SYMPOSIUM EE

Materials Science of Microelectromechanical System (MEMS) Devices III

November 26 – 28, 2000

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* Invited paper

TUTORIAL

FT EE: MICROELECTROMECHANICAL SYSTEMS (MEMS) - FABRICATION AND "HOT TOPICS"
Sunday, November 26, 2000
9:00 a.m. - 4:00 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 BioMEMS and MicroOptics will be presented.

The morning section on fabrication will cover photo-lithography, 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 BioMEMS section will highlight new research areas including drug delivery systems, cellular analysis, surgical microtools and microfluidic devices. The MicroOptics section will emphasize trends and future prospects in optical communications. MEMS optical device characteristics and system architectures will be discussed.

Instructors:
Arturo Ayon, MIT
Randy Giles, Lucent Technologies
Carol Livermore, MIT
Murat Okandan, Sandia National Laboratories

SESSION EE1: METROLOGY
Chair: Maarten P. de Boer
Monday Morning, November 27, 2000
Room 206 (Hynes)

8:30 AM *EE1.1
WHY MEASURING CONSTITUTIVE PROPERTIES IN MEMS DEVICES IS HARD, AND WHAT WE CAN DO ABOUT IT.
Stephen D. Senturia, Massachusetts Institute of Technology, Department of Electrical Engineering and Computer Science, Cambridge, MA.

The world of microfabrication conjures up an image of infinite dimensional precision on microscopic scales. Unfortunately, this image is inaccurate. As a percentage of dimension, the precision with which components of microelectromechanical systems (MEMS) can be fabricated is typically orders of magnitude worse than for macrofabricated macro-scale components. This creates several questions for constitutive property measurement: (1) Metrology–how can we determine the actual dimensions of the specimen we are testing? (2) Modeling–how can we efficiently model the real structure, rather than an idealization of that structure? and (3) Error checks–how can we test our data for accidental inclusion of systematic errors in measurement? The key to all three questions is the same: extract constitutive properties from measurements on a set of devices with systematically varied dimensions. The approach will be described with examples drawn from measurements of residual stress and elastic constants of thin-film materials using membrane load-deflection methods, electrostatic pull-in measurements, and resonance frequency shifts.

9:00 AM *EE1.2
MICROMECHANICAL DEVICES FOR FORCE MEASUREMENT.
Thomas Kenny, Stanford Univ, Dept. of Mechanical Engineering, Stanford, CA.

In recent years, many researchers have adapted lithography, deposition and etching techniques from the IC processing community to the fabrication of micromechanical sensors. Many of the signals that these sensors are intended to detect are expressed as forces which stress or deflect the micromechanical structure. As sensors are miniaturized, these forces naturally become smaller, and techniques for detection are required to improve. Our research group has been engaged in a variety of activities, all of which share an interest in improving the force detection capability of microinstruments. In this talk, an overview of these activities will be presented, beginning with simple strain-gauge sensors (micronewtons), sensors based on tunneling displacement transducers (nanonewtons), AFM cantilevers (piconewtons), and ultra-thin force sensing cantilevers (attonewtons). Opportunities for exciting scientific measurements will be highlighted, and challenges for application of MEMS devices to these measurements will be discussed.

9:30 AM EE1.3
TEMPERATURE DEPENDENT INTERNAL FRICTION IN SILICON NANOELECTROMECHANICAL SYSTEMS.
S. Evoy, Dept. of Electrical Engineering, Virginia Tech, Blacksburg, VA; A. Olkhovets, J.M. Parpia, H.G. Craighead, Cornell Center for Materials Research, Cornell Univ, Ithaca, NY; D.W. Carr, Bell Laboratories, Lucent Techologies, Murray Hill, NJ.

The mechanical properties of micro- and nanomechanical systems are of interest from both fundamental and technological standpoints. High-frequency mechanical resonators presenting high quality factors are of interest for the development of sensitive force detecting devices, and highly efficient RF electromechanical filters and oscillators. Mesoscopic dimensions and high frequency operation also opens fascinating possibilities for sensitive studies of extrinsic processes such as surface and near-surface effects, and of other fundamental phenomena. Internal losses are the combination of both extrinsic and intrinsic issues that must be well understood for the optimization of resonator quality, and for the experimental access of such mesoscopic mechanical phenomena. Nanomachining opens new avenues for the assay of structures whose dimension compare to typical nanostructural length scales. We have recently reported the fabrication and electrostatic operation of nanomechanical beams as thin as 30 nm and frequencies as high as 380 MHz. We have also reported the dynamical modeling and characterization of paddle oscillators operating in the 1-10 MHz range. Here we report the temperature dependent behavior of these paddle oscillators. Reduction of temperature leads to an increase of the resonant frequencies of up to 6.5 %. Quality factors as high as 1000 and 2500 are observed at room temperature in metallized and non-metallized devices, respectively. Although device metallization increases the overall level of dissipation, internal friction peaks are observed in all devices in the T = 160-180 K range. The position of those peaks is consistent with the Debye relaxation of previously reported surface and near-surface phenomena. This is to our knowledge the first reported observation of a Debye relaxation peak in a megahertz range nanomechanical structure.

9:45 AM EE1.4
MEMS METROLOGY USING RESONANT STRUCTURES.
A. Miller Allen and George C. Johnson, University of California, Dept of Mechanical Engineering, Berkeley, CA.

A method for determining geometric process errors from the resonant frequencies of simple MEMS structures is presented. The ability to accurately determine the as-built geometry of MEMS devices is important given the magnitude of the geometric uncertainty relative to the dimensions of these devices. Consider a 2 μm square beam with 0.1 μm uncertainty in width or thickness. The relative uncertainty in both width and thickness is 5%, and the associated uncertainty in the beam stiffness is even larger - on the order of 15%. As MEMS become smaller, the need for reliable metrology becomes even more important. In an ideal process, the thickness and in-plane width of a MEMS structure would be exactly as specified in the design, and the side-walls of the structure would perpendicular to the plane of the film. Actual MEMS structures, of course, differ geometrically from the ideal. The method presented here provides a way to determine the actual film thickness, the etch offset (the difference between the design width of a planar feature and the as-built width) and the average angle of the side-walls. An important feature of the approach presented is that by using frequency ratios for various structural modes, neither the elastic modulus nor the mass density of the film need be known.

SESSION EE2: MECHANICAL PROPERTIES
Chair: Michael Judy
Monday Morning, November 27, 2000
Room 206 (Hynes)

10:30 AM *EE2.1
MICROSTRUCTURES-PROPERTY RELATIONS IN MEMS MATERIALS.
A.H. Heuer, Case Western Reserve University, Dept of MS&E, Cleveland, OH.

We have developed an on-chip micromachined Si micro-actuator capable of generating up to 700 micronewtons of force; the device can be used statically in a DC mode, or in resonance mode for fatigue studies (the resonant frequency is ~20 kHz). With this device, we have studied the mechanical properties of polysilicon and a host of other materials of interest in MEMS technology, including silicon dioxide, silicon nitride, and aluminum. The database generated by this device is essential for defining reliability limits for MEMS devices.

11:00 AM EE2.2
EFFECTS ON ROUGHNESS AND RESIDUAL STRESSES OF POST-PROCESSING AND STORAGE ENVIRONMENT OF MEMS MATERIALS.
Ioannis Chasiotis, Wolfgang G. Knauss, Graduate

Aeronautical Laboratories, California Institute of Technology, Pasadena, CA.

Experimentation and testing of thin films require a thorough understanding of the behavior of test structures and their long-term stability and integrity. Surface roughness described by groove formation at the grain boundaries or on individual grains is distinctly dependent on the HF release time and on residual stresses in the film. The post-processing HF release has also been identified as an important parameter in the measurement of film properties. The surface undulations can be the cause of reduced fracture strength or induce errors into elastic modulus measurements. Other phenomena are described, pertaining to wet release, which can be significant for the long-term integrity of MEMS structures.

11:15 AM EE2.3
MECHANISMS OF FATIGUE IN POLYSILICON MEMS STRUCTURES. Winston O. Soboyejo, Seyed. M. Allameh, Alexander Butterwick, Anthony. G. Evans, Princeton University, The Princeton Materials Institute and Department of Mechanical and Aerospace Engineering, Princeton, NJ; Brian Gally, Stuart Brown, Exponent Failure Analysis and Associates, Natick, MA.

This paper presents the results of an experimental study of fatigue crack nucleation oand propagation in silicon MEMS structures. Following an initial description of the initial surface topology and nano-scale microstructure of polysilicon, the micromechanisms of crack nucleation and propagation are elucidated via in-situ atomic force microscopy examination of cyclically actuated comb-drive structures fabricated from polysilicon. Mechanism-based models are then described for the prediction of surface topology evolution and environmentally-assisted crack nucleation. The implications of the results are discussed for the estimation of polysilicon MEMS reliability within a mechanistically-based or purely statistical framework.

11:30 AM EE2.4
FATIGUE OF THIN METAL FILMS INVESTIGATED BY DYNAMIC MICROBEAM DEFLECTION. R. Schwaiger and O. Kraft, Max-Planck-Institut für Metallforschung and Institut für Metallkunde, Universität Stuttgart, Stuttgart, GERMANY.

Fatigue is a threat to metallic components in many technological applications. However, the role of fatigue in the reliability of micro systems has not yet been explored in detail. This is probably due to the fact that most micro-electro-mechanical systems are based on Si in which fatigue is not expected to be a problem, as long as there are no initial cracks present. However, the use of metallic components in such systems can make them vulnerable to fatigue failure. In MEMS, metal films can be used for switches, valves or similar devices due to their magnetic properties or high electrical and thermal conductivity. In this work, we have investigated fatigue in thin sputter-deposited Cu and Ag films with thicknesses in the range of 0.3 to 1.5 m on micromachined silicon oxide cantilever beams. For fatigue testing, the beams were cyclically deflected for up to 4 million cycles with a frequency of typically 45 Hz using a nanoindentation system. During the fatigue tests the stiffness of the microbeams was continuously monitored and the occurrence of fatigue damage was detected as a significant decrease in stiffness. This decrease in stiffness results from the formation of fatigue damage, such as extrusions and cracks, in the film, which would lead to a severe degradation of its functional properties. Lifetimes of the films were determined as a function of straining conditions and film thickness. As expected, the fatigue life decreases with increasing mean strain or strain amplitude. Furthermore, it was observed that thinner films appear to be more fatigue resistant than thick films. The formation of extrusions is also less pronounced in the thinner films. The ultimate aim of these studies is to obtain a fundamental understanding of fatigue mechanisms in small volumes which is required for a thorough reliability assessment of metal-based MEMS.

11:45 AM EE2.5
FATIGUE EFFECTS IN THIN METAL PLATED CANTILEVERS FOR MEMS. Randall L. Kubena, Deborah J. Kirby, Frederic P. Stratton, Richard J. Joyce, David T. Chang and Jinsoo Kim, HRL Laboratories, Malibu, CA.

During the past several years, we have developed high displacement sensitivity tunneling accelerometers using surface micromachining and metal electroplating techniques. These devices consist of a Au tunneling tip fabricated below a 1 to 2-μm-thick metal cantilever beam of electroplated Au or Ni. A thin film of e-beam evaporated Au on the underside of the cantilever serves as a tunneling counter electrode. In operation, a 100-mV bias is applied across the tunneling gap. A larger turn-on voltage is also applied between the cantilever and a control electrode located on the substrate to deflect the cantilever and maintain a constant tunneling current of 1 or 10 nA. Typical deflections of the end of 100 and 250-μm-long cantilevers are 0.5 μm during operation. We have observed the turn-on voltage as a function of time for different cantilever materials, tunneling currents, cantilever pre-stress, cantilever lengths, and with and without a low temperature vacuum anneal. We have observed that the turn-on voltage decreases over time for most devices with a larger drop observed for the Au cantilevers. This temporal dependence of the turn-on voltage was found to be temperature dependent. In addition, in all cases, this initial decay of the turn-on voltage was almost completely recoverable after the device was turned off for 24 hrs. This decay was not found to be strongly dependent on the tunneling current, but could be significantly reduced by pre-stressing the cantilever before operation. Finally, a vacuum anneal at 100°C could influence the measured temperature dependence of the turn-on voltage. The observed effects appear to be consistent with fatigue and creep phenomena in the cantilevers. These effects are reversible at room temperature and are dependent on the stress and temperature history of the devices. We will present the device designs, the detailed measurements, and some of our initial conclusions in this paper.

SESSION EE3: TRIBOLOGY
Chair: S. Mark Spearing
Monday Afternoon, November 27, 2000
Room 206 (Hynes)

1:30 PM *EE3.1
SURFACE ENGINEERING OF POLYSILICON-BASED MEMS. Roya Maboudian, Dept. of Chemical Engineering, Berkeley, CA.

Given the dimension of most micro-electro-mechanical systems, gravity and other body forces are negligible. In contrast, interfacial forces dominate due to their large surface-to-volume ratios. As a consequence, adhesion, friction, and wear are prevalent problems in many MEMS devices. Additionally, MEMS technology provides us with the opportunity to study tribology in the mesoscopic length scale. This presentation will discuss the use of several tribological microinstruments in conjunction with other surface characterization techniques, such as X-ray photoelectron spectroscopy and atomic force microscopy, to measure the surface forces present between polycrystalline silicon surfaces and to manipulate them by utilizing various surface treatments. The successes and the limitations of current surface coating technologies as well as areas for improvement will be discussed

2:00 PM EE3.2
EFFECT OF NANOTEXTURING ON INTERFACIAL ADHESION IN MEMS. M.P. de Boer[1], J.A. Knapp[2] and A.C. Hall[3], Sandia National Laboratories. [1]Intelligent Micromachining Dept. [2]Radiation Solid-Interactions Dept. [3]Interface Reliability Dept.

Understanding the effects of surface roughness on parameters such as adhesion, friction and wear is a central question in the tribology of MEMS. In this work, we have fabricated polysilicon cantilevers over textured surfaces of varying nm scale roughness, and measured the interfacial adhesion of the cantilevers to the surfaces. A hydrophobic monolayer lubricant is applied to the surfaces to eliminate the effects of capillarity. Adhesion is measured by a well-developed fracture mechanics approach, in which the beam deflections are quantified, and the energy release rate is equated with the interfacial adhesion. Contrary to expectations, the effect of roughness, when increased over a large range from 3 to 12 nm rms, reduced the adhesion only from 8 to 5 microjoules per square meter. We believe this weak dependence is a fundamental result for nanotextured surfaces. Namely, real contact areas are exceedingly small, on the order of ten billionths of the apparent contact areas. However, because of the planar manufacturing techniques in MEMS, the average surface separation is also small. Therefore, van der Waals forces between non-contacting areas dominate the interfacial adhesion. The average separation does not depend strongly on surface roughness because plastic deformation of asperities tends to reduce the initial separation. To investigate this line of thinking, we have quantified the surface topography by atomic force microscopy. The results are fed into a finite element program that includes the effects of surface forces as well as material plasticity, and the surfaces are mated in the computer. We expect that this modeling will exhibit a length scale dependence on sample area, and at large sample areas, the dependence on roughness should converge. 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.

2:15 PM EE3.3
NOVEL CHEMISTRY FOR SURFACE ENGINEERING IN MEMS. Xiaoyang Zhu, Department of Chemistry, University of Minnesota, Minneapolis, MN.

