam pieces conventionally machined to fit in the combustion chamber volume were chosen as the catalyst support material. An e-beam deposition process was used to coat the substrate with platinum. Previous experiments from us on the three-wafer combustor showed that hydrocarbon combustion could not be sustained in the device due to insufficient residence time. With the catalytic foam piece, combustion of both propane and ethylene was possible. We demonstrated the overall combustor efficiencies achieved for both fuels in this device over a range of mass flow rates. Efficiencies in excess of 40% were attained in both cases. Although performance was poor (significantly lower efficiency than hydrogen combustion), it represents the first stable hydrocarbon combustion in this device. In addition, it is important to note that hydrocarbon combustion was stabilized at the same mass flow rate as hydrogen. This resulted in power densities approximately Ω of that obtained with hydrogen rather than the 5-50 fold decrease expected for hydrocarbon-air reactions. This indicates that there is significant catalysis occurring.

B5.43
Abstract Withdrawn.

B5.44
PROCESS DEVELOPMENT OF SILICON-SILICON CARBIDE HYBRID MICRO-ENGINE STRUCTURES. Dongwon Choi, Robert J. Shinavski, S. Mark Spearing, Massachusetts Institute of Technology, Cambridge, MA.

A MEMS-based gas turbine engine is being developed for use as a button-sized portable power generator or micro-aircraft propulsion source. Power densitiesexpected for the micro-engine require high combustor exit temperatures (1300-1700 K) and very high rotor peripheral speeds (300-600 m/s). These harsh operating conditions induce high stress levels in the engine structure, and thus require qualified refractory materials with high strength. Silicon carbide has been chosen as the most promising material for use in the near future due to its high strength and chemical inertness at elevated temperatures. However, techniques for microfabricating single-crystal silicon carbide to the level of high precision needed for the micro-engine are not currently available. To circumvent this limitation and to take advantage of the well-established precise silicon microfabrication technologies, silicon-silicon carbide hybrid turbine structures are being developed using chemical vapor deposition of SiC on silicon wafers and wafer bonding processes. Residual stress control of SiC coatings is of critical importance to all the silicon-silicon carbide hybrid structure fabrication steps since a high level of residual stresses causes wafer cracking during the planarization, as well as excessive wafer bow, which is detrimental to the subsequent planarization and bonding processes. The origins of the residual stresses in CVD SiC layers have been studied. SiC layers (as thick as 30 micron) with low residual stresses (less than 15 MPa) have been produced by controlling CVD process parameters such as temperature and gas ratio. Wafer-level SiC planarization has been accomplished by mechanical polishing using diamond grit and bonding processes are currently under development using interlayer materials such as silicon dioxide or poly-silicon. These process development efforts will be reviewed in the context of the overall microengine development program.

B5.45
TENSILE TEST OF BULK- AND SURFACE-MICROMACHINED SIMOX SILICON FILM USING ELECTROSTATIC FORCE GRIP SYSTEM. Toshiyuki Tsuchiya, Jiro Sakata, Toyota Central Research and Development Laboratories, Inc., Nagakute, Aichi, JAPAN; Mitsuhiro Shikida and Kazuo Sato, Dept of Microsystem Engineering, Nagoya University, Nagoya, JAPAN.

0.14 μm thick SCS (single crystal silicon) films are tensile tested using a thin film tensile tester for sub-micrometer thick films. The tester has been newly designed for this very thin film. It uses a servo-controlled balance for small-force measurement. Its force measurement resolution is less than 100 mN. The tester also has an electrostatic force grip that allows chucking one end of a thin-film specimen by electrostatic force while the other end is fixed to a silicon substrate. This electrostatic force grip system has following advantages: Moderate holding force, easy handing and small damage to the specimen, simultaneous test for many specimens, capable of testing any material (both insulating and conductive), and small misalignment. (self-alignment is expected.) These advantages are more effective with thinner specimens, because the electrostatic force can be controlled by adjusting the voltage applied to the system. Sub-micrometer-thick SCS specimens were fabricated using bulk- or surface-micromachining starting with a (100) SIMOX wafer. The bulk micromachined specimens were released from a wafer by anisotropic etching from backside of the wafer. The specimen size was 0 to 500 μm long, 20 or 50 μm wide and 0.14 μm thick, and the loading axis of the specimen was $< 110 >$. The thinner (narrower) specimens were fabricated by surface micromachining and dry release process using XeF_2 and vapor HF etching to avoid stiction of the specimens. The specimen size was 0 to 300 μm long, 1 to 20 μm wide and 0.14 μm thick. The tensile strength of bulk micromachined specimens is ranged from 1.4 to 4.3 GPa for twelve samples. Young's modulus is 159 GPa using the differential stiffness method.

SESSION B6: PROCESSING TECHNIQUES I
Chair: Arturo Ayon
Tuesday Morning, November 27, 2001
Room 309 (Hynes)

8:30 AM *B6.1
IN-PLANE, BATCH-FABRICATED, MICRO-OPTICAL SYSTEMS VIA DEEP X-RAY LITHOGRAPHY. T.R. Christenson, W.C. Sweatt, Sandia National Laboratories, Photonics Microsystems Technologies, Albuquerque, NM.

The ability to realize refractive optical systems with microfabrication techniques has to date largely been restricted to a single lens surface with optic axis normal to the substrate. An approximation to a two-surface lens has been exploited in this work whereby two crossed plano-cylindrical surfaces are microfabricated in place to allow for distinct focussing in two perpendicular directions across a lens aperture. The term "X-Optics" has been applied to this scheme that uses deep x-ray lithography to form micro lens systems from x-ray photoresists (currently PMMA and SU-8-like). By exposing x-ray photoresists at $\pm$ 45 degrees relative to the substrate normal through an x-ray mask with synchrotron radiation, highly accurate and arbitrary lens surfaces may be defined including positive and negative curvature surfaces. With the lens optic axis oriented parallel to the substrate, a multitude of series elements may be patterned in place to form a multi-element lens directly. At the same time, stops, prisms and optical fiber couplers may be constructed in-place to allow complete fabrication of micro-optical systems with lithographically controlled tolerances. Current characterization includes measurement of lens surface accuracy and roughness as well as relevant optical properties of x-ray photoresist including transmission, refractive index and dispersion. Measurement of a 1:1 test optic with two lenses has revealed the ability to focus the output from a single mode optical fiber to a spot size of 10-12 microns with a focal length of 300 microns and an effective lens diameter of 150 microns.

9:00 AM B6.2
CHARACTERISTICS OF THICK SOL-GEL LEAD ZIRCONATE TITANATE FILMS FOR ANGULAR RATE SENSOR

APPLICATION. S.H. Lee[a], T. Iijima[a], K. Nakamura[b], S. Tanaka[b], M. Esashi[b]. [a]Smart Structure Research Center, AIST, Tsukuba, JAPAN; [b]Tohoku University, Sendai, JAPAN.

