

SYMPOSIUM MM

Materials Science of Microelectromechanical Systems (MEMS) Devices II

November 28 – December 1, 1999

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* Invited paper

TUTORIAL

FTm: POLYCRYSTALLINE SILICON AND SILICON CARBIDE AS MATERIALS FOR MEMS

Sunday, November 28, 1999
9:00 a.m. - 4:00 p.m.
Room 201 (H)

The first half of this tutorial is designed to teach newcomers to the field as well as those with a basic familiarity with integrated circuit manufacture about the emerging field of Microelectromechanical Systems (MEMS). Both manufacturing technologies for these devices and examples of sensor and actuator devices, including videos and sample demonstrations, will be presented. Integration issues with microelectronics will be addressed, and a brief overview of other MEMS manufacturing technologies will be included. With the basics of surface micromachining established, the second half of the tutorial will introduce silicon carbide as a new material for high-temperature and hard-coating applications in MEMS. Processing, packaging, and applications of SiC will be discussed.

Instructors:

Jeffrey Sniegowski, Sandia National Laboratories
Stephen Montague, Sandia National Laboratories
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SESSION MM1: DEPOSITION AND CHARACTERIZATION OF SILICON I
Chair: Arthur Heuer
Monday Morning, November 29, 1999
Room 313 (H)

8:30 AM *MM1.1

BUILT-IN STRAIN IN POLYSILICON: MEASUREMENT AND APPLICATION TO SENSOR FABRICATION. **H. Guckel**, University of Wisconsin - Madison, Dept. of Electrical and Computer Engineering, Madison, WI.

Surface micromachined sensors profit from the availability of low pressure vapor deposited polysilicon films with isotropic mechanical properties. Thin films of this type are normally grown under LPCVD conditions which nearly produce amorphous material and, for want of a better term, are called fine grained. The films typically exhibit compressive built-in strain fields. Since the strain level and its uniformity resolved to intended device dimensions have a first order influence on sensor performance simple strain measurements which are independent of other mechanical properties are necessary for process engineering as well as process monitoring. Concerns of this sort led to the use of Euler buckling of clamped-clamped, surface micromachined polysilicon beams versus beam length as a suitable method for strain measurement. This very simple technique led to the discovery that proper anneal cycles of the polysilicon films convert compressive strain to tensile strain. This result which has major beneficial implications on sensor construction techniques led to Euler buckling structures for tensile strain. The two major application areas for strain controlled polysilicon are pressure transducer fabrication and resonator structures. The classic pillbox-piezoresistive pressure transducer benefits from the use of predetermined tensile strain because flat, large area diaphragms can be manufactured with good yield. Mechanically resonant devices such as clamped-clamped beams in a vacuum shell require processing procedures in which the resonant member cannot touch the silicon substrate or the upper polysilicon shell during processing to avoid sticking problems. The use of tensile strain stiffens the beam and facilitates resonator manufacturing. Operation of resonators in long term testing situations provides drift data for the mechanical properties of polysilicon which yield the conclusion that the material is indeed very stable.

9:00 AM MM1.2

MICROINSTRUMENT FOR THE CHARACTERIZATION OF SUBMICRON SILICON FIBERS AT HIGH STRAIN. **J.M. Chen**, B.W. Reed, Cornell Univ, Dept of Applied Physics, Ithaca, NY; N.C. MacDonald, Cornell Univ, Dept of Electrical Engineering, Ithaca, NY.

We have developed a MicroElectroMechanical System (MEMS) to study the electrical and mechanical properties of nanometer-scale single crystal silicon fibers at elastic strains near the ideal fracture limit of silicon. With this new micro-instrument "on-a-chip", we have applied greater than 1% strain to the silicon nano-fiber at room temperature without fracture, and have measured resistivity change greater than 23% for the [110] oriented silicon sample under these strains. Occupying an area less than 1mm X 1mm, this microinstrument consists of a high aspect ratio silicon actuator connected to a single crystal silicon fiber with a diameter ranging

from 20-200nm¹ and a length of up to 100 μ m. The fiber and actuator are carved from a single silicon wafer and require no assembly at any step of the process. Thus our system inherently provides high quality electrical and mechanical connection to nanometer-scale silicon samples. Recently published results have shown an increase in the fracture strength of silicon samples miniaturized by micromachining technology^{2,3,4,5}. Until now, the dimensions were not reduced below the micrometer-scale, largely because of the difficulties involved in reliably attaching high force actuators to samples of this size. Similar difficulties have impeded electrical measurements of silicon at high strain. Our monolithic nano-fiber and actuator system solves these problems by integrating all components on a single wafer: the nano-scale silicon fiber, actuator and interconnects are all fabricated simultaneously from the silicon substrate, without any assembly. We will present the microinstrument and discuss our strain and piezoresistivity data. ¹Fabrication and STEM/EELS Measurements of Nanometer-scale Silicon tips and Filaments B. W. Reed, J. M. Chen, N. C. MacDonald, J. Silcox, and G. E. Bertsch, accepted to Phys. Rev. B ²Deformation and Fracture of Small Silicon Crystals G. L. Pearson, W. T. Read Jr., and W. L. Feldmann, Acta Metallurgica, vol. 5, pp181-191, 1957 ³Measurement of Forces and Spring Constants of Microinstruments M.T.A. Saif and N.C. MacDonald, Rev. Sci. Instrum. vol 69 (3), March 1998, pp 1410-1422 ⁴Microinstruments for Submicron Material Studies M.T.A. Saif and N.C. MacDonald, J. Material Research, vol. 13, no. 12, pp. 3353-3356 ⁵Tensile Testing of Silicon Film Having Different Crystallographic Orientations Carried out on a Silicon Chip K. Sato, T. Yoshioka, T. Ando, M. Shikida, T. Kawabata, Sensors and Actuators, vol. A70, pp148-152

9:15 AM MM1.3

TESTING OF CRITICAL FEATURES OF POLYSILICON MEMS. D.A. LaVan and **T.E. Buchheit**, Sandia National Labs, Albuquerque, NM.

The behavior of MEMS devices is limited by the strength of critical features such as thin ligaments, oxide cuts joining layers, pin joints and hinges. Devices fabricated at Sandia's Microelectronic Development Laboratory have been successfully tested to investigate these features. A series of measurements were performed on samples with gage lengths of 15 to 1000 microns, as well as samples that include the critical features of standard components. Specimens have a freely moving pin joint on one end that anchors the sample to the silicon die to allow rotation to reduce effects of bending. Each sample is loaded in uniaxial tension by pulling laterally with a flat tipped diamond in a computer-controlled Nanoindenter. Load is calculated by resolving the measured lateral and normal forces into the applied tensile force and frictional losses. The stress-strain curve of tensile samples is determined with specimen cross section and gage length dimensions verified by measuring against a standard in the SEM. Multiple tests can be programmed at one time and performed without operator assistance at a rate of 20 per day allowing the collection of significant populations of data. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

SESSION MM2: DEPOSITION AND CHARACTERIZATION OF SILICON II
Chair: Eric Peeters
Monday Morning, November 29, 1999
Room 313 (H)

10:00 AM MM2.1

WAFER-LEVEL STRENGTH TESTING OF POLYSILICON MEMS DEVICES. **H. Kahn**¹, N. Tayebi², R. Ballarini³, R.L. Mullen³ and A.H. Heuer¹, Case Western Reserve University; ¹Dept of Materials Science and Engrg, ²Dept of Mechanical and Aerospace Engineering, ³Dept of Civil Engrg, Cleveland, OH.

The mechanical properties of MEMS materials are often dependent on the micromachining techniques used. Therefore, measurements made at larger size scales cannot be applied to MEMS structures; the measurements must be made using MEMS devices. We have previously reported MEMS devices for measuring the fracture strength of notched polysilicon specimens, as well as sharp-cracked polysilicon specimens which were etched in two steps with an intermediate high temperature anneal. We can extend this wafer-level testing technique to measure the tensile strength and fracture toughness of polysilicon etched in a single step. Because the devices are fabricated in a single-mask process and the experiment takes place entirely on-chip using only standard electrical testing equipment, this technique can be easily transferred to any fabrication facility, for fast verification of materials properties. This technique can also be extended to other MEMS materials.

10:15 AM MM2.2

DEPOSITION AND CHARACTERIZATION OF IN-SITU BORON DOPED POLYCRYSTALLINE SILICON FILMS FOR MICROELECTROMECHANICAL SYSTEMS APPLICATIONS. J. Jay McMahon¹, Jeffrey M. Melzak¹, Christian A. Zorman¹, Juyong Chung², Mehran Mehregany¹; Case Western Reserve University, Cleveland OH; ¹Department of Electrical Engineering and Computer Science, ²Department of Materials Science and Engineering.

In an effort to develop thick, p-type polycrystalline silicon (polysilicon) films for microelectromechanical systems (MEMS) applications, undoped and in-situ boron-doped polysilicon films were grown in an RF inductively heated, cold wall atmospheric pressure chemical vapor deposition (APCVD) reactor at temperatures ranging from 625°C to 850°C. The deposition recipe uses silane as the precursor gas, argon as the carrier gas, and diborane as the doping gas. This process is shown to yield a deposition rate of 27 nm/min for undoped films grown at 625°C, more than three times the rate achieved by conventional low pressure chemical vapor deposition (LPCVD) of undoped polysilicon at 615°C. In-situ doping of the films with boron enhances the deposition rate to 34 nm/min at 625°C. As the temperature is increased, the deposition rate of boron doped films is further increased to 73 nm/min at 850°C. Sheet resistance of in-situ boron doped films is within an order of magnitude of in-situ phosphorus doped LPCVD polysilicon films; spreading resistance profiles show that the doping level is constant throughout the thickness of the film at a level of 2×10^{19} carriers/cm³. Residual film stress was monitored using a wafer curvature measurement system. Doped films grown at low temperatures exhibit compressive stress as high as 650 MPa, however, films grown at 850°C exhibit (compressive) stress as low as 130 MPa. This stress is somewhat higher than in-situ phosphorus doped LPCVD films, which can be deposited with compressive stress of about 30 MPa. Mechanical properties were further evaluated by fabricating and testing surface micromachined strain gauges, singly and doubly supported cantilevers, as well as straight and folded beam resonators in 5.0 micron-thick doped films. The extended paper will detail the deposition process, XRD and TEM characterization of the as-deposited films, and the results of the electrical and mechanical properties study.