It is well recognized that controlling surface forces is one of the key issues in the design, fabrication, and operation of micro-electromechanical systems (MEMS). Since the majority of MEMS devices are made of silicon from surface micromaching, an attractive approach has been the use of well-known alkly- trichlorosilane self-assembled monolayers (SAM) on oxidized silicon surfaces to control surface energy. While this approach has enjoyed some success in reducing adhesion in model MEMS structures, a major impediment to its implementation in a manufacturing setting is that it is highly irreproducible and very sensitive to a number of experimental parameters. In this report we present a novel strategy for the efficient assembly of organic monolayers directly onto the silicon surface via Si-O linkages. This is achieved by the reaction between an alcohol functional group and a chlorinated Si surface. The resulting molecular monolayers are thermally and chemically stable and are successfully demonstrated in adhesion reduction in a model MEMS structure, namely, a cantilever beam array (CMA). Polycrystalline beams with length up to 1.5 mm can be released. Major advantages of this new approach for surface control in MEMS include simplicity, reproducibility, and reliability.

2:30 PM EE3.4

THE FRICTION AND WEAR RESISTANCE OF TUNGSTEN COATINGS ON SILICON MICROMACHINED DEVICES RELATED TO THEIR SURFACE CHEMISTRY. M.T. Dugger, S. Mani, D.E. Peebles and G.A. Poulter, Sandia National Laboratories, Albuquerque, NM.

As processing technology for polysilicon surface micromachine devices evolves, design of actuators with mN-range force output and structures with high out-of-plane stiffness has become possible. These structures are much less susceptible to in-process adhesion during the release etch than their predecessors. However, in mechanical systems that rely upon the contact of moving surfaces to perform their function, a basic understanding of friction and wear is needed to predict initial performance as well as the performance as the devices age. To this end, we have investigated the relationship between surface chemistry, friction and wear for CVD tungsten coatings applied to polysilicon surface micromachine devices. The evolution of surface chemistry of the coatings from time of deposition to two weeks following deposition was examined using X-ray photoelectron spectroscopy. The tribological behavior of the coating was determined using a specially-constructed micromachined friction tester that permits quantification of friction forces between vertical sidewall surfaces. Results show that the initial composition of the tungsten coating consists of more than 40 atomic percent tungsten, but within a week of deposition the surface composition is dominated by adsorption of carbon and growth of oxide on the tungsten films. At this time, the coatings exhibit a friction coefficient less than 0.10 and no measurable wear after a million cycles of sliding contact in air. This paper will report on the relationship between composition and tribology of tungsten coatings for MEMS, and the mechanisms responsible for the observed friction and wear behavior. We will also discuss the effect of the oxygen and water vapor content in the operating environment on tribological behavior of these coatings.

2:45 PM EE3.5

ADHESION AND STATIC FRICTION MEASUREMENTS IN LIGA MEMS CONTACTS USING NOVEL NANOINDENTATION TECHNIQUES. Somuri V. Prasad, David A. LaVan, Todd R. Christenson and Mike T. Dugger Sandia National Laboratories, Albuquerque, NM.

LIGA, which combines synchrotron radition lithography, galvanoforming and plastic molding, is an emerging technology for producing net-shape components and structures for micro-electromechanical systems (MEMS). LIGA processed structures can be made of different materials such as metals (e.g., Ni and Cu), alloys and composites, deposited from suitable electrolytic baths. Like in other silicon based MEMS, adhesion plays a vital role in the performance of a LIGA processed MEMS device. The objective of our study was to characterize the adhesion and static friction between LIGA structures at length scales relevant to MEMS applications using novel nanoindentation techniques. Indentors of known radii of curvature, suitable for nanidentation experiments, were fabricated using the LIGA process. The Berkovich diamond indenter was replaced with LIGA fabricated metal tip, and the indentation experiments were performed on LIGA disks. The pull-off force (i.e. the negative load required to separate the LIGA tip from the flat surface after contact) was measured from the indentation experiments. The effects of applied load, surface topography, presence of oxide layers on the pull-force were evaluated. The setup was also used to perform static friction measurements at length scales relevant to LIGA MEMS.

SESSION EE4: ELECTRO-DEPOSITED METALS AND PZT
Chair: Hal Kahn
Monday Afternoon, November 27, 2000
Room 206 (Hynes)

3:30 PM EE4.1

ELASTIC AND INELASTIC PROPERTIES OF ELECTROPLATED NICKEL USED IN LIGA TECHNIQUE. Skandar Basrour, Laboratoire de Physique et Metrologie des Oscillateurs CNRS, Besanaon, FRANCE; Patrick Delobelle, Laboratoire de Mecanique Appliquee, Besanaon, FRANCE.

Pertinent simulations of Micro-Electro-Mechanical-Systems using powerful CAD tools requires a very precise characterization of the different materials involved in the fabrication processes. The development of new metallic microdevices fabricated by LIGA technique depends strongly on the mechanical characterization of the electroplated coatings. The aim of this paper is a wide investigation at the microscale of the elastic and inelastic properties of electroplated Nickel which is extensively used in the LIGA. The first part of this paper presents the experimental results obtained with a nano-indentation set-up. From conventional curves, load versus penetration depth $P(h)$, we have obtained the Berkovitch's Hardness H and the Young's Modulus E. These mechanical properties are affected by the current densities used during the electroplating as well as the nature of the substrates (Bulk Copper or Silicon wafers). The rms values of the roughness R_a are obtanied thanks to confocal microscopy The mean grain diameters Φ are deduced by SEM observations. These results are also tightly linked to the deposition parameters. We have found a linear behavior of H versus $\frac{1}{\sqrt{R_a}}$ or $\frac{1}{\sqrt{Phi}}$ leading to an Hall and Petch relation. An inverse analysis using a Finite Element Method (FEM) of the $P(h)$ curves, leads to the yield stress Y_0 and the strain hardening parameters in particular the ultimate strength Q. These plastic parameters are also sensitive to the deposition conditions. We have found a linear relation between Y_0Q and the hardness H of the coatings. The relation Y_0Q versus $\frac{1}{\sqrt{R_a}}$ seems to be independent from the deposition conditions and the nature of the substrates. The second part of this paper is devoted to the mechanical characterization of Nickel microbeams (clamped-free or clamped-clamped) obtained by LIGA technique. The experimental results deduced from the 2 and 4 points bending tests have been analyzed by a FEM. The data deduced from this technique (E, Y_0Q) are in good agreement with our results obtained by nano-indentation and also with previous results reported in the literature.

3:45 PM EE4.2

STRUCTURAL AND MICROMECHANICAL ASSESMENT OF ELECTROCHEMICALLY GROWN METAL LAYERS FOR Si MAGNETIC MICROACTUATORS. S. Martinez, N. Yaakoubi, A. Perez-Rodriguez, C. Serre, J.R. Morante, EME, Dept of Electronics, University of Barcelona, Barcelona, SPAIN; P. Gorostiza, SCT, University of Barcelona, Barcelona, SPAIN; J. Esteve, CNM-CSIC, Campus UAB, Bellaterra, SPAIN.

Electrochemical (EC) deposition constitutes one of the most interesting technologies for growing of metal layers for MEMS devices. EC has important advantages over alternative techniques. These are related to the higher rate of deposition, which allows the growth of thicker layers, the easy control of the thickness and composition of the layer, the possibility to process complex surfaces with non planar morphology and the low cost of the experimental set-up. Besides, EC is fully compatible with standard Si micromachining technology. The ability to selectively deposit metal layers on the window regions from a mask layout allows for the post processing of CMOS processed wafers. This work reports the EC deposition and microstructural assessment of metal layers for Si magnetic microactuators. These devices are based on the integration of a metallic coil onto micromachined Si wafers. Cu is very suited for these applications, because of its low resistivity, low cost and ease to grow electrochemically. The EC deposition of Cu wafers has been investigated in two kind of wafers: i) direct deposition onto the Si surface and ii) dielectric sacrificial layers (mainly SiO_2) on Si. In the first case, a two step process has been designed to solve problems related to the film adherence to the surface. On the other hand, EC deposition onto dielectric layers needs for the previous growth of a thin seed film. The structural analysis of the layers (residual stress, adherence, polycrystalline structure) has been performed by XRD, Electron Microscopy and AFM measurements. This has been completed by the design and fabrication of simple test structures, which involves selective deposition of metal layers on surfaces patterned with suitable photo resin layers. The deposition of multilayer Cu/Ni structures is also investigated, due to the interest in the use of the beneath Ni films as sacrificial layer.

4:00 PM EE4.3
MEMS ACCELEROMETERS USING LEAD ZIRCONATE TITANATE PIEZOELECTRIC FILMS. Richard Wolf[1], S. Trolier-McKinstry[1], Y. Wang[1], L.-P. Wang[1], R.J. Davis[1], K. Chandra[2], K. Deng[2], W. Shanks[2], T. Brooks[2]. [1]The Pennsylvania State University. [2]Wilcoxon Research, Inc. Gaithersburg, MD.

This paper reports on the design, fabrication, and properties of a MEMS (micro-electro-mechanical systems) accelerometer using lead zirconate titanate (PZT) 52/48 films as the active sensing element. The design incorporates 1 - 8 μm thick, electroded PZT film on a $Pt/Ti/SiO_2/Si$ cantilever structure with a large Si proof mass. The sol-gel PZT films show good dielectric and piezoelectric properties, with P_r values > 20 $\mu C/cm^2$, $\epsilon_r > 800$, tan$\delta < 3\%$, and —d_{31}— values > 40 $\mu C/cm^2$. Over a 4" diameter wafer, the thickness was quite uniform, with a standard deviation of ~2% from the average value. This, in turn, led to a 2 - 3% variation in the electrical properties, with a maximum variability of 5%, probably as a consequence of the thickness variation. The proof mass fabrication, as well as the cantilever release step, were accomplished via deep reactive ion etching of the Si substrate. It was found that for an accelerometer with a footprint of 1 cm^2, it was possible to achieve a charge sensitivity as high as 1.04 pC/g. Work is currently ongoing to optimize the output of the sensor, as well as to characterize the temperature dependence of the response.
This work is sponsored by a NIST ATP program.

4:15 PM EE4.4
DEFECTS AND FAILURE MODES IN PZT FILMS FOR A MEMS MICROENGINE. D.F. Bahr, B.T. Crozier, C.D. Richards and R.F. Richards, Mechanical and Materials Engineering, Washington State University, Pullman, WA.

Piezoelectric films for a MEMS microengine have been deposited using solution deposition routines onto platinized silicon wafers. These films are used as membranes above a bulk micromachined cavity. A dynamic bulge tester and interferometer are used to characterize the deformation of the films when pressurized. The mechanical stress at failure, as well as the fatigue behavior, have been characterized. Membranes between 1 and 4 microns thick have been shown to sustain over 350 million cycles at stress levels between 70 and 90% of the mean failure stress. Defects in the films due to abnormal precipitate growth and thermal stresses during processing, and their role in membrane failure, are identified. The effect of these defects on the electrical behavior of the films is also discussed. The results of the bulge test failures are compared to indentation induced failures and thermally induced failures which generate interfacial failure between the metalization layers and the underlying silicon oxide.

4:30 PM EE4.5
EXCIMER LASER LIFTOFF: AN ALTERNATIVE INTEGRATION SCHEME FOR FABRICATING $Pb(Zr,Ti)O_3$-BASED MEMS. Loucas Tsakalakos, Alberto Salleo, William S. Wong* and Tim Sands, Univ of California, Dept of Materials Science & Engineering, Berkeley, CA; Pamela Caton and Richard M. White, Univ of California, Berkeley Sensor & Actuator Center and Dept of Electrical Engineering & Computer Science, Berkeley, CA. *now at Xerox Palo Alto Research Center, Palo Alto, CA.

In recent years a wide variety of piezoelectric MEMS sensors and actuators have been demonstrated. Initial devices utilized ZnO thin films as the active material, a material with relatively small piezoelectric and electromechanical coupling coefficients. However, as thin film deposition approaches for $Pb(Zr,Ti)O_3$ (PZT) were developed and optimized, this superior material was also incorporated in MEMS devices. Typical conditions for growing PZT thin films are 600°C in a high pressure (100-300 mTorr) oxygen environment, making direct integration with fully processed Si or polymer substrates impossible, and integration with unprocessed Si or metal substrates very difficult or impractical. Recently we have developed an alternative approach to direct deposition of PZT films, Excimer Laser Liftoff, a process originally developed for GaN films. This is a "paste and cut" methodology in which an epitaxial or polycrystalline PZT film is grown on a UV-transparent single crystal substrate (e.g. MgO, sapphire, or quartz), bonded to a receptor substrate, and then separated from the original growth substrate by application of a single excimer laser pulse through the backside of the growth substrate. Such an approach allows for integration of oxide films with virtually any substrate. We have demonstrated that the process does not compromise the ferroelectric (and therefore piezoelectric) properties of PZT films transferred by Laser Liftoff. In this talk the materials and processing issues involved in fabricating MEMS devices are discussed in the context of fabrication of Flexural Plate Wave (FPW) sensors (on Si wafers) and cantilever beam actuators (on 25 μm thick stainless steel substrates), two devices fabricated in our laboratory utilizing Laser Liftoff. Topics to be discussed include bonding uniformity and the effect of laser fluence. The role of laser-induced acoustic waves on the success of substrate removal, and on the film properties has also been found to be important, and will thus be described in detail.

4:45 PM EE4.6
PULSED DIELECTRIC AND ULTRASOUND SPECTROSCOPY OF SHEAR MODE PZT MICROACTUATOR. Juergen Bruenahl, Alex Grishin and Sergey Khartsev, Dept of Condensed Matter Physics, Royal Institute of Technology, Stockholm, SWEDEN.

Ferroelectric lead zirconate titanite (PZT) is the main industrial product in piezoelectric ceramic materials used for sensor and actuator applications. High precision in manufacturing of microelectromechanical systems is essential to get demanded performance accuracy. An adequate and preferably non-destructive measurement technique is desired to characterize the materials properties at various stages of device fabrication as well as to find correlation between the processing parameters, microstructure and functional properties. We report on new pulsed technique which has been employed to characterize all relevant properties of special machined array of PZT channels. The proposed technique measures the following mechanical and electrical properties fast, reliable and reproducible: dielectric constant, loss factor (tan delta), electro-mechanical coupling coefficient, mechanical displacement, resonance frequency, mechanical quality factor (Qm) as well as temperature dependence of these parameters. Dependence on various aspect ratios between width, height, and length of elements of the array exemplifies the versatility of proposed technique.

SESSION EE5: POSTER SESSION
Chairs: Maarten P. de Boer, Michael Judy, Hal Kahn and S. Mark Spearing
Monday Evening, November 27, 2000
8:00 PM
Exhibition Hall D (Hynes)

EE5.1
MULTILAYER MATERIALS FOR ELECTROSTATIC SWITCHES. Mark A. Phillips, Brennan L. Peterson, Christine M. Esber, Jesse Hwang, Bruce M. Clemens, Stanford University, Dept of MS&E, Stanford, CA.

Metal multilayers are a unique way to manufacture reliable conductive members for MEMS devices. These members are particularly suited for electrostatic switches. Unlike elemental Al, which experiences curvature problems during processing, the stress in these beams can be controlled and calibrated during *in-situ* stress measurements to have repeatable shapes and stresses upon release. For example in this study, electrostatic beams with zero average stress and zero bending moment were designed by alternating tensile and compressive layers within an electrostatic switch. Moreover, these multilayer beams have low contact resistances compared to present materials like poly-Si. Resistivity measurements were also done to determine the optimal composition and the interface behavior of the multilayer for a low contact resistance. The mechanical and electrical behaviors of metallic multilayers are investigated in order to develop reliable electrostatic switches for MEMS devices.