Development of reliable actuation methods is one of many challenges in thin film microsystems devices. Piezoelectric actuation using bulk ceramic materials is well known, but widespread use in microsystems requires suitable deposition and integration methods, which are compatible with large-scale manufacturing. In this research we fabricated and measured the PZT thin film devices for application of microelectro mechanical systems. In our research, we had worked the thick Lead Zirconate Titanate (PZT) films of more than thickness of 1 μm using sol-gel method and evaluated the characteristics of the PZT thin film for the angular rate sensor. The crack-free thick sol-gel PZT films were successfully deposited on $Pt/Ti/SiO_2/Si$ structure by multiple spin-coating technique combined with rapid thermal annealing. The thickness of the PZT films was 1.5 μm and crystal structure of PZT film by rapid thermal annealing showed the (100) crystal orientation. The remnant polarization of the PZT films was 12.0 $\mu C/cm^2$. The PZT films were applied to the vibration angular rate sensor structure. The fabrication of PZT thin film angular rate sensor was performed by bulk micromachining method. The vibration of 1.78 μm in amplitude at the resonant frequency of 35.8kHz was obtained. In this case, the driving voltage of $5V_{p-p}$ by bulk piezoelectric materials was applied to the driving electrode with out of phase signal through voltage and inverting amplifier. The oscillating output voltage obtained by external actuation using a stacked piezo actuator showed the values of 0.76V and 0.87V in outer/inner driving electrode at driving voltage of $5V_{p-p}$.

9:15 AM B6.3
MECHANICAL PROPERTIES OF THIN FILM SILICON CARBIDE. Kamili Jackson[a], Richard Edwards[b], William Sharpe, Jr.[a]. [a]Johns Hopkins University, Department of Mechanical Engineering, Baltimore, MD. [b]Johns Hopkins Applied Physics Laboratory, Laurel, MD.

Silicon carbide is a very attractive material for a variety of applications. Thin film silicon carbide was originally considered for use in high power and high temperature electronics because of its large bandgap. Recently, designers of MEMS are also considering use of this material because of its other outstanding characteristics. These unique advantages over silicon include stability at high temperatures, resistance to corrosives, a higher stiffness, and resistance to radiation. However, as with any new structural material, its mechanical properties must be measured for design information. In the case of a thin film, mechanical properties may be different from the bulk polycrystalline material because of the microstructure developed during processing. Numerous electrical properties of silicon carbide have been measured but there are few studies of the mechanical properties, which are needed for structural design. This information will also be important to processors during fabrication refinement to find a correlation between mechanical behavior and deposition technique and parameters. This research measures the elastic modulus and strength of two different silicon carbides using microtensile testing. The first material is a thin film developed by Case Western Reserve University as a material for MEMS devices such as pressure sensors. It is deposited with atmospheric pressure chemical vapor deposition and has a thickness of 0.5 microns. Preliminary results give an average of 380 GPa for elastic modulus and strengths of 2 GPa. The second material is from Massachusetts Institute of Technology and has an average thickness of 40 microns. This material is being used in the development of a microturbine engine. Preliminary results show an elastic modulus of 485 GPa and strengths of 0.7 GPa. In addition to the most recent results, techniques used to obtain these results, microstructure investigations, and a comparison of the materials will be presented.

9:30 AM B6.4
REPLICATION TECHNOLOGY FOR ELECTROFORMING MICROMOLDS. Linda A. Domeier, Sandia National Laboratories, Materials Chemistry Department, Livermore, CA; Marcela Gonzales, Jill M. Hruby and Alfredo M. Morales, Sandia National Laboratories, Microsystems Processing Department, Livermore, CA.

LIGA is a leading technology for producing quality, high aspect ratio metal microparts. Potential applications include high reflectivity micromirrors, high current microswitches or relays, high density acceleration or orientation sensors, and low thermal expansion micro-optical positioning devices. Successful commercialization of such metal microparts, however, will require cost effective replication technologies which eliminate repetitive synchrotron exposure and development steps. One approach to avoid repeated x-ray lithography is the generation of intermediate plastic mold replicates which contain an electroplatable base. In this paper, a technique that incorporates metal microscreens into hot embossing or injection molding processes to produce sacrificial molds with conducting bases and insulating sidewalls is presented. After electroforming and planarizing, the metal microparts are released by dissolution of an intermediate layer of a sacrificial metal such as copper. Both contiguous and non-contiguous features can be replicated and the presence of insulating sidewalls in the replicated molds prevents premature cavity closure during plating. Synchrotron exposure and PMMA chemical development are only required for the initial fabrication of a high aspect ratio microstamp. In hot embossing, a commercial metal microscreen is placed on top of a thermoplastic disk with a microstamp contacting the opposite side of the screen. Under heat and vacuum, the microscreen is pressed by the LIGA-produced microstamp into the softened plastic disk forcing the resin to flow through the holes of the screen and fill the microstamp cavities. In injection molding, the metal screen is placed on top of a LIGA-produced microstamp in a vertical injection molding machine. Additional macroporous metal sheets may be placed on the microscreen to help fixture it and to moderate the plastic flow. Cooling and demolding in both processes produce the desired mold replicate with insulating plastic features mechanically locked to the metal screen. Process parameters including the resin properties, microscreen geometry and the use of multiple metal inserts will be discussed.

9:45 AM B6.5
MICROSTEREO LITHOGRAPHY AND FABRICATION OF 3D MEMS. Vijay K. Varadan and Vasundara V. Varadan, Pennsylvania State University, Center for the Engineering of Electronic and Acoustic Materials and Devices, University Park, PA.

Micro Stereo Lithography (MSL) is a poor man's LIGA for fabricating high aspect ratio MEMS devices in UV curable semiconducting polymers using either two computer-controlled low inertia galvanometric mirrors with the aid of focusing lens or an array of opical fibers. This technique has also been successfully used recently for fabricating 3D metallic and ceramic MEMS devices. Microfabrication techniques such as bulk micromachining and surface micromachining currently employed to conceive MEMS are largely derived from the standard IC and microelectronics technology. Even though many MEMS devices with integrated electronics have been achieved by using the traditional micromachining techniques, some limtations have nevertheless to be underlined: 1) these techniques are very expensive and need spcific installations as well as a cleanroom environment, 2) the materials that can be used up to now are restricted to silicon and metals, 3) the manufacture of 3D parts having curved surfaces or an important number of layers is not possible. Moreover, for some biological applications, the materials used for sensors must be compatible with human body and the actuators need to have high strain and displacement which the current silicon based MEMS do not provide. It is thus natural for the researchers to "look" for alternative methods to make 3D sensors and actuators using polymeric based materials. Applying laser microstereolithogrpahy technique (mSL) to micro fabrication, having improved spatial resolution appears to be an attractive alternative process to manufacture microactuators having a complex geometry. For mSL techniques to be successful as their silicon counterparts, one has to come up with multifunctional polymers. These multifunctional polymers have not only a high sensing capability but also a high strain and actuation performance. With the invention of organic thin film transistor, now it seems possible to fabricate polymeric based MEMS devices with built-in-electronics. Moreover, with combined architecture techniques, one can integrate silicon devices with the polymeric ones without much difficulty. Thus, the sensors can be implemented inside the human body. In this paper, the engineering of such devices will be presented with applications to engineering and medicine.

SESSION B7: ALTERNATIVE MATERIALS
Chair: Todd R. Christenson
Tuesday Morning, November 27, 2001
Room 309 (Hynes)

10:30 AM *B7.1
ADD CERAMIC 'MEMS' TO THE PALLET OF MICROSYSTEMS TECHNOLOGIES. David L. Wilcox, Sr., Jeremy Burdon, Raj Changrani, Steve Dai, Ramesh Koripella, Manny Oliver, Paul VonAllmen, Ceramic Technologies Research Lab, Motorola Labs, Tempe, AZ.