10:30 AM MM2.3

CONTROL OF RESIDUAL STRESSES IN AS-GROWN POLYSILICON BY MULTI-LAYER DEPOSITION. Jie Yang, Hal Kahn, Stephen Phillips*, Arthur A. Heuer, Case Western Reserve Univ, Dept. of Materials Science and Engineering, Cleveland, OH; *Case Western Reserve Univ, Dept of Electrical Engineering and Computer Science, Cleveland, OH.

LPCVD polysilicon thin films deposited between 550 and 600 degrees C have an equiaxed microstructure (resulting from crystallization of an initially amorphous deposit) and contain ~200 MPa residual tensile stresses after deposition, whereas polysilicon films deposited above 600 degrees C have a columnar microstructure and contain ~300 MPa residual compressive stresses after deposition. Both types of films also contain stress gradients. We have grown films containing multiple layers of polysilicon by cycling the growth temperature between 570 and 615 degrees C. The multilayer films thus formed are comprised of alternating tensile and compressive layers, and by control of the thickness of the individual layers, the overall stress of the polysilicon can display any value between that of the tensile layer and that of the compressive layer. We have focussed on producing films with zero overall residual stress, as measured by wafer curvature, and have characterized the microstructures by transmission electron microscopy (TEM) and X-ray diffraction (XRD). Because of the stress gradients present in both layers, devices made from films with zero overall residual stress may show distortions after release. We have measured the stress gradients in each type of layer and can design films with zero overall stress and zero overall stress gradients. It is also possible to design films with zero overall stress but with controlled curvature upon release.

10:45 AM MM2.4

HIGH-RESOLUTION MEASUREMENT OF CRACK GROWTH IN MICRO-MACHINED SINGLE CRYSTAL SILICON. Alissa M. Fitzgerald, Dept. of Aeronautics and Astronautics, Reinhold H. Dauskardt, Dept. of Materials Science, Thomas W. Kenny, Dept. of Mechanical Engineering, Stanford University, Stanford, CA.

Time dependent sub-critical cracking associated with environmental species such as moisture may have significant implications for the reliability of MEMS devices made of silicon. However, the existence of such stress corrosion phenomena in silicon remains controversial. Sub-critical crack-growth behavior in brittle materials is commonly characterized using crack velocity versus applied stress intensity curves (v-K curves). Crack velocity is inferred by curve-fitting crack length versus time data taken at low sample rates (<100 Hz) under the assumption that crack growth is a continuous process. However,

we have observed discrete crack growth behavior in a compression-loaded double cantilever beam sample of micro-machined proportions. The samples are fabricated from (100) single crystal silicon wafers. A thin film resistor sputtered onto the sample surface using a lithographic technique is used to directly measure crack extension. The crack growth in all samples is characterized by periods of rapid crack growth interspersed with long periods of arrest in which no evidence of sub-critical cracking was observed. High speed data acquisition (up to 1 GHz) was performed to accurately measure crack velocities as high as 4.2 km/s during these rapid growth periods. Likely reasons for the bursts of rapid fracture are considered and implications for the existence of a stress corrosion process in silicon are discussed.

11:00 AM MM2.5

STRUCTURAL MECHANICS OF MICRO-MACHINED STRUCTURES. O.L. Warren, A. Daugela, D.A. Crowson, L.L. Kuhn, T.J. Wyrobek, Hysitron Inc., Nanomechanics Research Laboratory, Minneapolis, MN.

The micro-scale dimensions of micro-machined structures in MEMS devices necessitate holding very stringent absolute dimensional tolerances during manufacturing. To make matters worse, the intended mechanical response of such structures often depends critically on the smallest dimension. In this study, we have evaluated the structural mechanics of commercially available micro-machined cantilever structures using nanoindentation instrumentation augmented by AFM-type topographical imaging capabilities. The sub-microNewton force sensitivity and the sub-nanometer displacement resolution of cutting-edge nanoindentation instrumentation are well suited for this application. Results of quasi-static loading experiments are consistent with the broad range of spring constants reported by the manufacturer. Load-unload cycles starting from air reveal rate-dependent attractive interactions upon initial contact and final tip removal, attributable to the rheological properties of the newly formed meniscus. The measured shift in the resonance frequency during dynamic testing is consistent with the quasi-statically-measured cantilever spring constant adding to the stiffness of the nanoindentation transducer, as would be expected for a parallel spring system. However, an additional softening mode exhibiting strong phase lead is observed at a frequency below the strongly coupled resonance. The origin of this mode can be traced to the non-linear behavior of weak coupling. The scope of using nanoindentation techniques toward evaluating the integrity of MEMS manufacturing processes will also be presented.

11:15 AM MM2.6

THE INFLUENCE OF WATER ON THE FRACTURE BEHAVIOR OF SINGLE-CRYSTAL SILICON MICROELEMENTS UNDER FATIGUE LOADING. Kohji Minoshima, Kyoto Univ, Dept of Mechanical Engineering, Kyoto, JAPAN; Tomota Terada, Nomura Research Institute, JAPAN; Kenjiro Komai, Kyoto Univ, Dept of Mechanical Engineering, Kyoto, JAPAN.

Simple bending fatigue tests were performed in single-crystal silicon microelements fabricated by photoetching. The tests were conducted in smooth microcantilever beam samples and notched ones. A focused ion beam was used to machine a sub-micron deep notch. An adequate machining condition was investigated, and smooth notches could be successfully machined. The radius of the curvature of the notch ranged from about 20 nm to 100 nm, and it decreased with an increase in notch depth. Fatigue tests were conducted in laboratory air and in pure water at a stress cycle frequency of 0.1 Hz, by using a specially designed fatigue testing machine for microelements, reported elsewhere. In laboratory air, no fatigue phenomenon was observed, and the strength under fatigue loading was determined by the quasi-static strength of the microelement. Beside this, nanoscopic fatigue damage, such as extrusion and inclusion observed in fatigue sensitive materials, could not be observed in single-crystal silicon microelements by using an atomic force microscope (AFM). However, in pure water, the fatigue lives decreased with an increase in immersion duration in pure water. In pure water, synergistic effects of water and dynamic loading caused a nanoscopic crack, which was crystallographically oriented. The final catastrophic failure of the microelement under fatigue loading was caused by the stress concentration induced by the nanoscopic crack, and the fracture mechanism was discussed from the nanoscopic points of view.

11:30 AM MM2.7

PROPERTIES OF LOW RESIDUAL STRESS SILICON OXYNITRIDES USED AS AN ETCH RELEASE LAYER. S. Habermehl, A.K. Glenzinski and J.J. Sniegowski, Sandia National Laboratories, Albuquerque, NM.

For microelectromechanical systems (MEMS) based on polysilicon surface micromachining, silicon dioxide deposited from tetraethoxysilane (TEOS) is commonly used as an etch release layer

between mechanical components. For the relief of mechanical stress in polycrystalline Si (poly-Si) components, high temperature anneals (> 1200 K) are very effective. Unfortunately, this temperature cycling does not stress relieve the oxide layers, and in practice it drives the oxide into a high state of compressive residual stress (~ -250 MPa). This level of residual stress can lead to excessive wafer bow that introduces wafer handling problems and ultimately film or wafer failure. Low stress silicon oxynitride films, as a substitute for silicon dioxide, are investigated to address the issue of stress. By varying the nitrogen content, thermally stable films with very low residual stress can be deposited. Wafer bow and radius of curvature are two figure of merits used to compare oxynitride versus oxide films processed with a single level of poly-Si. The results indicate that wafers processed with an oxynitride release layer are significantly flatter both before and after removal of films from the wafer backside. The wafer bow decreases by a factor of two while the wafer radius of curvature increases by a factor of three. Additionally, poly-Si layers processed on the oxynitride are observed to have a significantly lower stress gradient upon release. This is quantified by poly-Si cantilevers (475 micron length) fabricated with oxynitride which exhibit a typical radius of curvature in excess of 800 mm, compared to less than 600 mm for cantilevers fabricated with oxide release layers. It is proposed that this result is attributable to the enhanced thermal stability of the oxynitride and decreased thermal mismatch to poly-Si. It is concluded that substituting oxynitride for oxide results in reduced wafer deformation and flatter poly-Si components.

11:45 AM MM2.8

ON THE CHEMICAL CORROSION RESISTANCE OF ROOM-TEMPERATURE RF SPUTTERED Ta_2O_5 FILMS AND THEIR APPLICATIONS IN Si MICROMACHINING.

Miao-Ju Chuang, Kuang-Yeu Hsieh, National Sun Yat-sat Univ, Inst of Materials Science and Engineering, Kaohsiung, TAIWAN; Ann-Kuo Chu, National Sun Yat-sat Univ, Inst of Electro-optical Engineering, Kaohsiung, TAIWAN.

The commonly used dielectrics etch masks in EDP solutions for Si micromachining are nitride and oxide. However, to grow these masking materials requires heating the substrate to a very high temperature. This may cause fabrication incompatibility between micromechanical devices and conventional IC's. In this study, the chemical corrosion resistance of room temperature rf sputtered Ta_2O_5 has been investigated, and the use of Ta_2O_5 as an etch mask for bulk silicon dissolved processes is presented. For silicon wafers with 500 nm thick Ta_2O_5 masks deposited at an rf power of 150 W, the etching time can last for more than 4 h in a $120^\circ C$ EDP solution with negligible etch rate. After etched, the deposited Ta_2O_5 films were still mirror-like and the maximum roughness measured were 3.8 ± 0.5 nm and 8.2 ± 0.5 nm before and after EDP etch, respectively. However, the chemical corrosion resistance of the films was found to decline with the decreasing rf power. This can be caused by changes of the microstructure within the films during growth. The deposited films are amorphous and porous when the rf sputtering power is low. Further increasing the rf power to 150 W a preferred (002) orientation was observed. This is the case where there are few voids in the structure so that the film is dense and consequently is more resistant to the attack of chemicals. At a rf power of 300 W the corrosion resistance of the films declined due to the microstructure transition of the films. Based on the room-temperature technique proposed previously, we have successfully constructed a micro-cavity with Ta_2O_5 reflection mirrors using both the bulk and surface micromachinings. The detail information of the technique will be shown during the presentation.