EE5.2
ELECTRIC FIELD TRAPPING OF BIOLOGICAL CELLS INTO MICROMACHINED CAVITIES. Cengiz S. Ozkan, Mihrimah Ozkan, Erhan Ata, Sadik C. Esener, Electrical and Computer Engineering Department, University of California at San Diego, San Diego, CA.

This paper describes a novel technique for trapping live biological cells into micromachined cavities or channels on flexible and transparent polymer substrates. The substrates are fabricated by spin coating suitable polymer layers that are 100-150 μm thick on Silicon wafers. After properly baked, a 50 nm thick Aluminum layer is evaporation coated on top which is patterned via conventional contact lithography and wet etching. The thin metallization layer serves as a hard etch mask for patterning the underlying polymer layer in an RF plasma etching system. Live cells are introduced into the micro-machined structures in a media solution. The cells can be trapped onto the surface of the cavities using an electrical field where appropriate electrodes are placed or fabricated. Since cells are negatively charged due to sialic acid residues in their glycocalyx, they tend to move towards the positively charged electrode. We describe possible applications where the trapped cells can be made to react with various chemicals in the cavities for include biological warfare studies, drug studies and cell-protein interactions. Other possibilities of this technology include 2-D and 3-D patterning of cell arrays for tissue engineering. Transparent substrates will enable optical microscopy, interference or spectroscopic studies of the trapped cells and reacted media.

EE5.3
RELAXATION OF MEMBRANE STRESS USING ALUMINUM OXIDE AND SILICON DIOXIDE MULTI LAYER STRUCTURE.
Ryuichi Kubo, Yukio Yoshino, Kazuhiro Inoue, Takahiro Makino, Seiichi Arai; R&D Division, Murata Mfg. Co., Ltd, Kyoto, JAPAN.

Relaxation of internal stress for micro-electro-mechanical systems (MEMS) using multilayer thin films which are made of Al_2O_3 and SiO_2 has been studied. The relaxation of internal stress is necessary for MEMS devices, such as membrane devices, because the stress causes destruction or bending of its structure. In this experiment aluminum oxide thin films that have tensile stress and silicon dioxide thin films that have compressive stress were combined to make a low stress membrane. The aluminum oxide thin films were prepared by electron beam evaporation at room temperature, and the deposition rate was about 50nm/min. No peaks were observed in the X-ray diffraction pattern of the films. The composition ratio of Al and O kept the same ratio of Al_2O_3 target material. The internal tensile stress was about 300-400 MPa. The silicon dioxide thin films were prepared by thermal oxidation and RF magnetron sputtering method. The internal stress of thermal oxide films was about -440 MPa and that of the films deposited by sputtering was about -100 MPa. Those silicon dioxide thin films and aluminum oxide thin films were prepared in multilayer on Si(100) substrate, which was then backside etched to form a membrane structure of 1.6 mm square. The thickness of each film was controlled for relaxing the total stress of the membrane. The total stress of the membrane was almost 0 in optimum deposition condition of SiO_2 and Al_2O_3 films.

EE5.4
STRESSES AND DEFORMATION IN MINIATURE STRUCTURES AND MICRO-ELECTRO-MECHANICAL SYSTEMS (MEMS).
T.A. Venkatesh and S. Suresh, Dept. of MS&E, Cambridge, MA.

The optimization of electro-mechanical, thermal, optical, fluidic or magnetic functionality of MEMS devices requires a comprehensive understanding of a variety of materials and mechanics issues, particularly those dealing with the prediction, measurement and control of processing-induced and service-induced stresses as well as mechanical performance. A comprehensive analysis of stresses and deformation in small-volume structures requires a full consideration of several factors such as geometry, combination of materials used and the processing sequence adopted in fabricating these structures, and the chemical/thermo-mechanical environmental interactions that these structures are expected to encounter while in service, all within the context of length-scale dependence of fundamental elasto-plastic mechanical properties. Thus, a prediction of the overall mechanical response of a particular structure would typically mandate a tracking of all aspects of a given structure through fabrication and service. Hence, an alternate approach is presented here, whereby small-volume structures are classified based on their geometry, structural environment, and coupling characteristics, and a general framework for the analyzes of the mechanical response of such structures is constructed and analyzed. In parallel with this classification, a broad and complementary classification of experimental techniques that enables the extraction of the fundamental properties of materials is also developed. Key results of the various methods for (elastic, plastic, fracture and fatigue) property extraction are reviewed and the advantages and limitations of each method are analyzed. General trends in material properties at small size scales are discussed in the light of available results as well. Particular examples are also presented of situations where such approaches provide powerful methods for the study of mechanical response in miniature structures.

EE5.5
YOUNG'S MODULUS AND STRENGTH OF THREE POLY-SILICONS.
W.N. Sharpe, Jr., K. Jackson, G. Coles, Johns Hopkins University, Dept of Mechanical Engineering, Baltimore, MD; D.A. LaVan, Sandia National Laboratories, Albuquerque, NM; R. Mali, Standard MEMS, Inc. Hauppauge, NY.

Structural materials of a certain class, e.g. aluminum, have essentially the same Young's modulus, but different strengths depending upon manufacturing processes. One wonders if the same is true for materials used in MEMS. The authors have developed tensile test techniques and procedures for measuring Young's modulus and strength of polysilicon and have used the same test apparatus to test materials from three different sources. The tensile specimens were deposited onto silicon wafers; one end of each specimen is fixed to the wafer and its test section along with a one-millimeter square grip end is released by etching away an underlying sacrificial layer. An electrostatic probe is attached to the grip end, and the specimen is pulled by an electrostatic translation stage. Force is measured with a 100-gram load cell, and overall system displacement is measured with a capacitance probe. Young's modulus is extracted from the force-displacement record using a finite element model of the specimen and the measured stiffness of the gripping and loading system. Polysilicon from Cronos, Sandia, and Standard MEMS, Inc. (SMI) were tested, with the latter supplying two sets of wafers that had experienced different anneals. The specimens were either nominally 6 or 20 mm wide and either 250 or 1000 mm long. The Cronos and the Standard MEMS specimens had thicknesses of 1.5, 2.0, and 3.5 mm; whereas, the Sandia specimens were all 2.5 mm thick. There was no statistically significant difference in Young's modulus for the three materials. A total of 117 tests were run, and 45 tests of the Cronos material yielded a value of 158 /- 8 GPa. However, the strengths were quite different - 47 Cronos tests at 1.56 /- 0.25 GPa, 10 Sandia tests at 3.09 /- 0.18 GPa, 20 tests of one SMI type at 2.08 /- 0.35 GPa, and 24 tests of the other SMI type at 2.00 GPa /- 0.25 GPa. These results will be presented in detail along with a description of the test methods. Continuing research to understand the differences in strengths will be presented if time permits. This is the first direct comparison of polysilicon from different suppliers, and the fact that the strengths can differ by a factor of two is important. This research was conducted under the sponsorship of DARPA F30602-99-2-0553.

EE5.6
FRACTURE TOUGHNESS, STRENGTH AND WEAR TESTING OF SURFACE MICROMACHINED MEMS.
David A. LaVan, Kamilli Jackson, M.T. Dugger, M.P. de Boer, John P. Sullivan, Thomas A. Friedmann, Sandia National Laboratories, Albuquerque, NM.

Simple, quick and accurate methods to measure and qualify material properties are necessary for surface micromachining to be accepted as a standard manufacturing technique for critical components. The results from ongoing large-sample studies of polysilicon and amorphous Diamond (a-D) strength testing will be presented. Fracture toughness is being investigated using two techniques: FIB notched tension samples, and an electrostatically actuated device capable of applying cyclical loading (up to 2 GPa principal stress at a notch root) followed by a final high force loading cycle. Results for polysilicon and a-D will be presented for the notched samples, and for polysilicon only using the 5-level fracture toughness measurement device. New devices to directly probe strength using electrostatic actuation and interferometric microscopy will also be shown. Wear and adhesion limits the life of many devices. A device proven in wear studies of polysilicon has been modified for the a-D process; results for the wear of a-D will be shown.

EE5.7
MICROSTRUCTURE, STRENGTH AND PIN-ON-DISC WEAR TESTING OF LIGA MEMS.
Thomas E. Buchheit, David A. LaVan, Joseph R. Michael, Aaron Hall, M.T. Dugger, Somuri V. Prasad, Sandia National Laboratories, Albuquerque, NM.

LIGA manufactured MEMS devices are being rapidly adopted as their length scale is close to conventional miniature devices. However, few miniature components, manufactured using conventional techniques, are made from the pure metals or alloys used in LIGA devices. The behavior of LIGA materials is not expected to be the same as other engineering alloys. This work will summarize a large study completed on the process-microstructure-properties of LIGA Nickel, as well as introduce new results for Nickel alloys and Nickel nanocomposites. These new materials offer the promise of more stable properties over extended temperatures and more favorable tribological behavior. They have been characterized using a suite of analytical tools including nanoindentation, pin-on-disc wear testing, AFM, miniature tensile testing, metallography, and EBKP imaging of orientation.

EE5.8
BEHAVIOR OF MICRON-DIMENSION SILICON-GERMANIUM STRUCTURAL FILMS UNDER CYCLIC LOADING.
Christopher Muhlstein, University of California, Department of Materials Science and Engineering, Berkeley, CA ; Andrea Franke, Roger Howe, University of California, Department of Electrical Engineering and Computer Sciences, Berkeley, CA; Robert O. Ritchie, University of California, Department of Materials Science and Engineering, Berkeley, CA.

Unlike their macroscopic counterparts, microelectromechanical systems (MEMS) have seen the immediate application of brittle structural materials in safety-critical applications, such as airbag accelerometers and inertial guidance systems. In spite of the risks, the use of damage-intolerant structural films such as silicon will persist because of the large base of processing expertise and equipment, as well as the ease of integrating electronics. However, due to the real potential for premature failure, the long-term use of brittle structural films such as silicon mandates the investigation of subcritical crack growth phenomena such as cyclic fatigue and stress corrosion cracking. The ongoing work of the authors (Muhlstein and Ritchie) have established that time-delayed failures can occur in silicon-based films in under 1×10^{12} cycles at approximately one half the single cycle strength of the material. Recent advances in SiGe-based surface micromachining at the Berkeley Sensors and Actuators Center (BSAC) have prompted this investigation of the behavior of SiGe structural films under cyclic loading conditions. Intermediate compositions of p-type SiGe-based films have been successfully surface micromachined with integrated CMOS electronics (Franke and Howe). This recently developed processing technology was used to manufacture the electrostatically actuated, notched cantilever beam fatigue testing structure previously developed at Exponent, Inc. Initial testing indicates that the SiGe structural films have comparable strength to polycrystalline silicon films. However, the susceptibility of SiGe films to time-delayed failure under cyclic loading conditions has not been previously characterized. The stress-life behavior of the SiGe films in ambient environments will be presented in conjunction with a detailed characterization of fracture surface morphology using the scanning electron and atomic force microscopy (SEM and AFM). This investigation provides the first comparison of the behavior of SiGe to existing polysilicon films under cyclic loading conditions and is a critical milestone in the introduction of novel structural film material systems.

EE5.9
FINITE ELEMENT ANALYSIS OF THE PRECRACKED LINE SCRATCH TEST.
L.L. Mercado[1], A.A. Volinsky[1], V. Sarihan[1], W.W. Gerberich[2]. [1]Motorola Inc., Interconnect Systems Laboratories, Tempe, AZ. [2]University of Minnesota, Dept. of Chem. Engineering and Material Science, Minneapolis, MN.

In MEMS packages and devices, the interfacial adhesion is a critical reliability issue. A Precracked Line Scratch Test (PLST) is among one of the available tests to measure the thin line adhesion. In the test, an initial crack is introduced at the interface between the thin line and the substrate. The line is then loaded from the precracked end. The load is recorded continuously while the crack propagates before and after the line buckles. This precracked line scratch test has been applied earlier to the tungsten thin lines on silicon wafers [1]. Macroscopic version of the test was also performed to evaluate the analytical model [2]. In the macroscopic tests, polycarbonate lines are bonded to steel substrate with cyanoacrylate. In this study, finite element analysis is performed for the Precracked Line Scratch Test prior to the line buckling. The energy release rate and phase angle are calculated based on the corresponding load and crack length. The results are then compared to the closed-form solution. Both microscopic and macroscopic tests are evaluated. Finite element solution has provided a way to determine the interface fracture toughness as a function of the phase angle based on the load and crack length history. With the analysis in place, the precracked line scratch test can be used conveniently to study the adhesion of MEMS devices and packages on different scales.
1. M.P. de Boer, M. Kriese, W.W. Gerberich, J. Mater. Res. 12(10), 1997, 2673-2685.
2. A.A. Volinsky, J.C. Nelson, W.W. Gerberich, Mat. Res. Soc. Symp. Proc. Vol. 563, 1999.

EE5.10
DEFORMATION MECHANISMS OF A MICRO-SIZED AUSTENITIC STAINLESS STEEL WITH FINE GRAINS.
Guangping Zhang, Kazuki Takashima, Masayuki Shimojo, Yakichi Higo, Tokyo Institute of Technology, P&I Laboratory, Yokohama, JAPAN.

With the rapid growth of microelectromechanical systems (MEMS) with a high volume production and low unit cost, the demand for the understanding of the mechanical performance in micro-sized materials becomes more and more urgent in order to design highly reliable micro-sized machines. Previous studies on the mechanical properties of micro-sized materials mainly focused on the measurements of Young modulus and fracture strength. However, the basic deformation mechanisms of micro-sized materials, especially for the fine-grained materials widely applied to MEMS in the form of thin films, are still less well understood up till now. In this study, micro-cantilever beam type specimens with dimensions of 30 x 25 x 65 μm^3 were fabricated from austenitic stainless steel sheet with 1 μm grain size by focused ion beam machining. The deformation behavior of such micro-sized specimens was studied by the static bending tests. The results showed that the micro-cantilever beam type austenitic stainless steel is of two-stage deformation processes, namely rapid work-hardening of the stage I and subsequently slow work-hardening of the stage II. At the stage I, the deformation behavior is characterized by the extensive intragranular dislocation slips and their interaction with grain boundaries which led to the deformation incompatibility near grain boundaries and the appearance of the roughness on the specimen surface. With increasing in plastic stain, grain boundary sliding (GBS) and subsequently intergranular cracking occurred at the stage II. The differences in stress conditions and work-hardening behaviors on the front tension-side and rear compression-side surfaces of the micro-cantilever beam resulted in the different deformation behavior.

EE5.11
CORROSION FATIGUE TESTS OF MICRO-SIZED SPECIMENS.
Yuri Mizutani, Yakichi Higo, Yusuke Ichikawa, Atsushi Morita, Kazuki Takashima, Tokyo Institute of Technology, P&I Lab., Yokohama, JAPAN.

Fatigue properties and long-term reliability of micro-sized materials are extremely important to design actual MEMS devices. In particular, the evaluation of fatigue properties of micro-sized components in corrosive environments are essential in micro-biomedical devices, as the surface effect on the corrosion is more prominent than ordinary-sized components. However, there have been few studies to date which investigate corrosion fatigue properties of micro-sized materials, since there is no adequate corrosion fatigue testing equipment for micro sized materials. In this investigation, a corrosion fatigue testing machine for micro-sized materials has been developed. This testing machine was capable of applying very small cyclic load to micro-sized specimens with a load resolution of 10 μN and a displacement resolution of 5 nm, respectively. The specimen stage and solution cell is set on a precise X-Y stage,which can be moved to adjust the loading position by a stepping motor at a translation resolution of 0.1 μm. Fatigue tests were carried out both in air and in 0.9% NaCl solution. Cantilever beam type specimens with dimensions of 10 x 12 x 50 μm^3 were prepared from an Ni-P amorphous thin film and a type 304 austenitic stainless steel foil by focused ion beam machining. Small cyclic load is able to be applied to the specimens in the solution. Fatigue lives in both the specimens in corrosive environment was lower than that of in air. The effect of environment and that of specimen size on fatigue properties for micro-sized specimens are discussed.