Just as the 40 years of technology developments associated with the electronic application of semiconductor fabrication processes has morphed into micro-electro-mechanical systems in the past dozen years, so it seems may the multilayer ceramic fabrication technology associated with capacitor components and interconnect substrates for the integrated circuit industry, morph into MEMS applications. This paper highlights work underway in Motorola Labs aimed at exploring the potential to develop 3D multilayer ceramic structures to integrate (monolithic and hybrid) multiple functions to create microsystems for wireless, energy and life science applications. By multiple functions, we refer to the ability for a microsystem to perform electronic, fluidic, thermonic, photonic and mechatronic functions. Current capabilities of the multilayer ceramic materials and processes to achieve integrated functionalities for wireless applications will be described including the development of a new dielectric enabling increased performance for wireless applications. Also to be highlighted will be exploratory microscale fuel cell prototypes exploiting advances in the multilayer ceramic lamination and feature forming technologies enabling the insertion of 3D microchannels for microfluidic functions. These prototypes also feature the ability of the technology to provide thermonic functionality for microreactor devices. Feasibility of a light source that can be integrated into the technology platform hinting at photonic applications will be described. Many materials science and engineering advancements are needed to achieve the potential of this "old" but newly "morphing" technology and some of these will be noted.

11:00 AM B7.2
PALLADIUM BASED MICRO-MEMBRANE HYDROGEN GAS SEPARATOR-REACTOR IN A MINIATURE FUEL PROCESSOR FOR MICRO FUEL CELLS. Sooraj V. Karnik, Miltiadis K. Hatalis, Lehigh Univ, Dept of Electrical and Computer Engineering, Bethlehem, PA; Mayuresh V. Kothare, Lehigh Univ, Dept of Chemical Engineering, Bethlehem, PA.

Miniature fuel processors for in situ hydrogen production are of an interest in compact fuel cells, as alternative energy sources for high-end portable devices such as cellular phones and laptops. We report developments on a micro-reactor to produce hydrogen by the reaction of methanol with water. The reactor consists of the following components: (a) mixer/vaporizer of methanol and water; (b) catalytic steam reformer with copper catalyst where gaseous methanol and water react at 250°C to produce carbon dioxide, carbon monoxide and hydrogen; (c) water gas shift reactor/hydrogen separator consisting a palladium membrane supported on a perforated Cu catalyst where unwanted CO produced earlier in the steam reformer gets converted into hydrogen in presence of Cu at 280°C. Hydrogen in turn is separated by the Pd micro-membrane; (d) integrated resistive heaters/sensors and control electronics. In this work, we report progress on the water gas shift reactor/hydrogen separator. Recently designs of micro-membranes based on palladium have emerged for selective hydrogen separation. The novelty of our structure is that we have integrated hydrogen separator as well as carbon monoxide shift reactor in the same structure. Pd based micro-membrane structure is built in silicon substrate, using standard MEMS microfabrication processes. The membrane is a composite of four layers: copper, aluminum, spin-on-glass (SOG) and palladium. Copper, aluminum and SOG layers have a pattern of holes etched into them, so as to have perforations. They serve as a structural support for the main element of the membrane, the palladium film. Copper also acts as a catalyst in the shift reaction that converts unwanted carbon monoxide gas into hydrogen, which in turn is separated by the palladium micro-membrane. The passage of hydrogen through palladium micro-membrane is diffusion limited, hence a pressure gradient of hydrogen needs to be maintained across the micro-membrane without its rupture. An initial fabricated design of the composite micro-membrane withstood pressure gradients up to 10 psi without breaking. A detailed study of the mechanical strength of the micro-membrane with varying thicknesses of the copper, aluminum, SOG and palladium layers will be presented.

11:15 AM B7.3
THE MATERIALS SCIENCE OF "PERMEABLE POLYSILICON" THIN FILMS. George M. Dougherty, Timothy Sands, Dept of Materials Science and Engineering, University of California, Berkeley, CA; Albert P. Pisano, Dept of Mechanical Engineering, University of California, Berkeley, CA.

Polycrystalline silicon thin films that are permeable to fluids, known as permeable polysilicon, have been reported by several researchers. Such films have great potential for the fabrication of difficult to make MEMS structures, but their use has been hampered by poor process repeatability and a lack of physical understanding of the origin of film permeability and how to control it. We have completed a methodical study of the relationship between process, microstructure, and properties for permeable polysilicon thin films. As a result, we have determined that the film permeability is caused by the presence of nanoscale pores, ranging from 10-50 nm in size, that form spontaneously during LPCVD deposition within a narrow process window. The unusual microstructure within this process window corresponds to the transition between a semicrystalline growth regime, exhibiting tensile residual stress, and a columnar growth regime exhibiting compressive residual stress. A simple kinetic model is proposed to explain the unusual morphology within this transition regime. It is determined that measurements of the film residual stress can be used to tune the deposition parameters to repeatably produce permeable films for applications. The result is a convenient, single-step process that enables the elegant fabrication of many previously challenging structures.

11:30 AM B7.4
FABRICATION OF AN ULTRANANOCRYSTALLINE DIAMOND FULLY INTEGRATED MICROTURBINE. N.A. Moldovan, Experimental Facility Division-Advanced Photon Source, Argonne National Laboratory, Argonne, IL; A.V. Sumant, O. Auciello, J. Tucek, D. M. Gruen and J.A. Carlisle, Materials Science Division, Argonne National Laboratory, Argonne, IL; D.C. Mancini, Experimental Facility Division-Advanced Photon Source, Argonne National Laboratory, Argonne, IL.

In many Si-based rotating MEMS devices, the most common failure mode is either due to extensive wear at the point of contact between two rubbing surfaces or due to sticking of these components to each other by high adhesive forces, commonly referred as stiction. These failure modes are directly related to the tribological and mechanical properties of Si. Diamond possesses extreme wear resistant and outstanding mechanical properties. Additionally, diamond is chemically inert, it has very high thermal conductivity and negligible stiction, which makes it attractive for MEMS applications involving rolling and sliding motion of two surfaces in contact. Conventional CVD thin film deposition methods, involving 1%CH_4/99%H_2 plasma chemistry can be used to produce microcrystalline diamond-based MEMS structures. However, films produced by these methods have large grain size ($\geq$ 1μm), high internal stress, poor intergranular adhesion, and very rough surfaces (rms: 0.5-1 μm) and therefore are not particularly suited for MEMS applications. A new ultrananocrystalline diamond (UNCD) coating technology, involving a novel 1%CH_4/99%Ar plasma chemistry, developed at Argonne National Laboratory, produces phase-pure diamond with extremely small grains (2-5 nm) and smooth surfaces (rms roughness ~30-40 nm). These UNCD coatings exhibit tribological and mechanical properties that are ideally suited for the fabrication of rotating MEMS components. We have designed a processing sequence to fabricate a fully integrated UNCD based micro-turbine, without external assembly, by combining lithographic and patterning techniques in conjunction with the capability of lateral growth of UNCD coatings. Various fabrication aspects relating to conformality issues as well as use of sacrificial oxide layers are discussed in detail. The purpose of this work is to develop fabrication processes and demonstrate their applicability to the production of long endurance UNCD-based rotating MEMS devices. *Work supported by the U.S. Department of Energy, BES-Materials Sciences and Office of Transportation Technology, under Contract N0. W-31-109-ENG-38

11:45 AM B7.5
FABRICATION TECHNIQUES AND INTEGRATION PROCESSES FOR A NEW ULTRANANOCRYSTALLINE DIAMOND (UNCD) -BASED MEMS TECHNOLOGY AND CHARACTERIZATION OF UNCD MECHANICAL PROPERTIES. O. Auciello, A.V. Sumant, J. Birrell, D.M. Gruen, J.A. Carlisle, R.C. Birtcher, and M. Angadi Materials Science Division, Argonne National Laboratory, Argonne, IL; N.A. Moldovan and D.C. Mancini Experimental Facility Division, Advanced Photon Source, Argonne National Laboratory, Argonne, IL; H.D. Espinosa and B.C. Prorok, Department of Materials Science Northwestern University, Evanston, IL.