SESSION MM3: NEW MATERIALS AND PROCESSES FOR MEMS I

Chair: Stephen Montague
Monday Afternoon, November 29, 1999
Room 313 (H)

1:30 PM *MM3.1

NEW MATERIALS AND NEW PROCESSES FOR MEMS APPLICATIONS. Janake Schweitz, The Angstrom Laboratory, Uppsala University, Uppsala, SWEDEN.

The field of non-silicon MEMS is today rapidly expanding. The reasons are numerous: silicon has a limited functionality in many MEMS applications; the toolbox for micromachining of non-silicon materials is steadily growing in size and versatility; the price level of a variety of other advanced materials for MEMS has become more competitive etc. This development is here illustrated by a number of examples from a wide variety of materials classes, methods for synthesis and microprocessing, and fields of application. Among these examples are high-aspect ratio processing of different materials by ion track technology (MITE) and deep ion projection lithography (DIPL); laser microprocessing and free-space 3D laser writing; high-precision

3D diamond replicas from microstructured silicon masters; and microreplication in polymeric materials for microoptics and microsystems technology.

2:00 PM MM3.2

FABRICATION OF FUNCTIONAL MICROCOMPONENTS FROM CERAMIC NANOPARTICLES. Alfredo M. Morales, Marcela Gonzales and Jill M. Hruby, Sandia National Laboratories, Livermore, CA.

Microelectromechanical systems (MEMS) are currently built from silicon, some metals, and a few other materials. Very few composite or ceramic microcomponents are available, even though incorporating ceramics into microscopic devices would enable the use of novel properties such as increased toughness, high temperature inertness, chemical and biological compatibility, magnetism, piezoelectricity, and photochromism. We will present recent results on the fabrication of functional MEMS microcomponents from ceramic nanoparticles. Our fabrication technique consists of: 1) formulation of a nanoparticle/binder mixture; 2) mold filling; 3) curing and planarization; 4) chemical removal of the mold. By using particles with nanometer diameters, we are able to mold components with lateral dimensions in the order of a few microns. These components can be produced free standing or assembled on substrates. This process is compatible with existing integrated circuit manufacturing processes, so incorporation of this technique into semiconductor fabrication lines should be feasible. Government Notice: The submitted manuscript has been authored by a contractor of the United States Government under contract. Accordingly the United States Government retains a non-exclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for United States Government purposes.

2:15 PM MM3.3

AMORPHOUS DIAMOND MEMS. T.A. Friedmann, J.P. Sullivan, M.P. de Boer, B.P. Jensen, D.A. Lavan, C.I.H. Ashby, J.A. Knapp, D.M. Follstaedt, M.M. Mitchell, R.G. Dunn, Sandia National Laboratories, Albuquerque, NM.

Micromechanical structures of amorphous diamond-like carbon (aD) have been fabricated. These structures consist entirely of aD and are not simply coated parts. aD based microelectromechanical systems (MEMS) should be inherently more reliable than the current standard systems that use poly-Si. aD is extremely hard, stiff, wear resistant, low friction, and low stiction. aD has not been used to this point in MEMS because of the high residual compressive stresses (6-10 GPa) typically found in as-grown films. These high stresses are 3-4 orders of magnitude larger than those required for MEMS device manufacture (< 2 MPa), since moderate residual stress causes released devices to distort rendering them useless. A process for routinely fabricating 1-2 μm thick aD films over 100 mm diameter wafers with low residual stress (< 2 MPa) has been developed. These thick films have been processed into simple single-mask level MEMS structures using standard MEMS manufacturing techniques. The structures manufactured include single- and double-clamped cantilever beams, tensile test rings, comb-drive actuators, and resonant fatigue structures. An environmentally-controlled interferometric micro-probing station was used to measure deflections of the single- and double-clamped beams. This data was combined with finite element modeling to extract information on stiction, residual stress, strain gradients, and the elastic modulus of the aD film. Early evidence strongly suggests that problems with stiction in these devices are greatly reduced over poly-Si based MEMS. In addition, simple actuation of resonant structures with electrical biasing has been demonstrated. Importantly, stress-free aD appears to be entirely compatible with current MEMS production techniques. *This work was supported by the U.S. DOE under contract DE-AC04-94AL85000 through the Laboratory Directed Research and Development Program, Sandia National Laboratories.

2:30 PM MM3.4

MASKLESS PATTERNING AND STRUCTURING ON ULTRA-HARD FILM MATERIALS. J.K. Park, K. Mukherjee, High Energy Laser Processing Laboratory, Dept of Materials Science & Mechanics Michigan State University, East Lansing, MI; V.M. Ayres, J. Asmussen, Electronic and Surface Properties of Materials Center, Dept of Electrical and Computer Engineering, Michigan State University, East Lansing, MI.

Increasing demands on performance of microelectromechanical systems (MEMS) create a need for materials with superior tribological properties. Some ultra-hard film materials, such as CVD diamond, are uniquely qualified for applications to MEMS. Patterning and structuring on the film materials are primary means to produce functional micro devices. However, the conventional lithography-based chemical etching is difficult or impossible on the ultra-hard film materials, with high chemical resistance. Maskless patterning and

structuring employ an excimer laser ablation technique, combined with micro motion stage control. Focused excimer laser pulses are used for dry etching on the film materials, and a micro computer numerical control (micro-CNC) stage is used for patterning and structuring. The laser ablation process is optimized for diamond films to set up relationships among processing parameters, such as environments, energy fluence, repetition rate, number of pulses, and laser energy absorptivity. Modeling of the ablation process on diamond films is studied with the optimized parameters. A micro-CNC stage with 5 motion axes, x-y-z with rotation and tilting features, is used for effective 3 dimensional micromachining. Patterned structures, from the innovative maskless process, are compared with conventional lithography based patterning.

2:45 PM MM3.5

DEMONSTRATION OF TWO- AND THREE-DIMENSIONAL NANOCRYSTALLINE DIAMOND STRUCTURES FOR A HIGH RESOLUTION DIAMOND-BASED MEMS TECHNOLOGY*. O. Auciello¹, A.R. Krauss², D.M. Gruen², E.M. Meyer³, H.G. Busmann⁴ and M.Q. Ding⁵; ¹Argonne National Laboratory, Materials Science Division, Argonne, IL, ²Argonne National Laboratory, Materials Science and Chemistry Divisions, Argonne, IL, ³Institute for Microsensors, Actuators, and Systems (IMSAS), University of Bremen, Bremen, GERMANY, ⁴Fraunhofer Institute for Applied Materials Science, (IFAM), Bremen, GERMANY, ⁵Beijing Institute of Electronics, Beijing, PR CHINA. *Work supported by the U.S. Department of Energy, BES-Material Sciences, under Contract W-31-109-ENG-38.

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 with 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. We demonstrate here the use of phase-pure nanocrystalline diamond (NCD) films for the fabrication of MEMS components. NCD 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 to fabricate a two-dimensional cantilevered NCD-MEMS strain gauge with ~ 100 nm feature size. The NCD-coated Si structure is attached at the corners to a Si anchor, and cantilevered over a patterned SiO₂ release layer. Prior similar diamond structures were limited in resolution by the grain size (typically ~ 1 μm) of the conventionally grown diamond layer. We coated Si posts with conformal NCD films to produce shafts for rotor and gear support. A three-dimensional hollow hexagonal post with 300 nm thick smooth walls was fabricated by conformally coating a pyramidal Si structure with NCD, followed by selective etching of the Si core with HF. The stability of this free-standing structure can be attributed to an extremely low-stress NCD film due to a very small grain size. The ability to produce 3D structures with variable z-cross sections is unique in that it cannot be achieved by conventional lithographic etch/lift-off methods, and represents a break-through in nanoscale fabrication technology based on nanocrystalline diamond coatings.

SESSION MM4: NEW MATERIALS AND PROCESSES FOR MEMS II
Chair: Christian A. Zorman
Monday Afternoon, November 29, 1999
Room 313 (H)

3:30 PM *MM4.1

3D PHOTONIC CRYSTAL AND ITS OPTOELECTRONIC APPLICATIONS. Shawn-Yu Lin and J.G. Fleming, Sandia National Laboratories, Albuquerque, NM.

A three-dimensional (3D) photonic crystal is an optical analogue of a semiconductor that is useful for controlling and manipulating the flow of light on a semiconductor chip. In this work, we report the realization of a series of silicon 3D photonic crystals operating in the infrared (IR), mid-IR and most importantly the near-IR wavelengths, i.e. 1-2 micron-meters. The structure maintains its crystal symmetry throughout the entire 6-inch wafer and maintains a complete photonic bandgap. This demonstration opens the door for Si-based photonic crystal devices that are compatible with well-developed Si microelectronics processes and are suitable for large-scale photonic integration. Experimental results taken from a 3-D single mode cavity (with a modal volume of a cubic wavelength), thermal emissivity data and the modified spontaneous emission spectrum will also be described. This work was supported by the United States Department

of Energy under contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy.