EE5.12
ANISOTROPIC FRACTURE BEHAVIOR OF ELECTROLESS DEPOSITED Ni-P AMORPHOUS ALLOY THIN FILMS.
Kazuki Takashima, Akio Ogura, Yusuke Ichikawa, Masayuki Shimojo, Yakichi Higo, Tokyo Institute of Technology, P&I Laboratory, Yokohama, JAPAN.

Thin films deposited on substrates are expected to be applied to micro-electro-mechanical systems (MEMS) including biomedical and micro-photonics devices. In such devices, the film layer is machined into micro-sized elements which are used as mechanical components. Therefore, the fracture behavior of thin films on substrates is extremely important to ensure the reliability of such MEMS devices. Thin films deposited on substrates have been considered to have an anisotropy even if they are amorphous films, and the fracture behavior of in-plane direction (perpendicular to the deposition growth direction) is anticipated to be different from that of out-of-plane direction (parallel to the deposition growth direction). However, such anisotropic fracture behavior of deposited films has not yet been clarified because of the difficulty in the testing method. In this present study, fracture toughness tests have been performed for electroless deposited Ni-P amorphous alloy thin films with different crack growth directions, which are perpendicular and parallel to the deposition growth direction. Cantilever beam type specimens with dimensions of 10 x 10 x 50 μm^3 were prepared from an Ni-P amorphous thin film and notches with different direction were introduced by focused ion beam machining. Fatigue precracks were introduced ahead of the notches. Fracture tests were performed using a newly developed mechanical testing machine for micro-sized specimens. Fracture behavior is different between the two types of specimens. Although K_{IC} values were not obtained as the criteria of plane strain requirements were not satisfied for this size of the specimen, the provisional fracture toughness K_Q value of the specimen with crack growth being parallel to the deposition growth direction was 4.2 $MPam^{1/2}$ and that with crack growth being perpendicular to the deposition growth direction was 7.3 $MPam^{1/2}$, respectively. These results suggest that the electroless deposited amorphous alloy films have an anisotropy.

EE5.13
TEMPERATURE AND DOPING DEPENDENCY OF PIEZO-RESISTANCE IN P-TYPE SILICON.
Eivind Lund, Terje Finstad, Univ of Oslo, Dept of Physics, Oslo, NORWAY.

We have performed new measurements of the temperature and doping dependency of the piezoresistance effect in p-type silicon. Piezoresistance is the change of resistance due to an applied mechanical stress. The effect is well known in silicon and is one of the most common sensing principles of micro-electro-mechanical-systems (MEMS).
Our measurements are performed in a specially designed 4 point bending setup. The samples are 6 x 40 mm beams of full wafer thickness. To minimize leakage currents and to obtain uniform doping profiles we have used SIMOX (Separation by Implantation of Oxygen) substrate with a 5μm epitaxial layer. Spreading resistance measurements verify the uniform doping profile, while measurement of

zero stress resistance versus temperature verify low leakage current. Some of our samples even have resistors that are further isolated by dry etched recesses through the top silicon layer.
In this paper we present results for the doping range from $1E17$ Cm^{-3} to $1E20\ \mathrm{cm}^{-3}$ and the temperature range from -30 to 150 degrees Celsius. The results show a doping dependency well described by the analytical expression

$$\frac{F'_{s+(1/2)}(E_F(N)/\kappa_B T)}{F_{s+(1/2)}(E_F(N/\kappa_B T)}$$

Here F is the Fermi integral, F' its derivative with respect to E_F/k_BT, $E_F(N)$ is the Fermi energy at the doping level N, k_B is the Boltzmann's constant and T is the temperature. The temperature dependency of the piezoresistance coefficients fits a decreasing linear model where the absolute value of the temperature coefficient decreases with increasing doping.
We formulate a possible explanation to the deviation from current analytical models based on the temperature dependent change of the effective masses with applied stress. Previously published measurements of the effective mass show clearly how the effective mass changes under applied mechanical stress. The fractional change in effective mass is assumed to be strongly temperature dependent.
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EE5.14
CURVATURE MODEL FOR AN ION-MACHINED FREE-STANDING THIN FILM MEMS DEVICE. H.T. Johnson, Boston University, Dept of Aerospace & Mechanical Engineering, Boston, MA; P. Bierden, Boston Micromachines Corp., Boston, MA; T.G. Bifano, Boston University, Dept of Manufacturing Engineering, Boston, MA.

Free-standing thin film MEMS devices for optical applications often require nearly perfect planarity. However, numerous processing steps induce stress in a thin film structure, and release of the structure from a host substrate permits curvature as a stress relaxation mechanism. The mechanisms by which processing steps affect the microstructure of the film, and their connection to the resulting curvature of the free-standing structure are of great practical interest in the fabrication of novel MEMS devices. Here, the connection between process-induced stress and overall film curvature in a free-standing thin film MEMS micro-mirror device is studied theoretically. Various processes, including solid source diffusion doping and chemically neutral ion-beam machining are considered as sources of elastic mismatch strain in a thin poly-Si layer. Then, using a thin film mechanics model developed elsewhere, the resulting curvature of the film upon release from the host substrate is related to the through-thickness mismatch strain distribution. The ion-beam machining step affects the resulting curvature of a free-standing film both because of the effects of the ion bombardment in a layer near the surface of the film, and due to the effect of removal of stressed material upon continued exposure to the beam. The process is shown to be a promising tool for deliberately altering the curvature of a free-standing thin film structure. Comparisons of calculated curvature values to available experimental data show close agreement.

EE5.15
MECHANICAL BEHAVIOR OF MICROSPRING ARRAYS. M.W. Seto, M.J. Brett, Univ of Alberta, Dept of Electrical and Computer Engineering, Edmonton, Alberta, CANADA.

Porous thin films with helical microstructures have been fabricated with the Glancing Angle Deposition technique. These films consist of arrays of "microsprings" whose geometries can be engineered with nanometer scale control. Some of the mechanical properties of these helically structured films have been studied with a nanoindentation technique. Using a selection of spherical and flat punch indenter tips, several microscopic "springbed" films were tested over a range of forces. The geometries of the microsprings were varied, so that the helical pitch ranged from approximately 600 nm to 1300 nm for springs having 1 to 3 turns, and a number of different materials were used to fabricate these films, which were typically a few micrometers thick. A distinctive shape anisotropy also exists within these films, where the thickness of the microspring columns increases towards the top of the films. This effect of column broadening was taken into account in the simulations that were performed to model these films. Results of initial experiments, theory and simulations show that these microsprings behave in a manner analogous to macroscopic springs, and may offer some insight into how materials behave at the microscale. Devices incorporating these unique thin films can include structures such as physical resonators, and for MEMS applications, require study of elastic film properties and resonant frequencies.

EE5.16
MULTISCALE MODELING OF DISSIPATIVE PROCESSES IN MEMS RESONATORS. Robert E. Rudd, Lawrence Livermore National Lab, Livermore, CA; Jeremy Q. Broughton, Yale School of Management, New Haven, CT.

This work studies multiscale phenomena in silicon and quartz micro-resonators comprising the mechanical components of sub-micron Micro-Electro-Mechanical Systems (MEMS). The behavior of these next-generation MEMS is determined to a large extent by the interplay between physics at the Angstrom, nanometer and micron scales. Atomistic processes cause systematic deviations from the behavior predicted by conventional elastic theory, and lead to unconventional mechanisms for dissipation. The simulation of these effects is a challenging problem since they are coupled to long-range strain fields so that a large number of atoms are involved. We use molecular dynamics (MD) to simulate the most critical part of the devices, consisting of a few million atoms. Hundreds of millions more are in the proximal regions of the substrate, but here the strain gradients are much smaller, and a more efficient description suffices. So we couple MD to finite elements, or a nanoscale generalization we have developed known as Coarse-Grained Molecular Dynamics (CGMD). In this way we capture the relevant physical effects at two length scales simultaneously: the nanoscale atomistic processes in the crucial components of the device, and the micron-scale elastic fields extending out into the substrate to which they are inherently coupled. In this talk we present simulations of the vibrational behavior of micron-scale oscillators, and we focus on unconventional dissipation mechanisms at the nanoscale.
Acknowledgements:
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EE5.17
PHASE TRANSFORMATIONS IN FERROMAGNETIC NiMnGa SHAPE MEMORY FILMS. Manfred Wuttig, Corneliu Craciunescu, Jian Li, Univ. of Maryland, Dept. of Materials Science and Engineering, College Park, MD.

The paper presents recent work on ferromagnetic NiMnGa films sputter deposited on silicon cantilever substrates. The films are partially crystalline if the substrate is held at room temperature (RT) during the deposition. They become fully crystalline after annealing at 400°C. The internal friction (IF) of the RT deposition shows a peak that is attributed to the nanostructure of the film. Films deposited at RT and annealed and films deposited on substrates held above 385°C show a transformation comparable to NiTi shape memory films. However, the damping is one order of magnitude lower. Annealing does not significantly change the phase transition of films that were deposited at 385°C or above. 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.

EE5.18
EFFECT OF La ON THE GROWTH OF Cu_6Sn_5 INTERMETALLIC COMPOUND. Xin Ma, Hongyuan Fang, Yiyu Qian, Harbin Institute of Technology, Department of Materials Science and Technology, Harbin, CHINA; Fusahito Yoshida, Hiroshima University, Higashi-Hiroshima, JAPAN.

The thickness of the Cu-Sn intermetallic compounds(IMC) layer has played an important role on the reliability of microelectronic packaging at PCB level. Previous experimental results showed that, with the increasing of the thickness of Cu-Sn IMC layer, both the thermal fatigue life and the fracture ductility of solder joints decreased. Therefore, depressing the growth of Cu-Sn IMC should be an effective method to improve the reliability of solder joints. In this study, small amount of the rare earth element La (only 0.05wt.%) has been added into traditional Sn60-Pb40 solder alloy. The results of aging teats of solder joints showed that, after aged at 398K for 120 hours, the mean thickness of Cu-Sn IMC layer at the interface of solder joints decreased from 7.68μm to 5.16μm by adding small amount of La into solder alloy. Furthermore, the results of temperature cycling tests showed that, thermal fatigue life of solder joints was improved 3 times by La addition. In order to explain above experimental phenomenon, thermodynamic model has been introduced. At first, the calculating results of the interaction relation of constituent elements indicated that La has higher affinity with Sn in Sn-Pb system. Therefore, with the consideration that Cu will not react with Pb, the Cu-Sn-Pb-La quaternary system can be simplified to Cu-Sn-La ternary system. Thermodynamic calculating results showed that the driving force of formation of Cu_6Sn_5 IMC will be reduced by adding small amount of La, that is, the growth of Cu-Sn IMC will be depressed by La addition. On the other hand, the depressing effect of La is efficient only in a limited local mole fraction range. 0.16 at.% of La is the maximum value and 0.08at.% of La is the best value.

EE5.19
STRESS MEASUREMENTS IN THE LAYERS OF THIN FILM GAS SENSORS. Sung-Ho Choo, Youngman Kim, Chonnam National Univ, Dept of Metallurgical Engineering, Kwangju, KOREA.

The mechanical properties of thin film materials are known to be different from those of bulk materials, which are generally overlooked in practice. The difference in mechanical properties can be misleading in the estimation of residual stress states in micro-gas sensor with multi-layer structure during manufacturing and in service. In this study the residual stress of each film layer in micro-gas sensor was measured according to the five difference sets of film stacking structure used for the sensor. The Pt thin film layer was found to have the highest tensile residual stress, which may affect the reliability of micro-gas sensor. For the Pt layer the changes in residual stress were measured as a function of processing variables and during thermal cycling. In addition the effect of heat treatment on the microstructure was investigated during processing of the sensor. The residual stresses in the Pt layers caused by microstructural changes were also investigated.

EE5.20
CONTROL OF STRESS WITH GROWTH CONDITIONS AND MECHANICAL PARAMETERS DETERMINATION OF 3C-SiC HETEROEPITAXIAL THIN FILMS. C. Gourbeyre[1], T. Chassagne[2], M. Le Berre[1], G. Ferro[2], C. Malhaire[1], Y. Monteil[2], D. Barbier[1]. [1]Lab. Physique de la Matiere, UMR CNRS 5511, INSA de Lyon, Villeurbanne, FRANCE. [2]Lab. Multimateriaux et Interfaces, UMR CNRS 5615, U.C.B.L., Villeurbanne, FRANCE.

We report here on the influence of the epitaxial growth conditions on the residual stress of heteroepitaxial 3C-SiC, as an alternate material for MEMS, grown on silicon using APCVD and on the determination of its mechanical properties. 3C-SiC films were grown on (100) Si substrate in a vertical reactor by atmospheric-pressure chemical vapour deposition (APCVD). SiH_4 and C_3H_8 are used as precursor gas and H_2 as carrier gas. The growth procedure involves the formation of a carburization buffer layer at 1150°C under a mixture of H_2 and C_3H_8. The epitaxial growth occurs then at 1350°C by adding SiH_4. The measurement techniques implemented are: the substrate curvature, nano-indentation and load deflection measurements of self-suspended SiC membranes. The substrate curvature measurement leads to the determination of the residual stress in the deposited SiC film. We will show that we can achieve 3C-SiC layers with a compressive or a tensile state that is in a stress range from -550 MPa to 640 MPa. Whereas thermal mismatch just accounts for tensile stresses, we demonstrate the possibility of having compressive stresses using specific conditions for the buffer layer formation. The early stage of growth is indeed of major importance. Regarding the mechanical properties, the 3C-SiC Young's modulus was determined using nano-indentation. Its mean value reaches 360 GPa comparable to the calculated value of 320 GPa. As test structures, we have processed self-suspended SiC membranes. Load deflection measurements enables to determine the Young's modulus and the residual stress of the self-suspended films. In self-suspended SiC membranes, the absolute value of the residual stress in the SiC thin films steadily decreases and is around 200 MPa in a tension state. This stress decrease is due to the stress relaxation in the thin films when the self-suspended membranes are etched.

EE5.21
DOUBLE SIDE POROUS SILICON ON PATTERNED SUBSTRATES FOR THERMAL EFFECT MICROSYSTEMS. Stephanie Perichon, Volodymyr Lysenko, Boudjemaa Remaki, Daniel Barbier, INSA de Lyon, Materials Physics Laboratory (LPM), Villeurbanne, FRANCE.

Application of porous silicon in thermal microsystem structures often requires the formation of deep localized porous silicon layers. The most commonly used method to prepare the porous layers is the dc anodic etching of monocrystalline silicon in a fluorhydric acid based electrolyte. However inhomogeneity of the nanocrystallites size along the layer depth due to the decrease of HF concentration within the pores as well as the poor uniformity of the porous layer thickness limit the elaboration of deep porous layers. Thus we propose an original pulsed anodisation technique, using a double tank etching cell that allows to make localized porous silicon layers through the whole wafer thickness. An alternate pulsed anodisation current forms porous layers on each side of the initial silicon wafer. The switching time varies from 0.1s to a few seconds, the duty cycle (etching time/etching stop) from 0.1 to 1. Meso-porous silicon, 300 μm thick, was prepared on 0.02 $\Omega\cdot$cm silicon wafers and characterized in depth by micro-Raman spectroscopy showing an homogenous repartition of the crystallite size along the layer depth. Furthermore a selective double side pulsed anodisation of silicon was performed on patterned silicon substrates. Porous silicon is formed in pre-determined parts of the wafer using composite poly-silicon - silicon nitride masking layers. Technological solutions to get rid of porous layer thickness inhomogeneity due to non uniform current density distribution are discussed. Finally a toric porous silicon layer, crossing the whole silicon wafer, surrounding a 2 mm diameter monocrystalline silicon cylinder was successfully achieved ensuring a new approach of thermal isolation for thermal effect microsystems.