Current Si-based MEMS components exhibit serious performance limitations due to the relatively poor physical, mechanical, chemical and tribological properties of Si. Diamond and diamond-like carbon offer several advantages over conventional Si and even SiC as alternate MEMS materials. However, fabrication of diamond-MEMS components present challenges because conventional CVD processes result in diamond coatings with coarse grains ($\geq$1 μm) and rough surfaces (rms ~0.5-1mm) while vapor-deposited diamond-like coatings can not cover high aspect ratio MEMS features conformally, require high temperature post-deposition processing to relieve stresses, and exhibit lower hardness. A new diamond coating technology developed at Argonne National Laboratory yields phase-pure, low stress ultrananocrystalline diamond (UNCD) coatings with 2-5 nm grains and smooth surfaces (30-40 nm rms), We demonstrated the fabrication of high-resolution UNCD-based 2-D and 3-D MEMS components, such as micro-gears, cantilevers, and microturbines. As part of this work, we investigated ion beam techniques suitable for polishing as-deposited UNCD layers to mirror-like finishing. Both low energy (Kr - 500 eV) grazing incidence and high energy (Ga -30 keV) normal incidence ion bombardment result in UNCD surface roughness $\leq$10 nm. Ion beam polishing resulted in the highest hardness (97 Gpa; very close to the 100 value of single crystal diamond) observed today for our UNCD layers. In situ studies of ion bombardment-induced microstructural changes of UNCD, using a unique integrated ion accelerator/high resolution TEM, revealed that 30 keV Ga ions (with

40 nm range into the UNCD layer) produce a "amorphous" surface diamond layer without graphitization. The local bonding structure of this layer was studied using electron diffraction and NEXAFS. The ion beam polishing method could enable polishing of MEMS structures, which cannot be done mechanically. We will also discuss fabrication issues and mechanical and tribological properties of UNCD for application to MEMS.

SESSION B8: MICROSTRUCTURE AND PROCESSING II

Chair: Thomas E. Buchheit
Tuesday Afternoon, November 27, 2001
Room 309 (Hynes)

1:30 PM *B8.1
MATERIALS ISSUES IN THE APPLICATION OF SILICON NITRIDE FILMS IN SILICON MEMS. David F. Moore, Roger M. Bostock and Ewan H. Conradie, Cambridge University Engineering Department, Cambridge, UNITED KINGDOM.

This talk reviews bulk micromachining of Si, with particular reference to the microassembly and packaging of optical and electronic components. Taking as an example the use of 2.3 micrometer thick silicon nitride microclips to hold an optic fibre in place in a silicon V-groove, the potential of thin film packaging is briefly assessed. The fabrication process is to: [i] deposit a controlled stress silicon nitride layer onto a blank (100) oriented silicon wafer by chemical vapour deposition (standard LPCVD but silicon rich); [ii] pattern the silicon nitride clip mask shape by optical lithography and reactive ion etching; [iii] remove the underlying silicon to form a V-shaped groove with an anisotropic liquid etch process using the silicon nitride as a mask. From the measured deflection of a silicon nitride beam when a force is applied, the Youngs modulus is 400 GPa. The beams are rugged and a clipping force of 10 N per metre of optic fibre length is achieved. This approach to applying the mechanical properties of thin films is compatible with standard thin film technology, enables precise microassembly and has great potential to reduce manufacturing costs in a wide range of sensors and microsystems. In some applications high temperature processing must be avoided, even early on in the manufacturing process. Using low temperature processing, robust clips from silicon nitride made by plasma enhanced chemical vapour deposition (PECVD and hydrogen rich) are made but the Youngs modulus is 40 GPa and the clipping force correspondingly small. The talk will conclude by assessing the prospects for other thin film materials in mechanical MEMS and packaging.

2:00 PM B8.2
HIGH YOUNG MODULUS NANOPILLAR FABRICATED USING FOCUSED-ION-BEAM INDUCED CHEMICAL VAPOR DEPOSITION. M. Ishida, J. Fujita, Y. Ochiai, NEC Fundamental Res. Labs., Tskuba, JAPAN; T. Kaito, Seiko Instruments Inc., Shizuoka, JAPAN; S. Matsui, Himeji Inst. of Tech., Hyogo, JAPAN.

We have achieved a Young modulus exceeding 600 GPa in amorphous carbon nanopillars. The pillars were fabricated by irradiating a 30 keV Ga^+ focused ion beam (FIB) (SMI2050, Seiko Instruments Inc.) and simultaneously supplying a carbon-containing precursor gas (phenanthrene, $C_{14}H_{10}$) onto a silicon substrate. Pillar diameter was typically 100 nm, and length (10 $\sim$ 40 μm) can be controlled by adjusting the growth time. We have already confirmed that the product of the FIB-induced chemical vapor deposition (CVD) consists of amorphous carbon with a certain fraction of sp^3 bonds[1]. To investigate the Young modulus of the nanopillars, we observed the mechanical resonance of the pillars with a scanning electron microscope (SEM). A small piezoelectric actuator attached to a sample holder can induce the mechanical vibration. There was also spontaneous vibration from thermal excitation and/or environmental noises. With an electron beam adjusted so that it was incident on the side edge of a vibrating pillar, we were able to observe the oscillating output of the secondary electron detector. An analysis of the detector output spectrum indicated a distinct peak at the fundamental resonant frequency, and a mechanical Q-factor ranging from 200 to 1400, depending on pillar size. From the resonant frequency, we estimated the Young modulus of the pillars under the assumption that the density would be the same as that of graphite (2.3×10^3 kg/m^3). The Young modulus had strong dependence on both growth rate and ion-beam current. The highest Young modulus exceeded 600 GPa, which is almost same as that of diamond-like carbon (DLC) film, and was obtained from a pillar grown at 1 μm/min growth rate and 1 pA beam current. This high Young modulus was obtained despite the modest density assumed in the estimation. [1] S. Matsui *et al.*, J Vac. Sci. Technol. B **18**, 3181 (2000).

2:15 PM B8.3
MECHANICAL PROPERTIES OF 0.1 MICRON THICK SIMOX FILM MEASURED USING ON CHIP TENSILE TEST SYSTEM. Junpei Amano, Taeko Ando, Mitsuhiro Shikida, Kazuo Sato, Dept of Microsystem Engineering, Nagoya Univ, Nagoya, JAPAN; Toshiyuki Tsuchiya, Toyota Central R&D Labs Inc, Nagakute, Aichi, JAPAN.