4:00 PM MM4.2

INLAYED ATOM-LIKE THREE-DIMENSIONAL PHOTONIC CRYSTAL STRUCTURES CREATED WITH FEMTOSECOND LASER MICROFABRICATION. Hong-Bo Sun, Ying Xu, Kai Sun, Saulius Juodkazis, Mitsuru Watanabe, Shigeki Matsuo and Hiroaki Misawa, Graduate School of Engineering, The University of Tokushima, JAPAN; Junji Nishii, Optical Materials Division, Osaka National Research Institute, Osaka, JAPAN.

We present here a new means to fabricate three-dimensional (3D) photonic crystal structures with arbitrary lattice types using laser microfabrication technology. (1)PRINCIPLE: When a femtosecond laser beam is tightly focused, transparent materials in the focal point (size in the order of wavelength) are highly excited and expand transiently in an explosive way, leaving a void hole surrounded with a densified crust. Such a phenomenon has been reported and used for 3D optical memory. If the microexplosion holes are arranged the same way as atoms in Bravais lattice, photonic crystals with different Wigner-Seitz primitive cell such as fcc, bcc, sc are achievable. (2)EXPERIMENTAL: Laser of 400 nm wavelength and 150 fs width (FMHW) at a repetition rate 1 kHz (second harmonic of Ti:Sapphire laser) was focused into the glass silica plate by an objective lens (100×, and NA~1.35). Microexplosion spots were distributed in the samples by computer-controlled 3D piezoelectric stage through a preprogrammed CAD pattern. The whole fabrication process was in-situ monitored with a CCD camera-monitor set equipped to the optical microscope. Near-ball voids are obtained due to the large NA and the high reproducibility of their shape is favored by stability of output of laser pulse energy. (3)RESULTS: Photonic crystal structures of different lattice types were fabricated. Bandgap effect was evidenced by FTIR transmission. As an example, a FCC lattice with lattice constant 1.68μ gives a transmission minimum at 3710 cm⁻¹, which is well fitted with transmission simulation. Photonic crystal-based integrated optical waveguide structures are also demonstrated.

4:15 PM MM4.3

MATERIALS ISSUES IN SINGLE-CRYSTAL SILICON AND SILICON NITRIDE NANOFABRICATED MECHANICAL OSCILLATORS. L. Sekaric, D.W. Carr, S. Evoy, J.M. Parpia and H.G. Craighead, Cornell Center for Materials Research and Cornell Nanofabrication Facility, Cornell University, Ithaca, NY.

Nanoelectromechanical systems (NEMS) are of interest for mesoscopic studies of materials as well as for a promising range of applications such as highly sensitive sensors, optomechanical and biomedical devices. The sensitivity and performance of such devices is highly dependent on various materials issues, such as loss mechanisms and material damage. We have fabricated single-crystal silicon and silicon nitride oscillators with paddle dimensions as small as 2x2 microns and supporting rods of width 50-200 nm. To our knowledge, these are the first nanometer scale mechanical structures reported in silicon nitride. The resonant frequencies of these devices are in the range of 1-10 MHz and 4-26 MHz for silicon and nitride, respectively. Different modes of motion are identified and their dependence on paddle dimensions is identical for both silicon and nitride devices. The nitride oscillators have higher frequencies than expected, indicating that the stiffness of the LPCVD grown nitride is larger than expected. The quality factor of the single-crystal Si devices is about 1000 while that of the nitride devices is an order of magnitude smaller, as expected from amorphous materials. The observed low Young's modulus for the silicon devices suggests crystalline damage during processing. We are further addressing these materials issues with annealing and ion implantation studies. We are also considering the effects of thin metal films evaporated onto the devices on the dissipation processes in these structures. Preliminary results from single silicon wires have shown that thin metal films have negligible effects as the losses are mainly surface related.

4:30 PM MM4.4

PIEZOELECTRIC RELIABILITY OF LEAD ZIRCONATE TITANATE THIN FILMS. Ronald G. Polcawich*, Paul J. Moses, Susan Trolier-McKinstry, The Materials Research Laboratory, The Pennsylvania State University, University Park, PA, *currently at the Army Research Laboratory, Adelphi, MD.

Lead zirconate titanate (PbZr_{0.52}Ti_{0.48}O₃) thin films are extremely attractive for MEMS applications. In this work, the reliability of the transverse piezoelectric response, d₃₁, in PZT thin films was studied under high electric field drive conditions. It was found that during unipolar drive, PZT films show excellent reliability with devices exhibiting increasing piezoelectric constants over time due to progressive poling of the capacitors. Under these conditions, 99% of

the ~1.0 mm thick capacitors examined survived to 10^9 cycles at field levels of 120 - 200 kV/cm. Aging rates between 6 and 12% per decade were typical for normally poled PZT thin films. It was concluded that depoling, possibly due to the presence of an internal electric field, was responsible for the large degradation in the transverse piezoelectric coefficient during aging. Even faster degradation occurred during low field bipolar operation with d_{31} dropping as much as 50% after 10^9 cycles. A field induced depoling was deemed responsible for the rapid decline in d_{31} during low field bipolar drive. Thermal and ultraviolet imprint techniques were used to improve aging and bipolar drive reliability. For films poled at 150°C, defect dipole alignment reduced the aging rate of d_{31} to 2 - 4%/decade. Low field bipolar drive on thermally imprinted devices resulted in increasing piezoelectric coefficients due to an increase in the net polarization. UV illumination resulted in non-linear aging due to the presence of an internal space charge that develops from photo-induced charge carriers. As the space charge field decays over time, d_{31} increases until $10^5 - 10^6$ seconds after poling, at which time d_{31} remains constant or slightly declines. Thus, the changes in d_{31} were confined to 3 - 5% at 10^6 seconds after poling. Similar behavior occurred during bipolar operation with minimal changes in the piezoelectric coefficient occurring after 10^9 cycles.

4:45 PM MM4.5

OPTIMIZATION OF POROUS SILICON MICROSTRUCTURE FOR HUMIDITY SENSOR APPLICATIONS. J. Salonen, M. Björkqvist, E. Laine, Turku Univ, Dept of Physics, Turku, FINLAND; L. Niinistö, Lab of Inorganic and Analytical Chemistry, Helsinki Univ of Technology, Espoo, FINLAND.

Due to its large specific surface area and compatibility with silicon technology, porous silicon is a potential candidate for gas and humidity sensors. By varying the preparation parameters a wide range of porosity and structural dimensions can be covered. It has also been observed that there exist two size distributions in PS, which differ from each other as the porosity decreases. The optimization of porous silicon microstructure for sensor applications requires that the effects of the preparative parameters have to be known in order to ensure the optimal pore size distribution and specific surface area. In this work we have compared the results obtained using different methods of characterization for the studies of porous silicon microstructure. The specific surface area has been used to compare the gas adsorption method (BET) to small-angle X-ray scattering (SAXS) while the average particle size determined from the wide angle X-ray powder diffraction (WAXD) studies has been compared to the average chord length in matter calculated from the SAXS data. Altogether, these methods give comprehensive information about the relationship of the porous silicon microstructure and the preparative parameters.

SESSION MM5: POSTER SESSION

Chairs: Maarten P. de Boer and S. Joshua Jacobs

Monday Evening, November 29, 1999

8:00 P.M.

Exhibition Hall D (H)

MM5.1

MICROMIRROR ARRAY DESIGNS FOR OPTICAL BINARY SWITCHING AND AMPLITUDE MODULATION APPLICATIONS. Edward S. Kolesar, Peter B. Allen, Josh M. Wilken, Jeffery T. Howard, Noah C. Boydston, S.Y. Ko, Texas Christian University, Dept. of Engineering, Fort Worth, TX.

Five types of micromirror arrays were designed and fabricated using a three-level, polysilicon, surface micromachined, microelectromechanical systems (MEMS) process. Although the micromirrors were fabricated through the DARPA-sponsored Microelectronics Center of North Carolina (MCNC) Multi-User Microelectromechanical Systems (MEMS) Process (MUMPS), the design features are applicable to other surface-micromachining fabrication processes. The electrostatically deflectable micromirror designs included arrays of simple cantilever beams, torsion beams, tethered (piston style) beams, circular membranes, and oval membranes. The smallest micromirror element was the simple cantilever beam, measuring 50 microns square. The largest micromirror element was the oval membrane; it possessed an active optical surface that was 320 by 920 microns. Each of the remaining micromirror designs have gold-coated polysilicon optical surfaces with geometries between these two limits. Electrostatically induced vertical deflections on the order of 2.75 microns were achievable. The torsion beam micromirror design exhibits both in-plane and out-of-plane deflection. The other micromirror designs only manifest in-plane deflections. The modeling phase focused on the microdynamical behavior of the torsion beam micromirror. The IntelliCAD[®] finite element analysis program was used to generate a plot of micromirror deflection versus applied voltage. The following performance evaluation topics are reported for each micromirror

design: i) direct current (dc) threshold voltage required to induce observable deflection, ii) maximum dc operating voltage, and iii) reliability (number of operational cycles) of each micromirror design when operated with a rectified 60 Hz alternating current (ac) signal. Experimental evidence supporting the potential for using micromirrors as binary optical switches and amplitude modulators is addressed.

MM5.2

A MICROMACHINED POLYSILICON RESONATING XYLOPHONE BAR MAGNETOMETER. T.J. Kistenmacher, D.K. Wickenden, J.L. Champion, R.B. Givens, R. Osiander, J.L. Lamb, The Johns Hopkins University, Applied Physics Laboratory, Laurel, MD.

A miniaturized, polysilicon version of a resonating xylophone bar magnetometer with dimensions of order microns has been fabricated by microelectromechanical systems (MEMS) processing techniques. All devices tested to date have performed extremely well in static magnetic fields and exhibit mechanical quality factors, Q, of up to 30,000 at reduced pressures. The resonance frequencies of the fundamental mode of vibration of these polysilicon xylophone bars have been found to be sensitive functions of the torsional stiffness of the support arms, in accord with an analytical model based on Bernoulli-Euler theory. The output response of the polysilicon xylophone magnetometer as a function of impressed magnetic flux density has been shown to be linear up to 150 μ T; however, sensitivity is limited at present by the significant resistivity of the polysilicon xylophone bar. Various strategies are being formulated and implemented to bring the magnetometer sensitivity into the applications-dominated nT regime.