EE5.22
ON THE ROLE OF THE UNDERLYING MICROSTRUCTURE ON THE OBSERVED MACROSCOPIC PROPERTIES OF MEMS MATERIALS. Guy F. Dirras* and Kevin J. Hemker, John Hopkins University, Dept of Mechanical Engineering, Baltimore, MD. *Visiting from LPMTM-CNRS, University Paris XIII, FRANCE.

The flourishing new technology of Micro-Electro-Mechanical-Systems (MEMS) shows great promises in various areas, comprising for example critical applications such as weapons. Polycrystalline silicon thin films, of a few microns in thickness, are the primary structural support materials for MEMS applications. Nevertheless, a better understanding of the correlation between mechanical properties and thin films microstructure is needed for reliability of MEMS devices. In earlier work (Sharpe et al., Youngs Modulus and Strength of Three Polysilicons, this symposium), the mechanical properties of polycrystalline silicon thin films supplied by Cronos (MCNC), Standard MEMS Inc. (SMI) and Sandia National Laboratories have been tensile tested using a special microsample testing set-up. The strength of these films was found to be 1.6 GPa, 2.1 GPa and 3.2 GPa respectively. In the present work extensive Scanning Electron Microscopy, Atomic Force Microscopy and Transmission Electron Microscopy studies have been undertaken to characterize the underlying microstructure of CRONOS and SMI materials. Variations in sample geometry, surface roughness, grain size distribution and orientation will be quantified and compared to the differences in strength that have been reported. Special attention will be paid to processing-microstructure-property relations throughout. The cooperation with D.A. Lavan of Sandia National Laboratories and R. Mali of SMI in arranging supply of the specimens is gratefully acknowledged.
This research was conducted under the sponsorship of DARPA F30602-99-2-0553.

EE5.23
TEMPERATURE DEPENDENCE OF THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF LIGA NICKEL FOR MEMS. H.S. Cho, Dept of Mechanical Engineering and Materials Science and Engineering, The Johns Hopkins University, Baltimore, MD; W.G. Babcock, Naval Surface Warfare Center, Indian Head Division, Indian Head, MD; H. Last, Institute for Defense Analyses, Alexandria, VA; K.J. Hemker, Dept of Mechanical Engineering and Materials Science and Engineering, The Johns Hopkins University, Baltimore, MD.

Electrodeposited LIGA Ni structures have received considerable attention in recent years because high-aspect-ratio 3-D structures of these materials can easily be made with a high precision. The emergence of elevated temperature microelectomechanical systems (MEMS) applications have resulted in the need for an understanding of microstructural stability and high temperature mechanical properties of LIGA Ni. The dogbone shaped LIGA Ni microsamples used in the current study have been annealed at temperatures of 400°C to 800°C for 5min. to 60hours. Cross-sectional optical micrographs evidence a transition from the as-deposited columnar microstructure to more equiaxed grains at temperatures between 400-500°C after 1hour annealing. Extensive grain growth was observed above 500°C. The microhardness and tensile properties of the as-deposited and heat treated microsamples have been measured, and these results evidence a dramatic decrease in strength for specimens heat treated at temperature above 400°C. The evolution of the mechanical properties of the annealed microsamples will be combined with elevated temperature creep tests and used to develop an understanding of the elevated temperature-microstructure-properties relations that exist in LIGA Ni MEMS materials. The results of this study will also be contrasted with an intermediate temperature application that is currently under development at NSWC.

EE5.24

MECHANICAL PROPERTY CHANGES IN LPCVD DEPOSITED LOW-STRESS SILICON NITRIDE FILMS AS INFLUENCED BY STRESS TUNING OF DEPOSITION PARAMETERS. Kevin Roberts, Greg Cibuzar, Univ of Minnesota, Microtechnology Lab, Minneapolis, MN; Jeremy Thurn, Yvete Toivola, Robert Cook, Univ of Minnesota, Dept of Chemical Engr & Matls Sci, Minneapolis, MN.

A popular material in use today for surface micro-machining is LPCVD (Low Pressure Chemical Vapor Deposited) low-stress silicon nitride (i.e., silicon-rich nitrides with film stresses on the order of 150 MPa or less). Much work has been done outlining the effects of process parameters on the average wafer-level film stress. To gain greater insight into the mechanical aspects of these films, additional properties were studied while tuning these films to a particular stress. Low-stress silicon nitride films on the order of 1 micron thick were formed under varying LPCVD furnace conditions. Average film stress was adjusted by changes made to deposition temperature, deposition pressure, and gas composition ratio (Ammonia to Dichlorosilane ratio) for different sample groups. These sample groups were representative of desirable low-level stresses typical in MEMS applications (150 MPa compressive to 300 MPa tensile). In addition to the necessary determination of the average stress of these films, other mechanical properties of these films were determined and correlated to the process parameter used to adjust the stress. Additionally, in the case where the gas ratio was used to adjust the stress, the Si to N ratio of the samples was determined using X-ray Photoelectron Spectroscopy (XPS) and correlated to the material property change. The mechanical properties measured in these films were: modulus and hardness (using nanoindentation), thermal coefficient of expansion, and thermal stability. Understanding of the effects of deposition parameters on mechanical properties will lead to better control of these properties for advanced MEMS applications.

EE5.25

BEYOND SILICON: MICROFABRICATION WITH METALS, PLASTICS, AND CERAMICS. Alfredo M. Morales, Michelle A. Bankert, Douglas A. Chinn, Linda A. Domeier, Marcela Gonzales, John T. Hachman, Craig C. Henderson, Jill M. Hruby, Richard P. Janek, Dawn M. Skala, Michael R. Winter, Sandia National Laboratories, Microsystems Processing Department, Livermore, CA.

Modern devices in the macroscopic world often combine components made from metal, plastics, and ceramics in order to attain improved or novel functionalities. In contrast, microelectromechanical systems (MEMS) are fabricated mainly from silicon and a few metals, resulting in limited functionality. In order to increase the range of materials available for MEMS, our group has developed and combined different microfabrication and materials processing techniques. For instance, we have established a complete LIGA (x-ray based lithography) program to produce high aspect ratio metal microparts or microstamps by electrodeposition. We commonly plate nickel, copper, nickel-iron, and gold. A combination of LIGA, polishing, and electrodeposition enables us to make three dimensional metal cavities with micron size features on the internal surface. Such cavities have applications as microwave resonance cavities and microbellows for actuation. We have used LIGA fabricated microstamps to produce plastic replicates by either hot embossing or injection molding. Plastic microparts are desirable as enabling components of microanalytical and medical instrumentation. Ceramic or nanocomposite microparts, with uses ranging from high temperature applications to magnetics, were produced by micromolding ceramic nanoparticles into LIGA produced plastic molds. Finally, we have developed micro powder metallurgy techniques that allow us to make microparts from a variety of metals including stainless steel. This talk will describe our base-line processes, materials properties, and applications.

EE5.26

FABRICATION OF NiTiHf HIGH-TEMPERATURE SHAPE MEMORY ALLOY THIN FILMS. X.L. Liang, H.M. Shen and Y.N. Wang, National Laboratory of Solid State Microstructures, Nanjing University, Nanjing, PR CHINA.

Shape memory alloys (SMAs) thin films are one of the candidate materials for Microelectromechanical System (MEMS). A lot work has been done on the fabrication and shape memory effect (SME) of SMAs thin films. However, most work focus on NiTi thin films, which transformation temperatures are limited to 373 K. This hinders its application in high temperature environments. So it is imperative to develop high-temperature SMAs thin films. As we know now, the transformation temperatures of NiTi can be enhanced by ternary substitution Hf or Pd for Ti or Ni. In this paper, we try to fabricate NiTiHf thin films by magnetron sputtering. The crystallization conditions of sputtered thin films are investigated and the martensite structure of crystallized thin films is determined to be B19'. The transformation temperatures are above 373 K. Shape memory effect has been observed and measured by bending experiments.

EE5.27

UNSTABLE ETCHING OF SILICON(110) IN KOH. Z. Moktadir, K. Sato, M. Shikida and T. Shimizu, Dept. of Microsystems Engineering, Nagoya University, Nagoya, JAPAN.

Nowadays, silicon micromachining is a popular technique that is used to fabricate sensors and actuators. KOH and TMAH are the most common products used for etching and are known to be anisotropic. After etching, the smoothness of obtained surfaces is of crucial importance for the performance of the fabricated devices. It is therefore critical to study the mechanisms of the roughening and faceting of surfaces during silicon dissolution. Under specific experimental conditions, etching of Si(100) leads to the formation of hillocks with pyramidal shapes. These pyramids are often bounded by {111} vicinal planes and {110} ridges. For etched Si(110), the surface shows a hills and valley structure. The hills and valleys are formed along <100> direction leading to a pattern of lines that point in the <110> direction. In the present work, we suggest an interpretation for the early stage formation of facets in Si(110) etched with KOH. Silicon wafers oriented (110) were etched in KOH. The KOH concentration used is 30% and the etching temperature was 70°C. The surfaces were investigated using a laser scanning microscope (LSM) (ZAYGO NEWVIEW5030). Many studies show that the anisotropy of the surface tension is one of the causes of the formation of facets during crystal growth.[1-3]. Recently, A. Golovin and co-workers[4] have developed a phenomenological model for the formation of facets and corners in a thermodynamically unstable crystal surface (in the case of kinetically controlled growth). In their study, the faceting is caused by strongly anisotropic surface tension. In certain conditions, the formed surface structure shows grooves forming a quasi 1-dimensional pattern (oriented in x or y direction) for [110] crystallographic orientation of a crystal with cubic symmetry. We suggest that the early stage of formation of facets in Si(110), can be interpreted using the linearized version of the equation of Golovin et al. We performed a linear stability analysis to understand the origin of the faceting in Si(110) when etched with KOH. The linear theory predict a large band of wave vectors in (110) direction that grow in time, leading to the formation of facets. The results show a dominant wave vector which maximizes the structure factor. The results are qualitatively reproduced by the theory. These results show that anisotropic surface tension is responsible for the facets formation.

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EE5.28

Abstract Withdrawn.

EE5.29

SIMPLE AND LOW COST PATTERNING PROCESS FOR SPUTTERED $Pb(Zr,Ti)O_3$ THIN FILMS AND ELECTRODES FOR MEMBRANE-BASED MICROSYSTEMS APPLICATIONS. Cyril Millon, Christophe Malhaire, Emmanuel Defay, Daniel Barbier, Stephanie Perichon, INSA de Lyon, Materials Physics Laboratory (LPM), Villeurbanne, FRANCE.

Micromachined silicon membranes actuated by a piezoelectric thin film are of great interest for micro-fluidic or ultra-sonic applications. In this paper, we present a simple and low cost patterning process for sputtered Ti and Pt electrodes and $Pb(Zr,Ti)O_3$ thin films using lift-off and a wet HCl etching methods respectively. We have shown by finite element analysis the interest to pattern electrodes in order to get higher actuation performance. Moreover, in order to reduce the stress on the diaphragm, the PZT film has been also patterned. We propose an optimized flow chart that takes into account the different material properties in the multi-layer stack constituting the actuator and the need of a PZT crystallization annealing. To show the influence of the actuator layout, finite element models of a 20 μm thick, 3 x 3 mm^2 silicon membrane with variable electrode design, actuated by a 1 μm PZT (10 V bias) thin film have been developed. We found a maximum deflection for a 1.7 mm side length square electrode located to the membrane center. Sputtering was achieved on a non heated silicon substrate, in pure Argon, using a stoichiometric PZT target. The metallic electrodes have been structured by standard lift-off technique and bondpads have been located on the membrane frame to avoid stress effects. Different concentrations of HCl [4%-9%] with temperature up to 98°C have been tested. Well defined PZT microstructures were better achieved by wet etching of non annealed PZT film onto SiO_2 or Pt (6%, 95°C). The etching rate was 0.07 μm per second. The presence of the perovskite phase in the PZT films for a 700°C, 30s, in air, RTA, has been checked by X-ray measurements. Finally, the silicon membranes have been realized by anisotropic chemical etching in an aqueous KOH (40%, 60°C) solution.

EE5.30
MECHANICAL NANOMACHINING FOR MEMS APPLICATIONS.
Guohui Zhou, Yury Gogotsi, Drexel University, Department of Materials Engineering, Philadelphia, PA; Sang-Song Ku, Sabri Cetinkunt, University of Illinois at Chicago, Department of Mechanical Engineering, Chicago, IL.

The single point diamond turning (SPDT) machine was used to make grooves on (111) p-type single crystal silicon surfaces at room temperature. The SPDT machine consists of a high precision air bearing spindle and an X-Y stage plus a nanopositioner with about 2.5 nm resolution. Scratch tests have been performed with both a sharp diamond tool (Vickers or conical indenter) and a spherical diamond tool (Rockwell indenter). Material removal mechanisms appeared to be different for these tools. Pressure-induced metallization of Si allows for the ductile regime mechanical micromachining of wafer surfaces. Raman microspectroscopy and electron microscopy were used to determine the machining regimes that do not introduce cracking or other damage. For example, grooves (channels) for microfluidic devices can be engraved with the channel shape determined by the shape of the tool, thus, providing a possibility to create the structures that cannot be produced using etching. There is no limitation for the channel length/direction, and the channel width can be varied from nanometers to several microns. The surface of the groove after machining was covered by a mixture of metastable polymorphs of silicon or amorphous silicon. These phases can be transformed to cubic silicon by subsequent annealing. The maximum depth of cut in ductile regime has been determined for the given test conditions and tools. The developed technique can be used for micro- and nanomachining of Ge, GaAs and other semiconductors.

EE5.31
MICROFABRICATION OF CREVICE CORROSION SAMPLES.
Xiaoyan Wang, Michael L. Reed, Robert G. Kelly, Jason S. Lee, Michael L. Reed, School of Engineering and Applied Science, University of Virginia, Charlottesville, VA.

A major challenge in developing computer models for crevice corrosion lies in fabricating appropriate experimental cervice samples. The geometry and dimensions of these samples must be controlled to a high order of precision in order to be amenable for comparison to computational models. In this work we report on efforts to construct crevice samples with rigorously defined dimensions by using microfabrication techniques developed for microelectromechanical systems (MEMS). These techniques include microfabrication with SU-8, electroplating, chemical mechanical polishing, as well as basic semiconductor device fabrication techniques. The crevice substrates contain two-dimension arrays of electrodes of the metal to be studied, isolated by walls of SU-8. The electrodes have individual electrical connections so that spatial information of the in-situ corrosion process can be obtained. The crevice formers with SU-8 posts were coupled to crevice substrates to maintain a uniform crevice gap. Further, crevice formers with regular rectangular subcrevices were fabricated to study the roles of subcrevices in crevice corrosion.