We performed tensile test of 0.1 μm thick single crystal silicon film oriented in (110) direction on a SIMOX wafer. We integrated a thin film specimen and loading mechanism on a same silicon chip. The chip consists of a specimen, a loading lever, a pair of torsion bars and a supporting frame. As an external load is applied perpendicular to the lever, the lever rotated around the axis of the torsion bars and specimen is uniaxially stretched in the horizontal direction. Both ends of the specimen are connected to the loading mechanism, thus no slippage occurs at griped ends. The specimen was stretched until it fractured. Young's modulus, fracture stress and fracture strain are obtained. The results are compared to the values obtained from thicker specimens. We developed a fabrication process of the testing device starting from SIMOX wafer. Three steps of etching were applied; first for patterning the specimen profile, second and third for patterning loading mechanism from front and rear side of the wafer successively. An oxide layer of the SIMOX was used as an etching stop layer that allowed us to release self-supporting structure of the 0.1 micron thick specimen from substrate. The size of the chip was 15 mm square and 500 μm thick, and the dimensions of the specimen was 50 or 100 μm long, 45 μm wide, and 0.1 μm thick. One of the advantages of the on-chip tensile testing system is that there is no slippage at grip ends of the specimen, thus it is possible directly to measure fracture strain of the film material. The measured fracture strain was 3.6%.

2:30 PM B8.4
SILICON MICROFABRICATION: LASER ABLATION VS. INDUCTIVELY COUPLED PLASMA (ICP) ETCH.
Megan M. Owens, Joseph W. Soucy, Thomas F. Marinis, The Charles Stark Draper Laboratory, Electronics Packaging and Prototyping Division, Cambridge, MA.

Mechanical prototypes of a silicon interposer device, having high-aspect ratio straight walls, were required to qualify various packaging assembly options. The ideal solution would yield expendable, quickly-made, and relatively inexpensive replica parts. This need led to the investigation of the micromachining of silicon via laser ablation. We sought to optimize the results by varying several parameters, the most influential of which was shown to be laser wavelength. Silicon wafers of thickness $0.020''$ were machined using laser wavelengths of 10,600 nm, 1064 nm, and 355 nm. As was predicted according to the theoretical silicon absorption profile, the best results were obtained using 355 nm wavelength light with an ultraviolet laser source. Several post-machining cleaning routines, to remove melted silicon debris, were evaluated as well. Pictorial results of both laser processing and cleaning are presented and discussed. ICP was concurrently investigated as a viable alternative to laser micromachining. Resultant cuts were markedly cleaner and straighter than those induced by laser; additionally, cut features were more well-defined. Pictoral results are compared and contrasted with those of laser ablation. Cost and time are traded for quality when choosing between laser ablation and ICP etch. Therefore, process choice should be application-dependent. While laser micromachining is significantly lower in cost and manufacturing time, the process does produce residual thermal stress surrounding the cut region, machined edges are relatively rough, and fine features are more difficult to produce. Laser ablation is an attractive manufacturing option for quick-turnaround prototyping as well as high-volume production; ICP is better suited to producing high-definition parts without potential for thermal stress damage.

SESSION B9: MECHANICAL PROPERTIES II

Chair: William N. Sharpe
Tuesday Afternoon, November 27, 2001
Room 309 (Hynes)

3:15 PM B9.1
A NOVEL FRACTURE TOUGHNESS MEMS DEVICE: A DEFINITIVE TEST FOR THE ABSENCE OF STATIC STRESS CORROSION CRACKING IN POLYSILICON. Justin Bellante, Hal Kahn, Case Western Reserve University, Dept. of Materials Science and Engineering, Cleveland, OH; Roberto Ballarini, Case Western Reserve University, Dept. of Civil Engineering, Cleveland, OH; Arthur H. Heuer, Case Western Reserve University, Dept. of Materials Science and Engineering, Cleveland, OH.

Reliable fracture toughness data for thin films used in MEMS devices are important for understanding the severity of processing-induced, strength controlling defects and consequently, the maximum load such MEMS devices can support. Stress corrosion cracking could also play an important role in determining the long-term reliability of MEMS devices. Doubly clamped, tensile "dog-bone" specimens containing pre-determined tensile residual stresses and with a rigorous fracture mechanics design have been fabricated using typical MEMS surface micromachining processes. A Vickers microindenter was used to propagate a crack from the substrate neighboring the midsection of the beam into the edge of the beam. Due to the stochastic nature of the pre-crack, initial crack lengths varied from approximately 1/3 to 2/3 of the beam width. The beam portion of the device was subsequently released from the substrate using wet etching. The stress intensity at the crack tips upon release depends on the residual tensile stress level in the tensile microdevices and the crack length. Due to the geometry of the fixed grip test, the stress intensity vs. crack length curve increases to a maximum at about half the beam width, and then decreases. The dependence of stress intensity upon crack length enabled cracks of a critical length to propagate (and sometimes arrest), and allowed the critical stress intensity, also called the fracture toughness, K(crit), to be determined. Experiments on polycrystalline silicon yielded a K(crit) of 0.94 ± 0.03 MPa $m^{1/2}$, while for polycrystalline SiC, K(crit) was in the range 2.75 to 2.82 MPa $m^{1/2}$. Polysilicon specimens containing cracks where K was just at or below K(crit) were placed in a 90 percent relative humidity environment, and examined for crack growth; none was found.

3:30 PM B9.2
A COMPARISON BETWEEN TENSILE AND BULGE TEST METHODS FOR THIN FILMS. Stuart B. Brown, Exponent Inc., Natick, MA; Richard Edwards, Johns Hopkins Univ, Applied Physics Laboratory, Baltimore, MD; Ranjan Panth, Jorgen Bergstrom, Exponent, Inc., Natick, MA; George Coles, William N. Sharpe, Jr., Department of Mechanical Engineering, Johns Hopkins Univ, Baltimore, MD.

Accurate and reliable determination of thin film mechanical properties remains a substantial challenge to the MEMS industry. There are a variety of techniques available to measure mechanical properties, including cantilever deflection, microtensile testing, bulge testing, and the use of specific test structures that can be actuated on-wafer to measure elastic moduli, residual stresses, fracture toughness, and strength. There are, however, very few efforts that have compared any of these techniques to evaluate their relative advantages and accuracies. This article compares two property measurement techniques, microtensile testing and bulge testing, using a silicon nitride thin film as test material. Test specimens were fabrication for the two techniques on the same processed wafers. The microtensile testing was performed at Johns Hopkins University, and the bulge testing was performed at Exponent, Inc. The Young's Moduli determined from the two techniques were very close, yielding a value of 257±5 GPa for the microtensile technique and 258±1 GPa for bulge testing. The two techniques will also be discussed both for their capabilities for measuring additional properties and for sources of measurement variation and error.

3:45 PM B9.3
DEFORMATION BEHAVIOR OF SILICON DURING MICROINDENTATION AT ELEVATED TEMPERATURES. Vladislav Domnich, Yury Gogotsi, Drexel Univ, Dept of Materials Engineering, Philadelphia, PA; Yvonne Aratyn, Waltraud Kriven, Univ of Illinois, Dept of Materials Science and Engineering, Urbana, IL.