MM5.3

MICROMACHINED THERMOSENSITIVE Si-TIP SENSORS. Ding Xinfang, He Yunshuang, Southeast Univ, Microelectronics Center, Nanjing, P.R. CHINA; G. Chen, Q.Y. Tony, Duke Univ, Durham, NC.

A new design of Si-tip sensor is presented in this paper. Using the construction of AFM for reference, a Si-tip is constructed on the front part of the cantilever, the thickness of cantilever is about 6 μ m and radius of the top of tip is less than 50 nm. On the Si tip, a Si-Si₃N₄-Ti/Pt/Au structure is fabricated, which is the main part of sensor. By exerting voltage between Si and metal, the Si₃N₄ on the top of tip is broken down and forming a tunnel through Si₃N₄. The work principle of it is like the thermocouple. And temperature can be measured according to the different thermoelectromotive between Si and metal.

The cantilever of the sensor is etched by anisotropic corrosive. KOH solution is used for etching and heavily dope of boron is also used to control the thickness of the cantilever to reach about 6 μ m.

The bottom of the tip is about Φ 5 μ m. Isotropic corrosive, HF: HNO₃=1:5, is used to etch the tip, and the top of the tip can reach Φ 50 nm. Then the tip is sharpened by low temperature (about 900°C) oxidation technique. Then the tip can reach Φ 30 nm.

The V-I graph is measured between metal and Si. Before broken down, the V-I characteristic is just like a diode because of the potential barrier between Si and metal. After broken down, the V-I characteristic is like a resistance. That indicates Si-metal tunneling is formed in the infinitesimal area on the top of Si tip.

Because the diameter of the tip is very small so this sensor can be used to measure the temperature of small area, especially to detect the damage of small component in ICs. If we control the voltage to rise the temperature of tip, it can even repair the down leads of ICs.

MM5.4

DEVELOPMENT OF A MEMS XYLOPHONE BAR MAGNETOMETER USING OPTICAL INTERFEROMETRY FOR DETECTION. R. Osiander, J.L. Champion, D.A. Oursler and J. Miragliotta, The Johns Hopkins University, Applied Physics Laboratory, Laurel, MD.

We report the results of an optical interferometric study designed to measure the magnetic-field induced displacement of a resonating xylophone bar MEMS magnetometer. The MEMS magnetometer is a Lorentz force sensor, which transduces an alternating current and an orthogonal directed magnetic field into an alternating displacement of the xylophone bar. Under typical operational conditions, the presence of a nanoTesla field will produce bar displacements that are on the order of 1 to 10 angstroms. Therefore, the high sensitivity of optical interferometry to spatial displacements is well suited for use in this magnetometer design. Two different, path-stabilized interferometer systems were investigated: (1) the dual-beam Michelson, and (2) the multi-beam Fabry-Perot. In the Michelson study, theoretical calculations determined a noise-limited bar displacement sensitivity of ~ 0.01 angstrom at room temperature. In the Fabry-Perot design, where detection relies on the ability to Doppler shift the optical frequency of the probe beam via the motion of the bar, the noise-limited sensitivity was nearly an order of magnitude better than

the Michelson system. In both systems, the calculated sensitivity limits were well below the corresponding thermal noise deflection limit of 2.2 angstroms. Experimental results will be presented that illustrate the magnetic field sensing abilities of the two interferometer systems. Both systems are observed to have nanoTesla detection capabilities at room temperature. The results also indicate that the two interferometric approaches have a higher sensitivity to xylophone bar displacements when compared to a simple beam deflection approach.

MM5.5

HYBRID MICROCHEMICAL REACTORS FABRICATED BY BOTH CONVENTIONAL AND UNCONVENTIONAL TECHNIQUES. Rebecca J. Jackman, Reza Ghodssi, Martin A. Schmidt, Klavs F. Jensen, Massachusetts Institute of Technology, Department of Chemical Engineering and Microsystems Technology Laboratories, Cambridge, MA.

Microchemical reactors can offer advantages over their large-scale counterparts. These advantages can be a consequence of the low thermal mass of the device that can result in increased temperature control and fast thermal response times, of the small dimensions of flow channels that can be on the same order as diffusion lengths and can minimize problems of mass transfer, and of the high surface-to-volume ratio that can allow for efficient heat transfer and that facilitates interfacial reactions. Often silicon is chosen as the material from which to form these microchemical reactors because microfabrication techniques for silicon processing are well developed and they offer the opportunity to produce a fully integrated structure. In many cases, silicon functions well as the substrate for the reactor, but a different material (e.g. a polymer or ceramic) that could be patterned and processed at less expense or with shorter cycle time would be preferable. In some microchemical applications, silicon-based devices are not suitable because they are not compatible with the chemistry: alternative materials are needed. This presentation will discuss the fabrication of composite structures formed from silicon and other materials, including SU-8 (to form high-aspect ratio structures) and polydimethylsiloxane, that take advantage of both conventional and unconventional fabrication techniques to produce integrated microchemical reactors. Model electrochemical reactions in these hybrid chemical microreactors will be presented.

MM5.6

ROUGH CHARACTERIZATION OF Si(110) ETCHED IN TMAH BY ATOMIC FORCE MICROSCOPY. Zakaria Mokhtadir, Kazuo Sato, Matsumoro Akihito, Kayokawa Kimiharu, Mitsuhiro Shikida, Dept of Micro-system Engineering, Nagoya University, Nagoya, JAPAN.

Anisotropic etching of silicon has become a widely used technique to allow a fabrication of a variety of micro-structures. In general, these etched structures have a complicated surface morphology and etching leads to an impressive variety of surface texture depending on the crystallographic orientation. The purpose of our work is to analyze the surface roughness in different scales (200nm ~ 1 μ m) by Atomic Force Microscopy (AFM) for < 110 > oriented-silicon after etching with TMAH. AFM technique is rapidly becoming a powerful tool for surface characterization in material science. We use this technique to investigate the scaling properties of the root-mean-square roughness (rms) which is scale-dependent quantity. As a material is extracted from the surface by a stochastic process (for example sputtering or etching) one can expect the resultant morphology to be self-affine. This property has been observed in etched Si(110) using TMAH. Both the surface width and the power spectra scale as a power law of the system size. The surface width w scales as $w \sim L^\alpha$, where L is the system size and α is known as the roughness exponent which is related to the fractal dimension D of the surface by the relation $\alpha = 3 - D$ in 2+1 dimension. The fractal dimension of the observed surface have been determined using two different techniques (power spectrum density and the lake pattern algorithm) from which we deduce the parameter α . The roughness exponent was also computed by scaling analysis (the scale dependence of surface width). We have compared the obtained value of the scaling exponent in the current experiment with the KPZ (Kardar-Parisi-Zang) value obtained from literature in 2+1 dimension. Our value is larger than the KPZ value (~0.4). Further experimental work and studies will be carried out to understand this difference and to investigate if anisotropic etching of silicon belongs to KPZ universality class.

MM5.7

THE ETCH RATE VARIATIONS OF P+ SILICON WAFERS IN AQUEOUS KOH SOLUTIONS AS A FUNCTION OF PROCESSING CONDITIONS. Petteri Kilpinen, Eero Haimi, Veikko K. Lindroos, Laboratory of Physical Metallurgy and Materials Science, Helsinki University of Technology, Espoo, FINLAND.

The etching behaviour of highly boron doped (20 - 25 m Ω -cm) silicon wafers in aqueous KOH solutions were investigated in the present

study. The etch rate of (100) crystal plane and the fastest etching plane was measured as a function of processing conditions. The experimental temperatures of solutions were 65 - 75 °C and the concentrations were 5 - 40 w-% KOH. The maximum etch rate of (100) plane was measured to locate between 10 - 15 w-% KOH and the maximum etch rate of fastest etching plane was measured to locate between 0 - 5 w-% KOH in the whole temperature range. In lower concentrations (5 - 20 w-% KOH) the fastest etching plane was determined to be crystal plane (31X) (where X can be 0,1,2). In higher concentrations (25 - 40 w-% KOH) it was determined to be crystal plane (41X). In 5 w-% KOH the fastest etching plane was etched 2.3 times more rapidly than (100) plane whereas in 40 w-% KOH it was etched only 1.5 times more rapidly. The results of this study indicate that there are differences between etch reactions in different etch planes. The difference may be in number of transferring electrons. This could explain why the different etch planes have the position of etch rate maximums as a function of KOH concentration in different places. Furthermore, this could also explain why p+ type wafers in this study behave differently than p type wafers referred in the literature.

MM5.8

DEFINING CONDITIONS FOR THE ETCHING OF SILICON IN AN INDUCTIVE COUPLED PLASMA REACTOR. Huma Ashraf, J.K. Bhardwaj, S. Hall, J. Hopkins, A.M. Hynes, I.R. Johnston, S.A. McCauley, G.W. Nicholls, L.M. Lea; Surface Technology Systems Ltd, Imperial Park, Newport, UNITED KINGDOM; Paul O'Brien, Dept. of Chemistry, Imperial College of Science, Technology and Medicine, London, UNITED KINGDOM.

The processes which fundamentally limit the etching of silicon in high density fluorinated plasma processes are poorly understood. In an effort to improve our understanding of the performance of such systems, the etching of silicon wafers by an inductive coupled plasma reactor, using SF₆, has been studied. A systematic empirical investigation has allowed us to define many of the experimental parameters that control the etching rate. There is little temperature dependence on etching, indicating a low-activation energy process. This suggests a diffusion-based chemically dominated process. Systematic variation of other parameters controlling the rate of etching: total pressure in the reactor, flow rate, partial pressure of reactive species and the rf power supplied to the discharge enable us to accurately define the performance of the system under study. Experiments which segregate the physical and chemical components of the etching process supports the conclusion that etching is dominated by electrically neutral species. These various results are interpreted in terms of accepted models for the reactive chemistry in plasmas containing SF₆. As the MEMS industry matures, it places ever more demands on the processes. There is a growing requirement to achieve the same degree of anisotropy, and critical dimension control, but at much higher etch-rates. The approach outlined allows us to develop effective strategies for evolving improved systems for the high rate plasma etching of silicon.