EE5.32
PLASMA ETCHING OF A HIGH TEMPERATURE GLASS-CERAMICS. Junting Liu, Nikolay I. Nemchuk, Dieter G. Ast, Cornell Univ, Dept of Materials Science and Engineering, Ithaca, NY; Nan Jiang, J. Silcox, Cornell Univ, School of Applied Engineering Physics, Ithaca, NY; J. Gregory Couillard, Corning Incorporated, Corning, NY.

Fabrication of MEMS devices for applications in important areas such as optical communication, biotechnology, health care etc. requires transparent substrates compatible with conventional lithographic and micromachining techniques. Ability to withstand high processing temperatures is crucial to fabricate a chip where the MEMS structure is integrated with a signal processing or driving electronic circuits. Novel transparent, high strain point spinel LGA glass-ceramics recently developed by Corning Incorporated are suitable substrates for thin film polysilicon electronics and appear to be promising materials for MEMS processing. To explore the possibility of using these glass-ceramics as inexpensive substrates for MEMS fabrication and to investigate the effect of the structure and composition of the substrate on its etching behavior in the fluorocarbon plasma we processed wafers of LGA glass-ceramic, amorphous "green" glass and amorphous fused silica. The LGA glass-ceramic consists of 10 nm spinel crystals evenly distributed in a siliceous glass matrix, while the "green" glass is amorphous and has the composition of LGA glass-ceramic. A 400 nm sputtered Cr film patterned by photolithography was used as an etching mask. Etching experiments were performed in a Materials Research Corp. Model 720 Magnetron Ion Etcher. SEM, AFM, Microprobe, XPS, TEM and EELS techniques were employed to analyze the effect of CF_4 plasma on surface morphology of the substrates, their composition and structure near the surface. Etch rates as high as 80 nm/min were achieved in the multi-component "green" glass and glass-ceramics, however this value is lower than that of pure fused silica. Although the etch rates of "green" glass and glass-ceramic were similar, plasma etched amorphous "green glass" had a higher surface roughness than that of glass-ceramics. Changes in the surface composition and structure of the multi-component substrates will be discussed.

EE5.33
FABRICATION OF MEMS COMPONENTS BASED ON ULTRANANOCRYSTALLINE DIAMOND THIN FILMS AND CHARACTERIZATION OF MECHANICAL PROPERTIES.
A. Sumant, Materials Science and Chemistry Divisions, Argonne National Laboratory, Argonne, IL; O. Auciello, Materials Science Division, Argonne National Laboratory, Argonne, IL; A.R. Krauss, D.M. Gruen, Materials Science and Chemistry Divisions, Argonne National Laboratory, Argonne, IL; D. Ersoy, Materials Engineering Department, University of Illinois-Chicago, IL; J. Tucek, Materials Science and Chemistry Divisions, Argonne National Laboratory, Argonne, IL; N. Moldovan, D. Mancini, Experimental Facility Division, Argonne National Laboratory, Argonne, IL.

The mechanical, thermal, chemical, and tribological properties of diamond make it an ideal material for the fabrication of MEMS components. Cost-effective fabrication of these components will involve coating Si components with diamond or by producing MEMS components via etching or selective area deposition of diamond films. However, conventional CVD diamond deposition methods result in either a coarse-grained pure diamond structure that prevents high-resolution patterning, or in a fine-grained diamond film with a significant amount of intergranular non-diamond carbon. At Argonne National Laboratory, we are able to produce phase-pure ultrananocrystalline diamond (UNCD) films for the fabrication of MEMS components. UNCD is grown by microwave plasma CVD using C60-Ar or CH4-Ar plasmas, resulting in films that have 3-5 nm grain size, are 10-20 times smoother than conventionally grown diamond films, and can have a brittle fracture strength similar to that of single crystal diamond. We used lithographic patterning, lift-off, and etching, in conjunction with the capability for growing UNCD on SiO_2 to fabricate 2-D UNCD-MEMS cantilevers and pinwheels. We also coated Si posts with conformal UNCD films to produce shafts for rotor and gear support. We have performed initial characterization of mechanical properties by using nanoindentation technique. The values of Hardness(∼88 GPa) and Young modulus (∼864 GPa) measured are very close to those of single crystal diamond (100 GPa and 950 GPa respectively). The results show that UNCD is a promising material for future high performance MEMS devices.
Work supported by the U.S. Department of Energy, BES-Material Sciences, under Contract W-31-109- ENG-38.

EE5.34
ASYMMETRICAL POLYSILICON ELECTROTHERMAL ACTUATORS FOR ACHIEVING LARGE IN-PLANE MECHANICAL FORCES AND DEFLECTIONS. Edward S. Kolesar, Simon Y. Ko, Jeffery T. Howard, Peter B. Allen, Josh M. Wilken, Noah C. Boydston, Matthew D. Ruff and Richard J. Wilks, Texas Christian University, Department of Engineering, Fort Worth, TX.

Several electrically-driven microactuators have been investigated for positioning individual elements in microelectromechanical systems (MEMS). The most common modes of actuation are electrostatic, magnetostatic, piezoelectric and thermal expansion. This investigation reports a new polysilicon electrothermal actuator design. In the traditional electrothermal actuator design, the single-hot arm is narrower than the cold arm, and thus, the electrical resistance of the hot arm is greater. When electrical current passes through the device (both the hot and cold arms), the hot arm is heated to a higher temperature than the cold arm. This temperature differential causes the hot arm to expand along its length, thus forcing the tip of the device to rotate about a flexure. The new double-hot arm thermal actuator design eliminates the parasitic electrical resistance of the cold arm by incorporating an additional hot arm. The second hot arm results in an improvement in electromechanical efficiency by providing a return current conductor that is also mechanically active. Furthermore, in the new electrothermal actuator design, the rotating cold arm can have a narrower flexure compared to the flexure in the traditional device because it no longer needs to conduct an electrical current. The thinner flexure results in an improvement in mechanical efficiency. This research compares the performance of the single- and double-hot arm electrothermal actuator designs. Force and deflection measurements of both actuator designs as a function of arm length and applied electrical power are presented. The electrothermal actuator designs were accomplished with the L-Edit software program, and they were fabricated using the Multi-User Microelectromechanical Systems (MEMS) Process (MUMPs) foundry at the Microelectronics Center of North Carolina (MCNC).

EE5.35
ISSUES IN THE FLEXIBLE INTEGRATION OF SPUTTER-DEPOSITED PZT THIN FILMS WITH POLYSILICON AND Ti/Pt ELECTRODE LAYERS FOR USE AS SENSORS AND ACTUATORS IN MICROELECTROMECHANICAL SYSTEMS (MEMS). C.F. Knollenberg, T. Sands, Department of Materials Science & Mineral Engineering, University of California, Berkeley, CA; A.S. Nickles, Capacitor Division, Applied Materials, San Jose, CA; R.M. White, Department of Electrical Engineering & Computer Sciences, University of California, Berkeley, CA.

Sputter-deposited piezoelectric lead zirconate titanate (PZT) thin films with Ti/Pt and polysilicon electrode layers are being investigated for use in Microelectromechanical Systems (MEMS). Existing research shows the nucleation of the perovskite phase of the PZT is linked to the lattice spacing of the underlying Pt electrode and/or seed layers, and is key in obtaining PZT layers with good piezoelectric/ferroelectric properties. Our research with piezoelectric PZT films on Ti/Pt electrode layers aims at employing these films to generate and receive acoustic waves in flexural plate wave devices (FPWs). Our experiments indicate the formation of a random polycrystalline perovskite phase is linked to the emergence of oriented <200> Pt grains within the dominant <111>-oriented crystal structure during rapid thermal annealing in an oxygen environment. Pt films annealed in nitrogen, in contrast, retained their <111> preferential orientation without the formation of Pt <200> grains. PZT films deposited on these electrodes and annealed in nitrogen were strongly oriented in the <111> direction, but exhibited lossy ferroelectric behavior and were prone to delamination. We are also investigating the feasibility of using doped polysilicon electrode layers with PZT thin films. The multiple layers used with the Pt electrode (Pt, Ti, and silicon dioxide adhesion layer) have significant interactions with one another, and replacing these layers with a single electrode layer should alleviate these complications. A low-temperature PZT deposition process (300°C) and short annealing cycles (30 sec.) coupled with a titanium dioxide barrier/seed layer should prevent interdiffusion and reactions between the polysilicon and PZT layers. Our experiments show PZT films deposited and annealed on doped polysilicon layers develop a random polycrystalline perovskite phase, but are subject to tensile cracking. The use of polysilicon as an electrode layer should also facilitate the integration of piezoelectric PZT layers with polysilicon surface micro-machined structures using SiGe sacrificial layers.

EE5.36
FABRICATING STRESS COMPENSATED, PLANAR, SUSPENDED PIEZOELECTRIC THIN FILM BEAM RESONATORS. Ronald G. Polcawich, General Technical Services, Wall Township, NJ; Eugene Zakar, Brett Piekarski, Richard Piekarz, John Conrad, Madan Dubey, Army Research Laboratory, Adelphi, MD; George McLean, General Technical Services, Wall Township, NJ.

Residual stress plays an important role in the fabrication of suspended thin film piezoelectric devices. This work focuses on the role of materials in a suspended dielectric / Ta / Pt / PZT / Pt multilayer stack. PECVD SiO_2, Si_3N_4, and combinations of both were studied as the dielectric. Residual stress measurements via wafer curvature concluded that all of the materials possessed tensile stresses. The Pt layers possessed the largest values with the bottom Ta / Pt layer (859 ± 29.1 MPa) having a greater stress than the top Pt (50 - 600 MPa depending on annealing conditions). In contrast, relatively moderate to low tensile stress were present in the PZT (127 ± 18 MPa) and SiO_2 (48.8 ± 7.2 MPa) layers. This stress gradient resulted in non-planar suspended beams in initial devices. The large residual stress of the bottom platinum layer caused the beams to bend about the neutral axis. To produce planar resonators, these large tensile stresses were compensated through the use of additional dielectric layers (e. g., SiO_2 Si_3N_4 multilayer stacks) to stress balance the beams around the neutral axis. The relatively low stress SiO_2 layers offset the highly tensile Si_3N_4 layer (1.1×10^3 ± 290 MPa) from the highly stressed Pt layers and resulted in planar suspended beams. Resonator performance will also be presented for both planar and non-planar resonators.

EE5.37
MEASUREMENT OF ELECTRIC CHARGE DURING NANOINDENTATION EXPERIMENTS ON FERROELECTRIC THIN FILMS. Miguel Alguero, Andrew J. Bushby and Michael J. Reece, Department of Materials, Queen Mary and Westfield College, University of London, UNITED KINGDOM.

We have developed a procedure using nanoindentation with spherical tipped indenters capable of providing the elastic modulus of ferroelectric thin films for MEMS devices that allows for their anelastic deformation during indentation. The procedure also provides the anelastic and plastic contributions to the deformation as a function of the indentation stress. The procedure was demonstrated on a range of unpoled lanthanum modified lead titanate $(Pb,La)TiO_3$ thin films. It was shown that the elastic modulus of the films ranged from 110 to 147 GPa depending on the microstructure and orientation of the films. It was also shown that the anelastic deformation was associated with the movement of ferroelastic domain walls. An experimental method has been developed so that electric fields can be applied and the electric charge generated can be measured during nanoindentation. We present here results on the simultaneous measurement of penetration and electric charge generated during the indentation of poled $(Pb,La)TiO_3$ films. The possibility of measuring an indentation piezoelectric coefficient with high spatial resolution is discussed.

EE5.38
PROCESS AND FABRICATION DEVELOPMENT OF A PZT MEMS MAGNETOMETER. Eugene Zakar, Madan Dubey, Ronald Polcawich, John Conrad, Richard Piekarz, Brett Piekarski, US Army Research Laboratory, Sensors and Electron Devices Directorate, Adelphi, MD.

The overall program goal is to develop an extremely sensitive microelectromechanical (MEMS) magnetometer, based on a resonating xylophone bar structure. A preliminary prototype device for process development rather than operational performance has been completed. The structure was fabricated using sol-gel deposited PZT (Lead Zirconate Titinate -52/84) onto a $Pt/SiO_2/Si$ substrate, with the Pt layer forming the bottom electrode. A subsequent Pt layer deposited onto the PZT formed the device top electrode. The resultant $Pt/PZT/Pt/SiO_2/Si$ stacks were patterned with photoresist and defined by ion beam milling, and reactive ion etching in HC_2/ClF_4 plasma to form the xylophone bar structure. A PECVD SiO_2 isolation film was deposited over the electrode contact areas, and vias etched, followed by the formation of Ti/Au contact bonding pads using lift-off technique. The xylophone bar was then released. Measurements of residual stress in Pt/PZT/Pt films were also studied using a commercial stress analyzer.

EE5.39
WET-ETCH PATTERNING OF LEAD ZIRCONATE TITANATE (PZT) THICK FILMS FOR MICROELECTROMECHANICAL SYSTEMS (MEMS) APPLICATIONS. Li-Peng Wang, S. Trolier-McKinstry and R.J. Davis, Penn State University, PA.

Lead Zirconate Titanate (PZT) films are very attractive for Microelectro-mechanical-Systems (MEMS) applications because of their high piezoelectric coefficients and good electromechanical coupling. In this work, wet-etch patterning of sol-gel Lead Zirconate Titanate (PZT) films for MEMS applications, typically having films thicknesses ranging from 2 to 10 microns, was studied. A two-step wet-etch process was developed. In the first step, 10:1 buffered HF was used to remove the majority of the film at room temperature. Then, a solution of $2HCl:H_2O$ at 45°C was used to remove residue from the first step. This enables successful patterning of PZT films up to 8 microns thick. The high etch rate (0.133 μm /min), extremely high selectivity with respect to photoresist, and limited undercutting (2:1 lateral:thickness) were obtained. X-ray diffraction and scanning electron microscopy (SEM) were used to analyze the residue produced in the first etching step and examine the etching profile, respectively. In addition, two MEMS devices, an ultrasonic transducer and an accelerometer, were demonstrated using this etching process to pattern the active PZT films.

EE5.40
NON-LITHOGRAPHIC DIRECT-WRITING AND LASER PROCESSING OF DIELECTRIC AND PIEZOELECTRIC STRUCTURES FOR MESOSCOPIC APPLICATIONS. D. Young, J.M. Fitz-Gerald, R.C.Y. Auyeung, H.D. Wu, R. Modi, A. Pique, D.B. Chrisey, Naval Research Laboratories, Surface Modification Branch, Washington, DC.

The recently-developed MAPLE-DW deposition technique utilizes a focused, pulsed UV laser (355 nm) to transfer material from a transparent carrier onto a target substrate. In MAPLE-DW, the laser pulse propagates through the transparent carrier and impacts a coating that consists of the material to be transferred and an organic vehicle. The focused laser pulse decomposes the vehicle and propels the material from the carrier onto the substrate. This system is capable of concurrent in-situ laser machining, trimming and thermal processing. The flexible combination of additive and subtractive capabilities allows direct-writing of <10 micron resolution structures. MAPLE-DW has been designed to operate in air, at room temperature, and without the need for masking, lithography or etching. MAPLE-DW has been used to fabricate capacitor structures of barium titanate (BTO) and barium strontium titanate (BST) for dielectric applications. Similar lead zirconate titanate (PZT) structures were also produced for evaluation as piezoelectric actuators and sensing elements. The starting materials consist of an organic vehicle, dielectric powder, and metalorganic precursor. Low temperature processing (400C) of BTO and BST results in an adherent, optimally packed, porous material with dielectric constants of 75-100 and 150-200, respectively. In-situ processing with an IR annealing laser (1.064 or 10 microns) substantially improves both dielectric constant and loss tangent, and alters the piezoelectric properties of PZT. Dielectric measurements were performed as a function of frequency up to 1.5 GHz, and as a function of temperature from -130 to 150 Celsius.