Silicon is the primary engineering material for fabrication of integrated circuits, microelectromechanical systems (MEMS), as well as many optical and photoelectric devices, which emphasizes the importance of the investigation of Si behavior during contact interaction with other materials. In particular, it is known that the deformation of Si during contact loading at room temperature is often mediated by pressure-induced transformation into a ductile metallic phase (Si-II), which exists only under load and transforms into metastable crystalline phases (Si-XII and Si-III) or amorphous material during unloading. Here, we use a combination of Raman microspectroscopy and point-force indentation testing to investigate the deformation behavior of silicon at the microscale in the temperature range of 25 to 700°C. We show a correlation between the shape of the observed hardness vs. temperature dependence of Si and the phase composition within the indented area. Transformation kinetics and phase stabilities are discussed in detail. In particular, our results suggest the formation of a new, so far unindentified, crystalline Si phase during contact deformation at elevated temperatures. This phase can be quenched to the room temperature and preserved at ambient conditions within the hardness imprints or scratching microgrooves on the Si surface.

4:00 PM B9.4
LOCALIZED STRESS MEASUREMENTS OF Si MEMS STRUCTURES. B. Yang, A.R. Woll, P. Rugheimer, C.H. Lee, A. Lal and M.G. Lagally, University of Wisconsin-Madison.

We report what we believe to be the first use of low-energy electron microscopy (LEEM) to measure local stress on the surface of strained MicroElectroMechanical systems (MEMS) structures. MEMS devices have many potential novel applications: an issue in many of them is the local stress. We fabricate MEMS structures on Silicon-on-Insulator (SOI) (001) substrates. Differing amounts of uniaxial stress are imparted to these structures by deposition of Si_3N_4 on selected areas. LEEM provides a real-time quantitative probe of surface stress in these systems because it is able to measure quantitatively the ratio of areas of 2x1 to 1x2 reconstruction on Si (001) and because this ratio is affected by uniaxial stress. We present results of temperature and stress change data on these MEMS structures. We extend these results to Si MEMS structures on which heteroepitaxial 3D SiGe islands are grown via Molecular Beam Epitaxy (MBE). The surface morphology is followed in-situ during growth with RHEED and ex-situ post growth with atomic force microscopy (AFM) and scanning electron microscopy (SEM). Regions in which the substrate is subjected to tensile stress show a different local growth morphology than regions in which the substrate is not strained. Supported by NSF-MRSEC and by ONR.

4:15 PM B9.5
THE EFFECT OF CRYSTALLOGRAPHIC TEXTURE ON PIEZOELECTRIC CERAMICS. R. Edwin Garcia, W. Craig Carter, Massachusetts Institute of Technology, Cambridge, MA; Stephen Langer, National Institute of Standards and Technology, Gaithersburg, MD.

The piezoelectric response of a homogeneous phase may be understood through either empirical or first principles calculations. However, the macroscopic response of a polycrystalline piezoelectric material is not directly amenable to direct analysis because of constraints due to compatibility. In a polycrystal, the state of stress will depend on geometrical effects such as grain size and morphology as well as the degree of crystallographic alignment (texture). Furthermore, the microscopic stress state will also depend on residual stresses from processing and whether microscopic defects develop, either as a result of processing or from subsequent poling. Prediction of macroscopic response requires a model that incorporates all pertinent spatial and crystallographic aspects of microstructure and allows coupling between fields indirectly through coupled single crystal material properties. We examine the role of microstructure in polycrystalline piezoelectric materials by combining finite element techniques with image and orientation data from computer generated microstructures. We demonstrate a technique for predicting microstructural effects on the effective piezoelectric moduli under isothermal conditions by studying the effect of texture and number of grains with a reduced set of microstructural parameters by using the March-Dollase distribution function for a one textured axis. It is analyzed the entire range of Multiples of Random Distribution (MRD) for $BaTiO_3$ and PZN-PT. We propose scaling relations between the different physical parameters of the system in order to reduce the number of degrees of freedom.

4:30 PM B9.6
A CONSTITUTIVE MODEL FOR THE MECHANICAL BEHAVIOR OF SINGLE CRYSTAL SILICON AT ELEVATED TEMPERATURE. Hyung-Soo Moon, Lallit Anand, MIT, Dept of Mechanical Engineering, Cambridge, MA; S. Mark Spearing, MIT, Dept of Aeronautics and Astronautics, Cambridge, MA.

Silicon in a single crystal form is the material of choice for the first (demonstration) device of the MIT microengine project. However, silicon is not an ideal material for the extreme environment characterized by high temperature and stress because it has a relatively low melting temperature. In addition, preliminary work indicates a tendency to undergo localized deformation by slip band formation. Thus it is critical that we obtain a better understanding of the mechanical behavior of this material at elevated temperature in order to further exploit its structural capability. Creep tests in uniaxial compression with n-type single crystal silicon were conducted over a temperature range of 900 K to 1200 K and a normal stress range of 10 MPa to 100 MPa. The material orientations chosen for the compression specimen are $< 100 >$ and $< 111 >$ in the axial direction. The creep tests revealed that primary and secondary creep dominates for the experimental ranges of temperature, stress, and time of interest. It is believed that two major creep mechanisms are activated during the creep: first, fast dislocation multiplication is responsible for the initial accelerating creep rate, and second, creep via a complicated dislocation web yields a decelerating creep rate more similar to the conventional creep of metals. The activation energy of 213 kJ/mol and stress exponent of 2.96 obtained from the highest creep strain rates are in good agreement with Alexander and Hassen's results [Solid State Physics, pp 27-158, 1968]. A constitutive model that accounts for these two mechanisms is under development. This model is to be implemented in a subroutine UMAT of ABAQUS/EXPLICIT in support of the design of high temperature turbine structure of the microengine.

4:45 PM B9.7
FRACTURE STRENGTH OF POLYSILICON THIN FILMS AT STRESS CONCENTRATIONS. Joerg Bagdahn and William N. Sharpe Jr., Johns Hopkins Univ, Dept of Mechanical Engineering, Baltimore, MD.

Fracture of brittle materials is caused by the unstable propagation of cracks, which start at surface or volume defects. If the defects have a random distribution of size, orientation and location, the strength will increase as the size of the affected surface or volume is reduced. Greek at al. (1997), Tsuchiya et al. (1997) have demonstrated this for polycrystalline silicon tensile specimens and Jones et al. (1999) have examined the application of Weibull statistics to bend specimens, also of polysilicon. Microelectromechanical systems (MEMS) have stress concentrations and these will naturally affect the strength of the microdevice. A systematic study of the effects of stress concentrations on the strength of components is therefore in order and has been started. Three kinds of polysilicon specimens were used in this investigation - straight tensile specimens, tensile specimens with a central hole 5.0 microns in diameter, and a double-notched tensile specimen with semicircular notches 2.5 microns in radius. Two widths, 20 and 50 microns, were tested. The maximum stress was calculated using the stress concentration factors obtained from handbooks and by finite element analysis. A failure probability analysis based on the Weibull theory was carried out. It was found that the Weibull strength of the smaller straight samples was higher than the strength of the wider samples. The Weibull strength computed at the stress concentrations was significantly higher than the strength of the straight specimens. The local strength of the semicircular-notched samples was slightly higher than the samples with holes. Finite element analyses were invoked to analyze the influence of the stress field in the vicinity of the stress concentrators. The origins of failures were investigated using fractographic methods. This research was conducted under the sponsorship of DARPA F30602-99-2-0553 and by the Feodor Lynen program of the Alexander von Humboldt Foundation.