MM5.9

POROUS SILICON AS A SACRIFICIAL MATERIAL FOR MICROSTRUCTURES FABRICATION. M. Morel, M. Le Berre, V. Lysenko, G. Delhomme, A. Dittmar, D. Barbier; LPM-INSA LYON, UMR CNRS, Villeurbanne, FRANCE.

Porous silicon is generated by electrochemical etching in hydrofluoric acid (HF). Recently porous silicon has been applied to micro-machining and micro-devices as an alternate material, this material being used as a sacrificial layer. This technology competes with conventional techniques like surface and bulk micromachining regarding its speed, simplicity and reduced costs. A wide range of microstructures and free-standing structures can be fabricated with a large freedom of design in relation to the isotropic behavior of the etching. A sacrificial layer may be realized fast over varying thickness (etch speed 45 μ /h compared to 20 μ /h for KOH etching). This contribution is devoted to the materials aspects of patterning and processing; we will show how basic microstructures (channels, cantilevers, free-standing membranes) may be fabricated using a simple process based on a single photolithography. The important points are the choice of the mask, porous silicon properties as a function of its formation parameters and the choice of the solution removing the sacrificial layer. The morphology and porosity of the porous silicon layers are indeed mainly determined by the electrolyte composition and by the current density for a given substrate type. Optimized conditions (HF 15% and 80 mA/cm²) lead us to an appropriate porous silicon. Finally the applicability of this technology for various microsensors will be underlined.

MM5.10

β -SiC GROWN ON SiO₂ FOR ROBUST MEMS APPLICATIONS. J. Chen, A.J. Steckl, University of Cincinnati, Nanoelectronic Laboratory, Cincinnati, OH; J. Scofield, Air Force Research

Laboratory, Wright-Patterson AFB, OH; M.J. Loboda, Dow Corning Corporation, Midland, MI.

SiC is a promising material for the fabrication of MEMS devices that need to operate at high temperatures and in high radiation environment due to its superior physical and chemical properties. SiC can be epitaxially grown on Si substrates. Recently, we have successfully grown well-ordered cubic SiC(111) on SiO₂ surfaces directly, which provides an alternative material system for making robust MEMS devices. Additionally, SiC growth on Si₃N₄ and poly-Si is also being investigated for their suitability as surface micro-machining sacrificial layers. We report the growth of cubic SiC with organosilanes (silacyclobutane-SCB and trimethylsilane-3MS) on SiO₂-covered Si(100). A comparison to SiC growth on Si₃N₄ and poly-Si is presented in terms of SiC film quality and subsequent MEMS device characteristics. The oxides (300 to 1000 Å) were thermally grown on Si(100) substrates. In addition, Si(100) with native oxide and 3µm thick P-doped SiO₂ film were used as substrates. For all SiC growth experiments, the flow rates are 1 slm for hydrogen, 40 sccm for 3MS or SCB. The growth temperature ranged from 1100 to 1200°C. The SiC growth rate at these conditions is up to 1 µm/min. The surface and interface quality were examined by SEM. The top surface and the interfaces of SiC/SiO₂ and SiO₂/Si(100) are generally smooth. The voids normally generated during growth of SiC directly on Si are not observed in this case at either interface. In FTIR spectra, only the Si-C signal is observed after subtracting the SiO₂-related background. This indicates that the grown film is primarily SiC. X-ray diffraction spectra show cubic SiC oriented in the (111) direction was always grown. The XRD linewidth is around 0.25° for SCB growth and 0.21° for 3MS growth, which is similar to those of SiC grown on Si with the traditional two-step (carbonization plus growth) method. Measured material properties and performance characteristics of simple electrostatically actuated cantilever beam and materials diagnostic test structures fabricated in these SiC films are presented.

MM5.11

SU-8 PROCESSING ON A VARIETY OF SUBSTRATES.

Yuh-Min Johnson Chiang, Mark Bachman, Charles Chu, G.P. Li, University of California at Irvine, Department of Computer and Electrical Engineering, Irvine, CA.

SU-8 has become a popular material for micromachining high aspect ratio structures. Typically, SU-8 is spun on a polished silicon wafer for processing. After patterning, the SU-8 is used for micromachined structures directly (such as fluidic channels) or as a mold for electroforming. Non-silicon substrates offer the possibility of cheaper processing, improved mold designs, and multi-material devices. Successful SU-8 processing depends strongly on surface properties of the substrate itself as well as environmental conditions during the processing. We explore the issues involved in transferring SU-8 technology to non-silicon substrates such as glass, plastics and ceramics. Issues such as wettability, adhesion, and surface tension are explored in this study. The findings indicate the merits of non-spinning approaches, such as dipping, pouring and spraying, and point to new SU-8 processes.

MM5.12

Abstract Withdrawn.

MM5.13

CHARACTERIZATIONS OF METALLIZED PLASTIC MEMS.

Mark Bachman, Yuh-Min Chiang, Charles Chu, Fernando Gonzales, G.P. Li, University of California at Irvine, Department of Computer and Electrical Engineering, Irvine, CA.

Metallization of plastic devices is of interest to newer non-silicon based MEMS which are being developed for biomedical and other applications. The electrical properties of the metallization will depend, in part, on the material on which they are patterned, and the method of deposition. Of interest are the electrical characterizations during usage which must be studied to allow plastic MEMS designs to proceed wisely. Flexible microdevices may develop open circuits or shorts after excessive bending; joule heating may adversely affect the plastic substrates, leading to failure. We present some experimental studies on electrical characterization of metallized plastics as it relates to various processing conditions and usage scenarios.

MM5.14

TENSILE TESTING OF PZT FILM. W.N. Sharpe, Jr., G. Coles,

Johns Hopkins University, Department of Mechanical Engineering, Baltimore, MD; R.L. Edwards, Johns Hopkins University, Applied Physics Laboratory, Laurel, MD; M. Dube, E. Zakar, Army Research Laboratory, Sensors and Electronic Devices Division, Adelphi, MD.

PZT (lead zirconate titanate) films deposited from solution offer potential as actuators for microdevices. Measurement of the

mechanical properties is difficult because of the small size and fragile nature of the test specimens. Few measurements have been made; Young's modulus has been determined from resonant cantilever beam tests and from membrane bulge tests. In both cases, the PZT, which is one micron thick, is supported by thicker layers of other materials. We have developed new techniques and procedures for tensile testing of PZT film sandwiched between 0.2 micron thick layers of platinum. Strain is measured directly on the specimen by laser interferometry, and specimens of different PZT thicknesses are tested to eliminate the effect of the fixed-thickness outer layers. The specimen preparation and test methods are adapted from proven procedures for measuring the stress strain curves of thin-film polysilicon. The Pt/PZT/Pt sandwich is deposited onto a one cm square silicon die in a pattern that has the tensile specimen between two larger grip ends. The silicon wafer under the specimen is etched away leaving it supported between the grips, which are connected by support strips. The die is then mounted in a small test machine that has a linear air bearing to eliminate friction. The support strips are cut, leaving a freely suspended tensile specimen. Strain is measured directly with gold lines deposited on the specimen. By recording the stress-strain response of specimens with different thicknesses of PZT, one can subtract the effect of the outer layers and determine the Young's modulus of the PZT. PZT layers ranging from 0.25 to 2 microns are used in these experiments. The techniques and procedures as well as the results from a comprehensive series of tests will be presented.

MM5.15

STATIC AND DYNAMIC CHARACTERIZATION OF BUCKLED COMPOSITE SiO₂ MICROBRIDGES. Liviu Nicu, Christian Bergaud, Augustin Martinez, Laboratoire d'Analyse et Architecture des Systèmes, Centre National de La Recherche Scientifique, Toulouse, FRANCE.

Thin films of polysilicon, silicon nitride, silicon dioxide and some metals are extensively used in microelectronics or microelectromechanical systems (MEMS). Mechanical testing of thin films a few microns thick is important for investigative and design purposes. Most microstructures and microsystems can respond nonlinearly provided they are pushed into a nonlinear range of operation. Thus, adequate methods for reliably investigating the thin films mechanical properties are becoming increasingly important. In this paper, we will present a study of the static and dynamic behavior of buckled composite SiO₂-Au microbridges. 0.45 µm-thick SiO₂ microbridges were batch-fabricated using surface and bulk micromachining techniques. The width of the microbridges is 40 µm for a length varying from 150 µm to 300 µm with a 50-µm step. They were coated with a 0.1 µm-thick gold layer. First, these microbridges were characterized from a static point of view. In order to measure the residual stress in the SiO₂ and Au layers, the approach proposed by Lin et al.¹ has been used. Assuming the initial postbuckling deflections of the SiO₂ and SiO₂-Au microbridges measured using an atomic force microscope, the compressive residual stress in the SiO₂ layer was found to be about 275±9 MPa and the effective compressive residual stress in the SiO₂-Au bi layer was about 261±7 MPa. Then, the tensile residual stress in the Au layer was computed using a typical mixture law and it was found to be about 258±4 MPa.

Using the optical beam deflection technique, multimode non-linear responses of the initially deflected composite SiO₂-Au microbridges were obtained experimentally and compared for the first time to analytical predictions determined using the approach proposed by Nayfeh et al.² A bi-layer approach has been implemented to take into account the gold layer effect on the resonant frequencies. The comparison between theory and experiment shows a good agreement.

¹S.C.H. Lin and I. Pugacz-Muraszkiewicz, J. Appl. Phys. 43, 119 (1972).

²A.H. Nayfeh, W. Kreider and T.J. Anderson, AIAA J. 33, 1121 (1995).

MM5.16

Abstract Withdrawn.