EE5.41
ELECTRICAL POWER GENERATION USING PIEZOELECTRIC THIN FILM MEMBRANES. N.E. Apperson, D.F. Bahr, C.D. Richards, and R.F. Richards, Mechanical and Materials Engineering, Washington State University, Pullman, WA.

Piezoelectric thin films on bulk micromachined silicon cavities are shown to respond electrically to a cyclic pressure wave. Two materials have been tested for use in power generation, a ceramic thin film (PZT) and a polymer thin film (PVDF) using solution deposition and thermal bonding respectively. The relationship between the deflection of the membrane and the resulting charge development were found using a dynamic bulge tester. The frequency response of both materials has been characterized, and is found to have a optimal pressurization frequency for power generation. The charge development during pressurization has been used to directly determine the d31 piezoelectric coefficient of both materials. A variation in ceramic film processing utilizing a multilayer structure of alternating dopants (PLZT) with PZT has been shown to produce ferroelectric fatigue resistance of over 1 billion cycles. The ferroelectric fatigue behavior is compared to the power generation degradation during mechanical fatigue testing, and differences between the clamped ferroelectric fatigue test and the flexing membrane of the direct mechanical test are discussed.

SESSION EE6: PACKAGING
Chair: M. Okandon
Tuesday Morning, November 28, 2000
Room 206 (Hynes)

8:30 AM *EE6.1
MATERIALS SCIENCE CHALLENGES IN DMD^{TM} PACKAGING. S.J. Jacobs, S.A. Miller, R.A. Robbins, J.J. Malone, V.C. Lopes, Texas Instruments, Dallas, TX.

The Digital Micromirror Device^{TM} DMD^{TM} is a MOEMS spatial light modulator that forms the basis of projection displays used in business, commercial, and home entertainment settings. As with any MEMS device, packaging presents its own challenges, many of which are not addressed in the "standard" semiconductor processing arena. The constraints presented by the nature and application of the device require special attention to many aspects of assembly and test. This talk will cover the evolution of the DMD^{TM} package, back-end processes, and testing. The importance of materials science and analysis on the path to an understanding of the package impact on device performance, as well as effective, lower cost packaging will be discussed.

9:00 AM EE6.2
STRESSY METAL MEMS. David K. Fork, Xerox, Palo Alto Research Center, CA.

We report on a new type of micro-fabricated structure based on stress engineered metal thin films. Metals afford many enhanced properties compared with the conventional materials commonly used for fabricating micro-mechanical components. These properties include conductivity, hardness and intrinsic stress. DC magnetron sputtering is an effective technique for controlling the stress profile in a broad class of deposited materials. This has enabled the fabrication of micro-machined metal cantilevers that bend away from the substrate upon their release from the substrate. Useful application of this property is currently under investigation for flip-chip integrated circuit packaging and reusable spring contacts for circuit probing. The arrival of this technology may be timely given that bonding-pad densities on high-performance integrated-circuit chips are beginning to exceed the limits of available interconnect technologies. We have microlithographically fabricated functional off-chip interconnects from highly elastic cantilever springs in linear arrays on pitches down to 6 m. The very high compliance of the springs may permit the fabrication of packages that are highly resistant to thermal fatigue. This work is performed in collaboration with Georgia Tech, and NanoNexus Inc.
This work is supported in part by the NIST Advanced Technology Program under Award 70NANB8H4008.

9:15 AM EE6.3
CHARACTERIZATION OF SILICON FUSION BONDS USING A FOUR-POINT BEND SPECIMEN. K.T. Turner, Dept. of Mechanical Engineering; A.A. Ayon, Dept. of Electrical Engineering and Computer Science; D. Choi, Dept. of MS&E; B. Miller and S.M. Spearing, Dept. of Aeronautics and Astronautics, Massachusetts Institute of Technology, Cambridge, MA.

The increased number of MEMS devices that are fabricated by bonding two or more bulk micromachined silicon wafers has highlighted the need to produce reliable silicon fusion bonds. Silicon fusion bonds are fabricated by bringing two mirror polished silicon wafers into contact and then subsequently annealing. It is of great interest to determine the influence of various processing conditions on bond quality. In particular, there is a desire to reduce the annealing temperature because of the limitations it places on fabrication steps prior to bonding. In the current study, the annealing temperature and time as well as the contacting ambient and pressure were systematically varied among samples. The critical strain energy release rate, G_C, was chosen as the figure of merit by which to evaluate the bond quality. To measure the critical strain energy release rate, a four-point bend specimen composed of two bonded layers and an initial notch at the center was employed. This specimen geometry is advantageous because it does not require measurement of crack length to calculate the critical strain energy release rate and is insensitive to damage near the specimen edges. The specimens were fabricated by bonding two silicon wafers and die-sawing into individual specimens. To ensure that the crack propagated along the bonded interface, shallow trenches were etched in the surface of the wafers to reduce the area of the bonded region. This technique has proven to be repeatable and straightforward. Details of the test method and results will be presented.

9:30 AM EE6.4
PHENOMENOLOGICAL MODEL OF NON-EVAPORATED GETTER FOR MICROELECTROMECHANICAL SYSTEMS (MEMS) APPLICATIONS. Caroline A. Kondoleon and Thomas Marinis, The Charles Stark Draper Laboratory, Electronics Packaging and Prototyping Division, Cambridge, MA.

There has been increasing interest in the development and use of microelectromechanical systems (MEMS) for various applications. Some MEM devices such as gyroscopes, accelerometers and bolometers, must be sealed under vacuum, at pressures below 10 millitorr, for efficient operation. A gas absorbing material (getter) is placed in the packages of these devices, to help maintain vacuum levels over service lives of many years. Getter, of this type, is activated by heating under vacuum, just prior to sealing the package. This study was undertaken to develop a model that could be used to estimate the quantity of getter needed as well as optimize the activation process subject to process constraints on time and temperature. The material studied was a titanium and zirconium-based alloy (7:3 by weight) non-evaporable getter in the form of strips produced by SAES Getters. The zirconium alloy consisted of zirconium (70.0%), vanadium (24.6%) and iron (5.4%) by weight. The getter was analyzed under different ambient conditions of temperature, time and atmospheric pressure. Auger electron spectroscopy (AES) depth profiling was used to analyze the diffusion depth of the contaminant gases absorbed by the getter material under each condition. The data acquired from the depth profiles were fit to a simple diffusion model. This model is currently being validated, by activating the getter material under various ambient conditions, and measuring pressures and gas compositions inside packages using a residual gas analyzer (RGA). The utility of this model for optimization of getter activation and estimating package vacuum levels over time will be discussed.

9:45 AM EE6.5
HIGH TEMPERATURE, HIGH PRESSURE FLUID CONNECTIONS FOR POWER MICRO-SYSTEMS. Todd S. Harrison, Adam P. London and S. Mark. Spearing, Department of Aeronautics and Astronautics, Massachusetts Institute of Technology, Cambridge, MA.

High power density micro-systems offer the potential to revolutionize technologies for portable electrical power generation, propulsion and flow control. Devices are being designed and fabricated which include micro-gas turbine engines, micro-rocket engines, micro-motor-compressors, micro-pumps and micro-hydraulic transducers. Common to all of this family of devices is the need to create packages that service the devices, and interface them with the macro-scale environment. Fluid interconnections are a particularly demanding packaging element for this class of devices. In order to achieve high power densities, these devices are required to operate at high pressures and, in some cases, high temperatures. This paper will describe the design, analysis, fabrication and testing of high pressure, high temperature fluid connections for the micro-engine and micro-rocket applications. A glass bonding technology has been developed to allow the creation of multiple fluidic connections consisting of Ni/Fe alloy tubes to silicon devices. Emphasis will be placed on strategies for minimizing the mismatch in coefficients of thermal expansion between the components, the minimization of flaws in the glass and the importance of wetting of the glass to both the tubes and the silicon. Test results will be presented which correlate the strength and statistical reliability of such bonds to the processing conditions, choice of glass and surface preparation prior to bonding. Data from complete devices under high temperature, high pressure fluid testing will also be presented. Recommendations will be made for the elements of a successful power micro-systems packaging strategy.

SESSION EE7: NEW MATERIALS
Chair: Hal Kahn
Tuesday Morning, November 28, 2000
Room 206 (Hynes)

10:30 AM *EE7.1
DEVELOPING A NEW MATERIAL FOR MEMS: AMORPHOUS DIAMOND. J.P. Sullivan, T.A. Friedmann, M.P. de Boer, D.A. LaVan, R.J. Hohlfelder, C.I.H. Ashby, M. Mitchell, R.G. Dunn, Sandia National Laboratories, Albuquerque, NM; A.J. Magerkurth, Cornell Univ., Ithaca, NY.

Silicon is often the MEMS material of choice due, in large part, to the extensive infrastructure developed to support this material. In many instances, alternative materials possess superior mechanical, chemical, physical, or tribological properties, but most of these materials have obstacles for their widespread application. In this talk, we explore the potential and challenges encountered with one new surface micro-machined MEMS material, amorphous diamond (a-D). This material possesses many advantages over silicon, including a naturally hydrophobic surface that resists stiction, excellent wear resistance, chemical inertness, high fracture strength (> 8 GPa), and bio-compatibility (e.g., for bioMEMS). In addition, the material possesses a two-phase microstructure (a nanophase mixture of 4-fold and 3-fold coordinated carbon) that permits a host of exotic properties, such as plastic-like deformability while maintaining an ultra-high elastic modulus, > 700 GPa, a tunable stress state from compressive to tensile, and widely tunable electrical conductivity. Unfortunately, the requisite deposition process (pulsed laser deposition or cathodic arc deposition) does not permit conformal deposition. This obstacle is overcome by redesigning the standard multi-layer MEMS process that was developed for conformal poly-Si deposition. The structures, solutions, and trade-offs with this approach are described. The complete cycle from basic materials science to MEMS fabrication is described for this material and serves to illustrate the key issues which need to be addressed for any replacement MEMS material.
Sandia is a multiprogram laboratory operated by Sandia Corp., a Lockheed Martin Co., for the U.S. Dept. of Energy under contract DE-AC04-94AL85000.

11:00 AM *EE7.2
CERAMIC MICROMACHINING TECHNOLOGY – SENSORS TO COMBUSTORS. Mark Allen, Georgia Tech, Dept. of Electrical and Computer Engineering, Atlanta, GA.

ABSTRACT NOT AVAILABLE

11:30 AM EE7.3
PREPARATION OF MESOPOROUS OXIDES FOR MEMS STRUCTURE. Jong-Ah Paik, Nobuaki Kitazawa, Bruce Dunn, University of California at Los Angeles, Dept of MS&E; Shih-Kang Fan, C.J. Kim, University of California at Los Angeles, Dept of Mechanical and Aerospace Engineering, Los Angeles, CA; Ming C. Wu, University of California at Los Angeles, Dept of Electrical Engineering, Los Angeles, CA.

The high porosity and extremely regular pore size provided by mesoporous films offer interesting opportunities for MEMS device structures. This paper concerns the development of mesoporous films for MEMS torsion mirrors. The resonant frequency of such mirrors is determined by a number of parameters including the density of the mirror material. By decreasing the density, it is possible to increase the resonant frequency of the mirror structure, leading to better vibration and shock resistance than with conventional silicon or polysilicon mirrors. Mesoporous silica films were deposited by spin coating. The coating solution consisted of tetraethoxysilane, water, ethanol and an amphiphilic poly (alkylene oxide) block copolymer (Pluronic F127 from BASF) as the structure-directing agent. After coating, the film were heated in air to 400°C to remove the block copolymer and stabilize the silica network. The resulting mesoporous film exhibited an average pore diameter of 7 nm and was about 70% porous with a very narrow pore size distribution. The root mean square roughness of the film was determined to be 3nm and gold films deposited on the mesoporous oxide retain their optical reflectivity. The mesoporous films have been subjected to standard photo-lithographic processes along with wet dry etching and seem to integrate quite well with MEMS fabrication methods. Mesoporous alumina and titania have also been prepared by the block copolymer route and results with these materials will be presented.

11:45 AM EE7.4
SILICON-GERMANIUM EPITAXY: A NEW MATERIAL FOR MEMS. Jeffrey T. Borenstein, Nicole D. Gerrish, Robert White, Charles Stark Draper Laboratory, Cambridge, MA; Matthew Currie, Eugene Fitzgerald, Department of MS&E, MIT, Cambridge, MA.

A wide array of materials have been investigated as candidate fabrication templates for precision microelectromechanical structures, including boron-diffused silicon, boron-doped epitaxial silicon, polysilicon, silicon-on-insulator, and wafer-thick bulk structures. Here we present the latest fabrication results for epitaxial silicon-germanium alloys, a new class of materials which possess excellent crystalline structure, are compatible with non-toxic etchants in bulk micromachining, and are capable of on-chip integration with electronics. For MEMS applications, silicon-germanium alloy layers are grown using a graded buffer approach, resulting in very high quality micromachined structures. Very low defect densities are obtained through the use of these relaxed buffers. Original etch-stop studies determined that Ge doping provided a very weak selectivity in anisotropic etchants such as KOH and EDP. However, by extending the range of Ge concentration to over 20%, we have found extremely high etch selectivities in a variety of etchants. Unlike boron-doped layers, SiGe exhibits etch stop characteristics in the non-toxic, process compatible solution TMAH. The combination of independence from boron doping concentration and etchant compatibility make SiGe a material which is ideal for integration with on-chip electronics. In this work we present the latest fabrication data on comb-drive resonators built using SiGe epitaxial layers. Process compatibility issues related to wafer curvature, surface finish and reactive-ion-etching chemistries are addressed. An unexpected result of the fabrication process, curvature of released structures, is resolved by annealing wafers after the SiGe deposition. Changes in Young's modulus arising from the high atomic fraction of Ge in the device can be determined by simple beam analysis based on observed resonant frequencies. The quality factor of the motor drive resonance is determined by operation of the gyroscopes in vacuum. Overall, build precision for these devices is excellent. We conclude by addressing the remaining challenges for wide-scale implementation of silicon-germanium epitaxial MEMS.

SESSION EE8: OPTICAL MICROSYSTEMS AND PROCESSING TECHNIQUES I
Chair: Arturo A. Ayon
Tuesday Afternoon, November 28, 2000
Room 206 (Hynes)

1:30 PM *EE8.1
MECHANO-OPTICAL MICROSYSTEMS. Paul V. Lambeck, MESA Research Inst, Univ of Twente, Enschede, THE NETHERLANDS.

Monolithical integration of integrated optical and micromechanical subsystems offers the prospect of small robust microsystems with a large variety of functionalities. On the one hand the integrated optical part can be used for the read out of the position and hence also of the movement of micromechanical components such as membranes and cantilevers. On the other the micromechanical sub-system can be utilized for modulating the properties of the integrated optical sub-system as is applied e.g. in switching matrices for the optical telecommunication. Several principles used in these mechano-optical MOEMS- systems will be highlighted. The monolithical integration requires the compatibility of the technologies needed for realising the micromechanical and optical part and this sets limits to the materials, that can be applied whithin these systems. A promising approach is to fabricate in a first step both individual sub- systems and to bond them together in in a second step. This can be well done e.g. by aligned waferbonding, if both the micromechanical and the integrated optical subsystem are made by Silicon- based technologies, the optical part e.g. being realised bij a (doped) Silicondioxide technology or a Siliconoxynitride technology. Special attention will be paid to the latter technology and to the additional steps needed for combining both sub-systems. Some monolithically integrated opto-mechanical systems will be treated.