SESSION B10/P6: JOINT SESSION
SURFACE ENGINEERING ISSUES IN MEMS STRUCTURES AND DEVICES
Chairs: Kathyrn J. Wahl and S. Mark Spearing
Wednesday Morning, November 28, 2001
Room 311 (Hynes)

8:30 AM *B10.1/P6.1
SURFACE TREATMENTS FOR MICROSYSTEMS: COATING ISSUES AND TRIBOLOGICAL MEASUREMENT METHODS. M.T. Dugger and S.V. Prasad, Sandia National Laboratories, Albuquerque, NM.

Several fabrication routes are available to realize mechanical devices with structural elements that are microns to tens of microns in size. In particular, recent developments in surface micromachining (SMM) and patterned electrodeposition (LIGA) have resulted in complex machines with actuators and countermeshing gears. Such machines include impacting and sliding surfaces in which friction and wear will determine the machine's performance and reliability. The materials in contact are usually determined by processing constraints or material properties such as strength, density or magnetic behavior, rather than friction and wear performance. Efficient operation of these devices will require an engineered surface that is tailored to meet friction and wear requirements. Application of surface treatments to three-dimensional shapes at this size scale presents significant challenges, including modification of hidden surfaces, control of treatment thickness relative to gap dimensions, and the generation and trapping of third bodies. Further, understanding the behavior of surface treatments at pressure and velocity regimes relevant to microsystem operation requires the development of new tools for quantifying interface performance. A focus of our research is the development of special SMM and LIGA structures that contain isolated tribological contacts from which the friction forces may be extracted. Methods of quantifying static and dynamic friction in surface micromachined contacts will be shown, including measurements where the interface is parallel to the wafer surface (planar) and where the interface is perpendicular to the wafer surface (sidewall). Newly developed techniques for quantifying dynamic friction in LIGA contacts will be presented. Use of these approaches will be illustrated with several examples, including coupling agents and selective tungsten to modify the frictional behavior in polycrystalline silicon contacts, and a diamondlike nanocomposite coating for LIGA nickel structures.

9:00 AM B10.2/P6.2
DIAMOND LIKE NANOCOMPOSITE (DLN) COATINGS FOR LIGA MEMS: TRIBOLOGICAL BEHAVIOR AND COATINGS METHODOLOGY. Somuri Prasad, Todd Christenson, Sandia National Laboratories, Albuquerque, NM.

Many microelectromechanical systems (MEMS) fabricated by LIGA utilize electrodeposited metals such as nickel and Ni alloys. While Ni alloys may meet the structural requirements for MEMS, their tribological (friction and wear) behavior remains somewhat undefined. In a number of Microsystems applications such as gear trains, comb drives and transmission linkages, tribological considerations, particularly amongst sidewalls, are of paramount importance. The objective of this research is to device a novel coatings strategy that can be integrated into the mainstream LIGA technology to coat the sidewalls of intricate LIGA MEMS elements. As a first step in this direction, we applied a 100 nm thick diamond like nanocomposite (DLN) coating on a wafer containing LIGA MEMS elements by commercial plasma enhanced chemical vapor deposition (PECVD) technique. The MEMS elements were subsequently released from the wafer per standard LIGA process after backsputtering to clear the coating on the planar surface. Coverage of the DLN coating on the sidewalls was confirmed by cross sectional TEM analysis. Focused ion beam technique was used to prepare XTEM specimens from a DLN coated LIGA gear tooth. Tribological studies were conducted on planar test coupons using a ball-on-disk apparatus in different test environments. As compared with pure electrodeposited Ni, DLN coated Ni showed significant improvements in friction (0.04 versus 0.6-1.2), debris generation and stick-slip behavior.
*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.

9:15 AM B10.3/P6.3
CONFORMAL DEPOSITION OF AMORPHOUS HYDROCARBON BASED COATINGS ON METALLIC HIGH-ASPECT-RATION MICRO-SCALE STRUCTURES (HARMs) BY LIGA MICRO-FABRICATION. D.M. Cao, T. Wang, W.J. Meng, K.W. Kelly, Mechanical Engineering Department, Louisiana State University, Baton Rouge, LA.

A high-density plasma assisted hybrid CVD/PVD tool was used to deposit Ti-containing amorphous hydrocarbon (Ti-C:H) coatings comformally over electrodeposited Ni high-aspect-ratio micro-scale structures (HARMs) fabricated by the deep X-ray lithography based microfabrication technique LIGA. Mechanical properties and tribological characteristics of Ti-C:H coatings are reviewed. Ti-C:H deposition over Ni HARMs substrates was studied as a function of the HARMs dimension and aspect ratio. Potential applications of surface engineered metallic HARMs will be discussed.

9:30 AM B10.4/P6.4
DEPOSITION OF LOW SURFACE ENERGY, WEAR-RESISTANT FILMS ON MEMS-LIGA DEVICES USING AN ENERGETIC PULSED PLASMA IMMERSION PROCESS. Kumar Sridharan, University of Wisconsin, Madison, WI; Alfredo Morales, Sandia National Laboratory, Livermore, CA; Erik Wilson, University of Wisconsin, Madison, WI; and Joseph Woodworth, Sandia National Laboratory, Albuquerque, NM.

Low surface energy diamond-like carbon films modified with fluorine, and exhibiting moderately high hardness, have been deposited on gear-shaped MEMs-LIGA parts. Film deposition was performed with the non-line-of-sight plasma immersion ion implantation and film deposition process operated in the low energy regime, using a mixture of acetylene and a fluorinated precursor gas. The film-substrate interface region was compositionally-graded by adjustments in energy and deposition precursor content. Conformality to the device features was observed, and improvements in conformality of the film to the contours of the devices were achieved by adjusting the plasma process parameters. Conformality studies are being supported by modeling of plasma-target interactions during pulse-biasing of the MEMs-LIGA devices in the plasma, with the goal of optimizing process parameters. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94-AL8500. This work supported by SNL grant No. 16546.

9:45 AM B10.5/P6.5
STUDIES OF TRIBOLOGICAL PROCESSES ON ULTRA-NANOCRYSTALLINE DIAMOND (UNCD) THIN FILMS FOR MULTIFUNCTIONAL MEMS DEVICES. A.V. Sumant, O. Auciello, A.M. Dhote, D.M. Gruen, and J.A. Carlisle, Materials Science Division, Argonne National Laboratory, Argonne, IL; A. Erdemir, J. Andersson, and R.A. Erck, Energy Technology Division, Argonne National Laboratory, Argonne, IL; M.N. Gardos, Raytheon Electronic Company, El Segundo, CA.

Many MEMS devices involve rolling, sliding, rotating or bending operations that impose severe constrains on their performance and durability imposed by the tribological and mechanical properties of the base materials. In terms of mechanical and tribological properties,

diamond clearly outperforms almost all materials. Additionally, diamond has other outstanding properties such as chemical inertness, very high thermal conductivity, high flexural strength and negligible stiction. All these properties make diamond an outstanding candidate material for fabrication of MEMS devices. Conventional CVD thin film deposition methods can be used to fabricate diamond MEMS structures. However, these films have large grain size, high internal stress, poor intergranular adhesion, and very rough surfaces, and are consequently ill-suited for MEMS applications. We have recently demonstrated a novel ultrananocrystalline diamond (UNCD)-based MEMS technology based on a UNCD coating technology developed at Argonne National Laboratory. This technology produces phase-pure diamond with extremely smooth surfaces (RMS roughness ~30-40 nm) with morphological and mechanical properties that are ideally suited for MEMS applications. However, before these UNCD-MEMS components are ready for integration into commercial MEMS devices, it is necessary to perform extensive research on their mechanical and tribological properties in various gas environments and under a wide range of test conditions. We have performed comprehensive friction and wear studies on UNCD films in high vacuum and controlled atmospheres (e.g., N_2, O_2, H_2, OH_2, inert gases) at room temperature to explore the specific roles of surface adsorbates in frictional properties. We will discuss results from tribological tests in correlation with studies of the effect of surface adsorbates on the UNCD friction coefficient, using integrated time of flight mass spectroscopy of recoiled ions (MSRI) and X-ray photoelectron spectroscopy techniques to determine the nature of the adsorbates and their chemical state on the UNCD surface.
Work supported by the U.S. Department of Energy, BES-Materials Sciences and Office of Transportation Technology, under Contract N0. W-31-109-ENG-38.