MM5.17

MECHANICAL PROPERTY MEASUREMENT OF ELECTROPLATED GOLD MICROSTRUCTURE USING RESONANCE METHOD. Chang-Wook Baek, Yong-Kwon Kim,

School of Electrical Engineering, Seoul National Univ, Seoul, Korea; Yoo-Min Ahn, Dept of Mechanical Engineering, HanYang Univ, Ansan, KOREA.

Young's modulus and residual stress of the electroplated gold microstructures were determined by measuring resonance frequencies of microbeams. Micro cantilever and bridge structures were fabricated by electroplating surface micromachining technique using photoresist sacrificial layer and UV-lithography for thick photoresist mold. In order to obtain an anchor close to ideal fixed-end boundary condition for preventing change of resonance frequency in conventional anchor,

seed layers on the sacrificial layer and substrate are separated so that the structural part is automatically plated after anchor hole is filled with the deposited material. Dimensions of fabricated beams are typically 100 μm to 1000 μm long, 100 μm wide, and the thickness is varied by controlling the electroplating time. Beams are electrostatically driven by applying AC voltage between the structure and the bottom electrode, and the resonance frequency is measured by monitoring the vibration amplitude of the beam with position-sensitive PIN photodiode. Young's modulus and residual stress are extracted using the frequency equation of cantilever and bridge respectively[1,2]. Measured data show that electroplated gold is in the tensile stress, and released cantilevers are bended upward due to the internal stress gradient. Experimental results and effects of the plating process parameters like current density, mechanical agitation on the mechanical property will be presented.

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- S. Bouwstra and B. Geijselaers, "On the resonance frequencies of microbridges," *Proc. 6th Int. Conf. Solid-State Sensors and Actuators (Transducers 91)*, San Francisco, CA, USA, June 24-27, 1991, pp. 538-542

MM5.18

HIGH-TEMPERATURE THERMOMECHANICAL PROPERTIES OF SILICON NITRIDE FILMS USED IN MEMS. Haruna Tada, Ioannis Miaoulis, Peter Wong, Tufts University, Thermal Analysis of Materials Processing Laboratory, Medford, MA; Patricia Nieva, Paul Zavracky, Northeastern University, Microfabrication Laboratory, Boston, MA.

Microelectromechanical systems (MEMS) has potential application in high temperature environments such as in thermal processing of microelectronics. The MEMS designs require an accurate knowledge of the temperature-dependent thermomechanical properties of the materials. Techniques used at room temperature often cannot be used for high-temperature property measurements. MEMS test structures have been developed in conjunction with an apparatus designed to allow image analysis in-situ in order to find the modulus of elasticity and thermal expansion coefficient of thin films at high temperatures. The MEMS test structure is the common bilayered cantilever beam which undergoes thermally induced deflection at high temperatures. An individual cantilever beam on the order of 100 μm long can be viewed up to 1000°C. Moreover, with image analysis the curvature of the beam can be determined as well. The results for studying silicon nitride films on silicon oxide are presented for a range of temperatures.

MM5.19

FATIGUE TESTING MACHINE OF MICRO-SIZED SPECIMENS FOR MEMS APPLICATIONS. Y. Higo, K. Takashima, M. Shimojo, Tokyo Institute of Technology, Precision and Intelligence Lab, Yokohama, JAPAN; S. Sugiura, Nissei Sangyo Co. Ltd., JAPAN; B. Pfister, CSIRO, AUSTRALIA; M.V. Swain, Univ of Sydney, Dept of Mechanical Engineering, AUSTRALIA.

Fatigue properties and long-term reliability of micro-sized materials are extremely important to design actual micromachines and MEMS, since many micro-sized moving components are involved in such devices and the components are subjected to cyclic loading as they move. However, there have been few studies to date which investigate fatigue properties of micro-sized materials, since there is no adequate fatigue testing equipment for micro-sized materials. In this investigation, a fatigue testing machine for micro-sized materials has been developed. The fatigue testing machine consists of a magnetostriuctive actuator which is able to impart small displacements to a specimen upto 20 μm with resolution of 5 nm. The actuator is connected to a metal shaft and a diamond tip of 5 μm in radius is attached to the end of the shaft. Small displacements are applied to the specimen through the diamond tip. This makes it possible to construct a high stiffness loading fixture. The magnitude of load applied to the specimen is measured by a strain gauge type load cell with a load resolution of 10 μN . The specimen stage and load cell can be moved to adjust the loading position by a stepping motor at a translation resolution of 0.1 μm . Cantilever beam type specimens with dimensions of 30 x 12 x 50 μm^3 were prepared from an Ni-P amorphous thin film by focused ion beam machining. Very small cyclic load ($\Delta P = 0.3 - 40 \text{ mN}$) was able to be applied to the specimen successfully. This machine appears to be promising for evaluation of fatigue properties for micro-sized specimens.

MM5.20

FATIGUE PROPERTIES FOR MICRO-SIZED Ni-P AMORPHOUS ALLOY SPECIMENS. S. Maekawa, K. Takashima, M. Shimojo, Y. Higo, Tokyo Institute of Technology, Precision and Intelligence Lab,

Yokohama, JAPAN; M.V. Swain, Univ of Sydney, Dept of Mechanical Engineering, Sydney, AUSTRALIA.

Fatigue life and fatigue crack propagation tests have been performed for micro-sized Ni-P amorphous alloy specimens to investigate the fatigue properties of micro-sized specimens. The material used was a Ni-11.5 mass%P amorphous thin film, and cantilever beam type specimens with dimensions of 30 x 12 x 50 μm^3 were prepared by focused ion beam machining. Notches with depth of 3 μm were introduced in some specimens. All fatigue tests were performed using a newly developed fatigue testing machine in air at room temperature under a constant load ratio of 0.1. The fatigue life curve was obtained for un-notched specimens, and the fatigue strength of the specimen is determined to be approximately one-thirds of the static bending strength. Fatigue crack propagation tests were performed on notched specimens. Fine equispaced markings were observed on the fatigue surface. These markings were aligned perpendicular to the crack growth direction and are considered to be striations. Therefore, the crack is deduced to extend by cyclic plastic deformation at the crack tip even in micro-sized amorphous alloys.

MM5.21

RESISTANCE OF SU-8 TO HARSH CHEMICAL AND RADIATION ENVIRONMENTS. Frank Zee, Jack W. Judy, University of California, Los Angeles, Electrical Engineering Department, Los Angeles, CA.

An inexpensive thick-film high-aspect-ratio lithography process is needed for many MEMS applications (e.g., electroplating molds, microchannels and reservoirs for microfluidics, etc). The use of UV photolithography provides a more economical alternative to expensive X-ray LIGA processing for high-aspect-ratio MEMS devices. SU-8, which is an epoxy-based negative-imaging resist with a sensitivity in the near-UV (350-400 nm), has been developed for thick resist applications where high aspect ratios and resistance to harsh conditions are required. In our studies, we have exposed SU-8 structures to various harsh environmental conditions to study its robustness. An array of test structures, up to 300 μm thick, was fabricated using SU-8 100 (MicroChem Corp.) on a < 100 >-silicon wafer. The SU-8 devices were exposed to solvents, bases, and acids, as well as to gamma and electron radiation. The solvents used were isopropanol, ethanol, methanol, acetone, toluene, tetrahydrofuran, and dichloromethane. The alkaline and acidic solutions were potassium hydroxide, tetra-ammonium hydroxide, hydrofluoric acid, nitric acid, hydrochloric acid, and sulfuric acid. The gamma and electron radiation dosage was varied from 10 rad to 20 Mrad. The devices are incrementally characterized using a SEM to monitor for any variations in deformation, cracking, swelling, erosion, and adhesion. Material testing was conducted at the end to monitor for any changes in mechanical and electrical properties. The devices were also characterized under Raman spectroscopy. This paper will describe in more detail the SU-8 test structures, the experiments that were conducted, and the their results.

MM5.22

TEST CHANNELS FOR FLOW CHARACTERIZATION OF PROCESSED PLASTIC MICROCHANNELS. Yandong Chen, Zhongping Chen, Yonghua Zhao, J. Stuart Nelson, Mark Bachman, Yuh-Min Chiang, Charles Chu, G.P. Li, University of California at Irvine, Department of Computer and Electrical Engineering, Irvine, CA.

Characterization of the flow properties in microfluidic channels is important for designing and building biomedical microdevices, many of which depend on precise fluid flow for their operation. Similarly, in complex fluidic systems, it is important to identify flaws in processing which will potentially restrict, or short circuit the flow of the device. We explore the characterization of flow in plastic microfluidic systems through various test geometries. Issues such as flow rate, flow profile, bubble formation and dead flow regions are studied for different flow geometries and materials.

MM5.23

X-RAY MICROPROBE STUDIES OF MATERIALS PROBLEMS RELATED TO MICROELECTROMECHANICAL SYSTEMS (MEMS) STRUCTURES. Nicholas Moelders, Louisiana State Univ, CAMD, Baton Rouge, LA; Paul J. Schilling, Univ of New Orleans, Dept of Mechanical Engineering, New Orleans, LA; Jost Goettert, Herbert O. Moser, Forschungszentrum Karlsruhe, ANKA Project Team, Karlsruhe, GERMANY; Volker Saile, Forschungszentrum Karlsruhe, Institut fuer Mikrostrukturtechnik, Karlsruhe, GERMANY.

The understanding of the physical, chemical and mechanical properties of materials used in micro-environment is essential for the successful performance of MEMS components. Previous investigations of such materials at the Center for Advanced Microstructures and Device (CAMD), Louisiana State University, including the

non-destructive characterization of various electroplated Permalloy foils and other alloys, used in the LIGA process, by x-ray absorption spectroscopy presenting the local structural analysis and thickness/density measurements. However, these studies were limited to a scale of a few millimeters. Recently at CAMD, we developed a new tool which can non-destructively characterize materials on a microscopic scale of $20\ \mu\text{m} \times 7.0\ \mu\text{m}$. This new tool has been used to study various materials problems related to the production of MEMS devices. In this presentation, we will show spatially-resolved x-ray transmission measurements of the graphite substrate of an x-ray mask indicating the transmission fluctuations due to high-Z trace elements. Furthermore, the characterization as well as the spatial location of radiation induced production of sulfur functional groups in a new potential x-ray resist indicative of the dose deposition.