2:00 PM EE8.2
RAPID PROTOTYPING OF MICROMAGNETIC SYSTEMS FOR MANIPULATION OF MAGNETIC BEADS. Tao Deng, George M. Whitesides, Harvard Univ., Dept. of Chemistry and Chemical Biology, Cambridge, MA; Gary Zabow, Mala Radhakrishna, Mara Prentiss, Harvard Univ., Dept. of Physics, Cambridge, MA.

Current-carrying circuits fabricated using soft lithography are used to create strong magnetic field gradients for controlling the position and/or velocity of paramagnetic microbeads in aqueous suspension. Such paramagnetic beads are widely used to manipulate or sort biological material that can be selectively attached to the surface of the beads. We show that the beads can be captured from solution, held in a fixed position, and released at will. We also show that the beads can be moved along complicated paths and that simply changing the current flowing in the circuits can dynamically reconfigure the paths. The selective capture, transport, and confinement of magnetic beads using microfabricated circuits should expand current sample handling capabilities, and can be used with or without the presence of net fluid flow and microfluidic channels.

2:15 PM EE8.3
A ROBUST CO-SPUTTERING FABRICATION PROCEDURE FOR TiNi SHAPE MEMORY ALLOYS FOR MEMS. B.-K. Lai[1], H. Kahn[1], G. Hahm[2], L. You[2], S.M. Phillips[2] and A.H. Heuer*, [1]Dept of MS&E, [2]Dept of Electrical Engineering and Computer Science, Case Western Reserve University, Cleveland, OH.

Co-Sputtering has been used to fabricate equiatomic thin films of TiNi, a shape memory alloy, which form the basis of microactuators with many applications in MEMS. The difficulties involved in obtaining equiatomic TiNi thin films with high transformation temperatures, and a robust procedure suitable for batch fabrication in a production environment, will be described. The performance of the TiNi actuators has been characterized, with regards to actuation force, recovery strain, switching speed, and fatigue resistance. The performance of microfluidic devices using these actuators will also be presented.

2:30 PM EE8.4
PROCESSING OF THICK DIELECTRIC FILMS FOR POWER MEMS: STRESS AND FRACTURE. Kuo-Shen Chen, National Cheng-Kung University, Tainan, TAIWAN; Xin Zhang, Massachusetts Institute of Technology, Cambridge, MA; Reza Ghodssi, University of Maryland, College Park, MA; S. Mark Spearing, Massachusetts Institute of Technology, Cambridge, MA.

Thick dielectric films for high power MEMS-based devices are required in order to reduce capacitive loading and enhance surface charge density. However, such thick deposited layers often result in severely bowed wafers while deleterious effects of residual stress impose major restrictions on the device performance. The present work is focused on the characterization of residual stress and modeling of fracture behavior of dielectric films. 10-30 microns silane or tetraethylorthosilicate (TEOS) based PECVD oxide films were deposited using a Novellus^{TM} Concept One System. To counteract the high compressive stress inherent to the oxide film, a thin layer (less than 1 micron) of highly tensile silicon nitride film was deposited by LPCVD, either prior to deposition of the oxide film, or after the thick oxide deposition, or sandwiched between two oxide layers. The motivation for this work is to elucidate the factors contributing to residual stress in thick films and to optimize the deposition process so as to reduce wafer bow and to avoid cracking. Deformation of dielectric films between room temperature and 500°C was measured by a wafer curvature measurement system. The corresponding residual stress, at different stages of dielectric film deposition, was calculated by laminated plate theory as a function of temperature. Based on the nominal material properties, in situ intrinsic stress was then determined by extracting thermal stress component. Fracture toughness of nitride films was measured using a microindentation system while scratch resistance was measured using a nanoindentation system. Surface composition of oxide films was examined using SIMS and RBS. Dissolved gases were found to play an important role in governing intrinsic stress. Both diffusion-reaction and constrained sintering models were constructed to characterize the bulk transportation of hydrogen in order to calculate the gradient of intrinsic stress in the films. Fracture mechanics was applied to analyze the crack growth and propagation in the thick dielectric films and the critical temperature (or critical film thickness) needed to avoid cracking was further predicted. Finally, optimized dielectric films were tabulated which exhibited less and even zero wafer bow for a specific combination of film thickness.

2:45 PM EE8.5
MICROBRIDGE TESTING OF SILICON OXIDE/SILICON NITRIDE BILAYER. Cai Fu Qian, Yan-Jing Su, Ming-Hao Zhao and Tong-Yi Zhang, Department of Mechanical Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong, CHINA.

The present work further develops the microbridge testing method to characterize mechanical properties of bilayer thin films. We model the substrate deformation with three coupled springs and consider residual stress in each layer to formulate deflection versus load under large deformation, resulting in a closed-form formula. If the mechanical properties of one layer are available, the closed formula is able to simultaneously evaluate the Young's modulus and residual stress of the other layer and the bending strength of the bilayer films from the microbridge test. The analytic results are confirmed by finite element calculations. Using a load and displacement sensing nanoindenter system equipped with a microwedge probe, we conduct microbridge tests on low-temperature silicon oxide/silicon nitride bilayer films prepared by the microelectromechanical technique. The experimental results verify the proposed method. The results show that for small deformation, the load-deflection relationship is approximately linear. Under large deformation, bridge deflection couples with the neutral plane stretch such that both the tensile-equivalent and bending-equivalent Young's moduli are involved. Under small deformation, one may use the bending- equivalent Young's modulus and the residual force, or the tensile-equivalent residual stress to reduce the bilayer bending problem to that of single-layer beams. For silicon substrates, the substrate deformation plays a very important role in extraction of the elastic constant and the residual stress from the microbridge test under either large or small deformation.
Supported by the Hong Kong Research Grants Council.

SESSION EE9: PROCESSING TECHNIQUES II
Chair: Carol Livermore
Tuesday Afternoon, November 28, 2000
Room 206 (Hynes)

3:30 PM *EE9.1
UNDERSTANDING AND CONTROLLING WET CHEMICAL ETCHING OF SILICON. M. Elwenspoek[1], W.J.P. van Enckevort[2], J.G.E. Gardeniers[1], A.J. Nijdam[1], J. van Suchtelen[1], E. van Veenendaal[2], E. Vlieg[2]. [1]University of Twente, MESA Insitute, Enschede, THE NETHERLANDS. [2]University of Nijmegen, Nijmegen, THE NETHERLANDS.

In this paper we review the progress that has been made during the past 5-6 years to understand the physical and chemical processes which underlie wet chemical etching of single crystals, in particular silicon. The etch rate of single crystalline silicon depends strongly on the crystallographic orientation in certain etching solutions (such as aqueous KOH solutions, hydrazine) with a deep etch rate minimum in the <111> direction. It is by now well established that the minimum is due the fact that the {111} plane of silicon etches via a step mechanism: atoms are removed at edges of steps of a height of one or more atoms or more which are abundant on {111} faces with a slight misorientation. New steps must nucleate on perfectly oriented {111} faces. From the chemical point of view the most important insight was that the {111} is hydrogenated during etching in KOH, and that at the rest potential the number of electrons exchanged during etching is very small: chemical etching dominates. It is thought now that the water molecule reacts with silicon, the hydoxile ion being a catalisator. Once having accepted this basic fact, a great number of phenomena can be understood. We sum a few: (i) Steps can only exist on faces below the roughening transition. Monte Carlo simulations showed indeed that a crystal face stays rough on an atomic scale even at infinitely large understaturation - which is thought to be equivalent to the etching situation. The {110} being as S - face, it is rough and etches fast, although each atom in the flat face has three back bonds. (ii) New steps on the {111} face must nucleate. Nucleation centers seem to be the outcrops of edge dislocations. Since edge dislocations grow - themselves nucleated at oxide precipitates - by thermal treatment, {111} faces appear rougher (on a macroscopic scale), showing craters centered around edge dislocations. This has consequences of technological relevance: The crystal front adjacent to masks become stepped themselves. (iii) Nucleation of steps can be enhanced at the interface between a mask and the crystalline material. Measuring etch rate and its anisotropy using a wagon wheel patters may give misleading results. (iv) Hillocks of pyramidal form and roof like structures which appear under circumstances on {001} and {110} oriented surfaces, respectively, can be understood by the step flow together with a mechanism which hinders the etching of the atoms at the tip of the structures. (v) Step flow allows us to calculate the etch rate as a function of the orientation using physically transparent parameters which, in principle, can be determined independently.

4:00 PM EE9.2
LOW TEMPERATURE Si DIRECT BONDING BY PLASMA ACTIVATION FOR FABRICATION OF MEMS DEVICES. Yonah Cho, Department of Materials Science and Engineering, University of California, Berkeley, Berkeley, CA; Nathan W. Cheung, Department of Electrical Engineering and Computer Sciences, University of California, Berkeley, Berkeley, CA.

Waferscale Si direct bonding is a critical process in the fabrication of cavities, microchannels and multi-level-microstructures commonly used in MEMS applications. Direct bonding elimiates needs for the application of intermediate layers and external forces such as electric field used in anodic bonding. When done at a low temperature, Si direct bonding can further place itself as the most robust and device compatible bonding process for MEMS applications. However, the first reported Si direct bonding by Lasky[1] and Shimbo et al. [2] required post-bonding thermal annealing above 1000°C due to initially weak room temperature bonding. The first single crystal Si resonant pressure sensors demonstrated by Petersen[3], also, utilized high temperature Si fusion bonding. High temperature bonding or annealing is detrimental to today's microsystems in terms of thermal mismatch and electrical degradation in metallized device structures. Recently, chemical and dry surface activation methods have been demonstrated. In this paper, plasma activated low-temperature Si direct bonding is presented. Dramatic improvement of bonding strength after exposure to oxygen plasma is measured by a crack opening method[4]. Post-bonding annealing as low as ~100°C was sufficient to acheive full bonding strength. Bonding activation energy is investigated and surface chemical analysis is utilized to study the enhanced bonding phenomenon.

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4:15 PM EE9.3
MEMS MATERIALS AND FABRICATION TECHNOLOGY FOR LARGE AREAS: THE EXAMPLE OF AN X-RAY IMAGER. Jurgen H. Daniel, Brent Krusor, Marcelo Mulato, Raj Apte, Robert A. Street, Xerox Palo Alto Research Center, Palo Alto, CA; Adela Goredema, Peter M. Kazmaier, Xerox Research Center Canada, Mississauga, Ontario, CANADA.

Micromachining has potential applications for large area image sensors and displays, but conventional MEMS technology, based on crystalline silicon wafers cannot be used. Instead, large area devices use deposited films on glass substrates. This presents many challenges for MEMS, both as regards materials for micro-machined structures and the integration with large area electronic devices. We are exploring the novel thick photoresist SU-8, as well as plating techniques for the fabrication of large area MEMS. As an example of its application, we have applied this MEMS technology to improve the performance of an amorphous silicon based image sensor array. SU-8 is explored as the structural material for the X-ray conversion screen and as a thick interlayer dielectric for the thin film readout electronics of the imager. Medical X-ray imagers have a thick (200-500 micron) layer of phosphor, which converts the X-rays into visible light, placed directly on top of the photodiodes. Spatial resolution is limited because of light scattering in the phosphor. In order to obtain the full resolution of the pixel array the phosphor layer needs to be micro-patterned into cells which collimate the generated light. This cell structure was patterned with a 300-400 micron thick layer of SU-8. The SU-8 cell walls need to be made reflective in order to prevent light from scattering into a neighboring cell which is achieved by sputtering a thin metal layer. Subsequently the cells are filled with a phosphor. A second application is the use of SU-8 as a thick interlayer dielectric to reduce noise due to capacitive coupling in the thin film electronic circuit of the imager. Nickel electroplating is used to metallize the deep contact vias. The compatibility of SU-8 with thin film deposition methods, such as amorphous silicon PECVD, will be discussed. Processing challenges which are particularly important for large area fabrication, will be addressed.

4:30 PM EE9.4
MICROPATTERNING OF CERAMICS ON SUBSTRATES TOWARDS GAS SENSOR APPLICATIONS. M. Heule, L. Meier, L.J. Gauckler, ETH Zurich, Dept of Materials, Zurich, SWITZERLAND.

Many research efforts are made to widen the range of materials into the fabrication of Microelectromechanical Systems (MEMS). Ceramic powders in gas sensor fabrication facilitate the use of a wide range of complex oxide compositions compared to gas phase processes such as chemical (CVD) and physical vapour deposition (PVD).
Miniaturization of thick film gas sensors offers advantages like the lower heating power consumption. Transition metal-doped tin oxide is widely known for its use as gas sensitive material in both thick and thin film sensor technologies.
We used conventional as well as soft lithography, namely micro-molding in capillaries (MIMIC) for the fabrication of micro-structured ceramics on different substrates. Colloidal dispersions of fine ceramic powders were successfully applied in MIMIC similar to techniques using metal-organic ceramic precursors. The attained feature size strongly depends on the powder size but on the preparation of the colloidal suspension as well. We applied these techniques to produce ceramic micro-lines of tin oxide. Using 0.25 micron oxide powder, structures of 10 microns were well reproduced. A method to apply electric wiring to micro-structured ceramic parts was developed in order to get a functional device. Our sensors cover an area of only 60x80 square-microns including 4-point mode platinum circuit lines. Sensor responses to hydrogen gas concentrations of a few ppm were recorded.

4:45 PM EE9.5
THE MECHANICAL PROPERTIES OF POLYCRYSTALLINE SILICON CARBIDE FILMS DETERMINED USING BULK MICROMACHINED DIAPHRAGMS. Shuvo Roy, The Cleveland Clinic Foundation, Department of Biomedical Engineering, Cleveland, OH; Christian A. Zorman, Mehran Mehregany, Case Western Reserve University, Department of Electrical Engineering and Applied Physics, Cleveland, OH.

Polysilicon is a dominant material in the fabrication of micro-electromechanical systems (MEMS); however, its mechanical properties begin to degrade at elevated temperatures (>350°C), making it increasingly unsuitable for high-temperature applications. In contrast, SiC is well known for its mechanical hardness, chemical inertness, high thermal conductivity, and electrical stability at temperatures well above 600°C. These properties make SiC an attractive alternative for MEMS in harsh environments, yet relatively little is known about its thin film mechanical properties. This paper presents the results of a study comparing the Youngs modulus, residual stress, and burst strength of polycrystalline SiC (polySiC) films grown on as-deposited (610°C) and annealed (1280°C) polysilicon substrates. XRD and TEM are used to characterize the microstructure of the polySiC films. PolySiC films grown on as-deposited polysilicon are highly textured (110) films, while polySiC films grown on annealed polysilicon have a mixed texture comprised of (110) and (111) grains. Youngs modulus and residual stress are determined using an interferometric load-deflection measurement apparatus on suspended polySiC diaphragms, which are fabricated by bulk micromachining. The load-deflection data are analyzed using a least squares curve-fitting technique to extract Youngs modulus and residual stress. The value of Youngs modulus of polySiC depends on grain microstructure, averaging ~350 GPa for highly textured (110) films with columnar grains to ~470 GPa for equiaxed films with a mixture of (111) and (110) grain orientations. The residual stress of polySiC is independent of grain microstructure and averages ~440 MPa. Burst strength is determined using a combination of finite element analysis and burst pressure measurements of the suspended diaphragms. The burst strength values of polySiC diaphragms range from ~1320 GPa for films with a relatively high grain boundary density to ~1720 GPa for films with a relatively low grain boundary density.