10:30 AM *B10.6/P6.6

MECHANICAL TESTING OF FREE-STANDING THIN FILMS.
W.N. Sharpe, Jr., Johns Hopkins University, Department of Mechanical Engineering, Baltimore, MD.

Microelectromechanical systems (MEMS) are usually planar microdevices with the mechanical or structural part consisting of freestanding thin-film material. Young's modulus and Poisson's ratio are needed for elastic response, and the strength of the material is needed to determine the allowable forces and displacements. Tensile testing is the preferred approach for structural materials because its uniform stress and strain fields enable direct determination of mechanical properties according to their definitions. Tensile testing of small thin-film specimens presents three challenges - preparation and handling of the specimen, measurement of small forces, and measurement of strain in the specimen. The author and colleagues at Hopkins have developed techniques and procedures for tensile testing of polysilicon, silicon nitride, and silicon carbide thin films. It is easier to measure mechanical properties of MEMS materials indirectly by modeling microdevices and extracting properties. One can fabricate a comb-driven resonant structure and use the measured resonant frequency to determine the modulus. Thin membranes of different shapes can be pressurized, and the measured displacements used to determine both Young's modulus and Poisson's ratio. Cantilever or fixed-end beams can be deflected electrostatically to measure modulus. However, none of these indirect approaches permit simultaneous measurement of all three properties (modulus, ratio, strength) as does the tension test. This presentation summarizes the current state-of-the-art in terms of the test methods and the values of the properties of polysilicon and other materials used in MEMS. Similar techniques have been used to conduct tensile tests of the bond-coat layer in thermal barrier coatings and those will be discussed. This research was conducted under the sponsorship of DARPA F30602-99-2-0553.

11:00 AM B10.7/P6.7

ROLE OF MICROSTRUCTURE IN FRACTURE AND FATIGUE PROCESSES IN MICRON-SCALE SILICON STRUCTURAL FILMS.
Christopher L. Muhlstein, University of California, Dept of Materials Science and Engineering, Berkeley, CA; Eric A. Stach, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA; William G. Stratton, University of Minnesota, Dept of Materials Science and Engineering, Minneapolis, MN; Stuart B. Brown, Exponent Inc., Natick, MA; Robert O. Ritchie, University of California, Dept of Materials Science and Engineering, Berkeley, CA.

Fracture and fatigue resistance are important properties for the design and long-term durability of micromechanical structures. A thorough understanding of these phenomena is particularly important to avoiding failure of brittle, silicon-based, structural films in the high-performance and safety critical applications commonly found in computing, biological, and aerospace components. Recent work by some of the authors (CLM,EAS,SBB,ROR) have established that such thin, micron-scale, films of single- and polycrystalline silicon are susceptible to premature failure under cyclic loading conditions, with studies focused on characterizing the fatigue life behavior and mechanisms of fatigue crack initiation and growth. These investigations emphasized the importance of the surface chemistry and environment in the fatigue process; in addition, the underlying microstructure was found to play an important role in the failure of the material. The current work is centered on examining the general features of the polycrystalline silicon thin film microstructure that are relevant to the fracture and fatigue processes. Results are based on fifty stress-life fatigue tests that have been conducted on surface micromachined polysilicon films using Exponent fatigue test structures with a variety of surface and environmental conditions. High voltage transmission electron microscopy was used to observe the crack path, surface morphology, and underlying microstructure of polycrystalline silicon. In addition, cross-sections of the patterned films were thinned and evaluated using conventional transmission electron microscopy. Particular attention is given to the importance of grain boundary structure, oxide precipitates, and grain morphology in fracture and fatigue of polysilicon structural films.

11:15 AM B10.8/P6.8

STICTION IN AMORPHOUS-DIAMOND (A-D) THIN FILM STRUCTURES.
R.V. Ellis, T.A. Friedmann, J.P. Sullivan, M.P. de Boer, J.A. Knapp, W.K. Schubert, M. Mitchell, Sandia National Labs, Albuquerque, NM.

Stiction between surfaces of moving thin film structures places a critical limit on the performance and reliability of micromechanical devices. Thin film amorphous-Diamond (a-D) structures may be less susceptible to stiction problems than structures built with other materials, since a-D film surfaces are nearly hydrophobic as deposited, and they do not develop an oxide. Our preliminary observations were that surface micromachined a-D structures can be released with a simple dehydration bake at 200 - 400°C under inert atmosphere, and that these structures do not re-adhere over time. To better quantify these observations, we are measuring adhesion and surface wetting as a function of surface termination. These measurements are made using an array of singly-clamped a-D cantilever beams that are electrostatically brought into contact with a ground plane. Two ground plane materials are being tested, both poly-Si and a-D. An interferometric microscope is used to measure the length of the beam that is adhered to the ground plane. Using this measurement and simple beam mechanics within a Griffith crack formalism, one can extract the adhesion energy per unit area. We will discuss the effects of various surface treatments and humidity on the adhesion in these systems and compare the results to those obtained from poly-Si based devices. We are also investigating the number of contacts to failure and the effects of electrical current across the contact.
*This work was supported by the U.S. DOE under contract DE-AC04-94AL85000 through the Laboratory Directed Research and Development Program, Sandia National Laboratories.

11:30 AM *B10.9/P6.9

SURFACE MECHANICAL PROPERTIES AND TRIBOLOGY: A COMBINED NANOSCALE AND MACROSCALE APPROACH.
Kathryn J. Wahl, U.S. Naval Research Laboratory, Washington, DC.

Mechanical properties of surfaces and interfaces are important for understanding the behavior of tribological contacts, where changes in interfacial properties can result from surface treatments, contaminant films, or sliding processes. At all scales - from atomically thin surface films to chunks of wear particles - interfacial mechanics and dynamics play an important role in friction and wear. To address these problems, we are combining two approaches: 1) *in situ* macroscopic tribological studies allowing visualization and chemistry of the buried interface and 2) "hybrid" nanoindentation, coupling high spatial resolution and surface sensitive, quantitative mechanical properties measurements of films as thin as a few nanometers. By combining these techniques, the macroscopic tribological behavior can be correlated with mechanical properties of the nanoscale films and structures, e.g. the "third bodies" found in the sliding interface. In this presentation, I address the issues of measuring and interpreting the mechanical response of ultrathin films and nanostructures. Combining these nanomechanical techniques with the *in situ* tribological studies yields a powerful approach to examine worn surfaces and interpret tribological response. For example, one can learn "how" third body films form on the stationary counterface, "what" are their composition and mechanical properties, and "why" they provide low friction and prevent wear. Such studies can provide a better understanding of the transformation and behavior of tribological interfaces, which is a key need for devices and applications at all scales.