MM5.24

CRYSTALLIZATION OF RELATIVELY THICK AMORPHOUS SILICON FILMS FORMED BY LPCVD BETWEEN 550°C AND 580°C. A.Q. He, J. Yang, H. Kahn, S.M. Phillips and A.H. Heuer, Dept. of Materials Science and Engineering and Dept. of Electrical Engineering and Computer Science, Case Western Reserve University, Cleveland, OH.

Crystallization of amorphous silicon films has been studied for many years because of its utilization in integrated circuits. Previous studies have generally focused on thinner films (200 to 6000Å) than are used in MEMS devices; surface-micromachined polysilicon MEMS devices are usually $2\ \mu\text{m}$ thick or greater, and invariably utilize a SiO_2/Si wafer substrate. We have used TEM to show that LPCVD amorphous silicon films in this thickness range deposited between 550°C and 580°C begin crystallizing before the deposition is complete. Crystallization occurs by heterogeneous nucleation at the $\text{SiO}_2/\text{amorphous silicon}$ interface and by homogeneous nucleation within the amorphous silicon itself. TEM further reveals that the crystallites are roughly ellipsoidal in cross section, and that the polysilicon grain size varies with deposition temperature.

MM5.25

GRAIN STRUCTURE AND TEXTURE OF INDIVIDUAL POLYSILICON LAYERS FOR MEMS. D.A. LaVan, T.E. Buchheit and J.R. Michael, Sandia National Labs, Albuquerque, NM.

Advanced surface micromachined MEMS devices can now be fabricated from five polysilicon layers at Sandia's Microelectronic Development Laboratory. Each layer is annealed after deposition, a procedure that creates significant differences in thermal history between the first and last polysilicon layers. The evolution of the microstructure with multiple anneal cycles is not understood. An automated Electron Backscatter Kikuchi Pattern (EBKP) equipped SEM was used to characterize the grain structure and texture of each individual layer. The spatial resolution for acquiring each Kikuchi pattern was between 0.05 and 0.1 microns. Understanding the grain orientation and texture is critical to predict the variation in response of devices because of the increased significance of each grain in micro-scale devices as the grain size approaches the feature size. Data was collected from polysilicon deposited directly onto polysilicon, as well as in layers separated by oxide. 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.

MM5.26

CHARACTERIZATION OF MULTISTACK ANODIC BONDS USING A NON-DESTRUCTIVE METHOD. Jose A. Plaza, Estrella Gonzalez, Jaime Esteve, National Centre of Microelectronics, Barcelona, SPAIN.

Anodic bonding is a commonly used technique in microelectronics for bonding wafers. It is especially used for packaging of micromechanical sensors and actuators in order to reduce the mechanical stress induced by the packaging process. Glass Pyrex #7740 or similar glasses, such as Hoya SD-2, are used because the thermal expansion is similar to that of silicon, between room temperature and the bonding temperature. The technique was established by Pomerantz 30 years ago, and has been used by research companies and institutes. Many commercial microdevices have been fabricated by this technique. However, the process is still not completely understood, and all current bond quality tests are destructive. In this paper we present a non-destructive test for characterizing anodic bonding, which can also be used to study multistack anodic bonds in microelectronics. An important parameter during anodic bonding is the electrostatic pressure between the surfaces to be bonded. Higher electrostatic pressure increases the effective bond area. The test consists of determining the electrostatic pressure during bonding. Cavities with precise dimensions are made on silicon wafers. If the electrostatic pressure exceeds the stiffness of the cavity during the process, the cavity itself will be bonded. The value of the electrostatic pressure

can be obtained from the dimensions of the cavities. Development of a successful process for multistack bonding is important for the fabrication of microdevices. Much depends on the configuration of the electrodes during bonding. A study of multistack bonds using the non-destructive test is also presented.

MM5.27

POLYIMIDE MEMBRANES AS SUPPORT FOR MICROWAVE CIRCUIT ELEMENTS AND PRESSURE SENSING STRUCTURES. A. Muller, I. Petrini, V. Avramescu, S. Iordanescu, D. Vasilache, G. Simion, N. Nitescu, V. Badilita, D. Dascalu, IMT Bucharest Romania; G. Konstantinidis IESL FORTH Heraklion, GREECE; R. Marcelli, CNR-MMT, Rome, ITALY; G. Bartolucci, Tor Vergata University, Rome, ITALY.

It is the purpose of this paper to present the manufacturing and characterization of polyimide membranes supported microwave and millimeter wave circuit elements as well as pressure sensing structures. Manufacturing of membrane supported microwave and millimeter wave lumped or distributed circuit elements as well as antennas is one of the most exciting last years applications of microtechnologies. As an effect of micromachining, dispersive and radiative losses are reduced and resonance frequencies are substantially increased. The manufacturing of these circuit elements on $1.5\ \mu\text{m}$ thin $\text{SiO}_2/\text{Si}_3\text{N}_4/\text{SiO}_2$ membranes obtained by micromachining of high resistivity silicon has been reported [Katehi, Rebeiz et al., IEEE Trans. on MTT, 43, 1995, p. 534, IEEE MTT-S Digest, 1996, p. 1145, ; A. Müller, I. Petrini, D. Dascalu et al., Proc. of Micromechanics Europe Workshop MME'97, Sept. 1997, pp. 59-62, Proc. of Micromechanics Europe Workshop MME'98, Norway, June 1998, p. 151; European Semiconductors, 11, p. 27, Oct. 1997]. In this paper the manufacturing of $2.2\ \mu\text{m}$ thin polyimide membranes on high resistivity silicon and seminsulating GaAs substrate is presented. Planar inductors and interdigitated capacitors were patterned on these membranes. Mechanical properties (planarity, roughness) of these structures were analyzed using interferometric and AFM techniques. Microwave measurements of polyimide membrane supported microwave circuits were performed in the range 1 - 40 GHz and were compared with those of similar circuits manufactured on bulk SI GaAs as well as on $\text{SiO}_2/\text{Si}_3\text{N}_4/\text{SiO}_2$ membranes. Polyimide membrane supported lumped elements have excellent microwave performances (resonance frequencies increase by a factor of 2 compared with; for the same circuit manufactured on $\text{SiO}_2/\text{Si}_3\text{N}_4/\text{SiO}_2$ membrane the increasing was by a factor of about 1.8 compared with bulk SI GaAs). Compared with the $\text{SiO}_2/\text{Si}_3\text{N}_4/\text{SiO}_2$ sandwich membrane, the polyimide membrane is extremely reliable. This is important especially if a large area is imposed by the device geometry. Polyimide membranes on GaAs substrate were also experimented as support for a gold on chromium resistive pressure sensing element. The variations of $\Delta R/R$ vs. pressure and $\Delta R/R$ vs. temperature are presented. The sensitivity of the element $\Delta R/R\Delta p$ vs. temperature was also measured. This sensing element can be used in high temperature applications. Acknowledgement This research was supported by INCO-COPERNICUS Project No. 977131 MEMSWAVE, financed by the European Community.

SESSION MM6: LIGA

Chair: Eric Peeters

Tuesday Morning, November 30, 1999

Room 313 (H)

8:30 AM MM6.1

MEMS MATERIAL MICROSTRUCTURE AND ELASTIC PROPERTY RESEARCH. Howard R. Last, Naval Surface Warfare Center, Indian Head Division, Indian Head, MD; Ronald Witt, TexSem Laboratories, Inc. Draper, UT; Kevin J. Hemker, The Johns Hopkins Univ, Dept of Mechanical Engineering, Baltimore, MD.

Results from initial efforts to develop a material microstructure - elastic properties relationship for electroplated LIGA nickel will be presented. Microspecimen tensile testing, microstructural characterization including grain size and grain orientation measurements using orientation imaging microscopy (OIM), and finite element modeling results are discussed. The use of mechanical property results and microstructural characterization results in the development of a finite element modeling approach to represent the elastic mechanical response of meso-scale MEMS materials will be highlighted.

8:45 AM MM6.2

EFFECT OF AS-PROCESSED AND ANNEALED MICROSTRUCTURES ON THE MECHANICAL PROPERTIES OF LIGA Ni MEMS. Z.L. Xie¹, D. Pan¹, H. Last² and K.J. Hemker¹; ¹Dept. of Mechanical Engineering, The Johns Hopkins University, Baltimore, MD, ²Naval Surface Warfare Center, Indian Head Division, Indian Head, MD.

Microstructure - mechanical property relations of 200 μm thick LIGA deposited Ni films have been investigated with a combination of cross-sectional microscopy and microsample tensile testing. Measurements of the grain size and morphology of nine different as-deposited films evidenced a predominantly columnar microstructure with significant variations in grain size. These as-deposited microstructural variations were not found to have a significant effect on the measured values of the Young's modulus, yield strength, or ultimate tensile strength (UTS) of these films. By contrast, significant changes in both the microstructure and mechanical properties were observed when these films were annealed at 800°C for 1 hour. Recrystallization and grain growth replaced the columnar grains with large equiaxed grains, which resulted in a dramatic decrease in the tensile strength and corresponding increase in the ductility of the annealed films. LIGA Ni, MEMS, grain, Young's modulus, yield strength, ductility.

9:00 AM MM6.3

NOVEL X-RAY MASKS BASED ON POLY(VINYLDENE FLUORIDE). Kim Kihong, James D. Ross, Miguel H. Wood, Dept of Electrical and Computer Engineering, Louisiana State Univ, Baton Rouge, LA; Harish M. Manohara, J. Bennett Johnston, Sr. Ctr for Advanced Microstructures and Devices, Louisiana State Univ, Baton Rouge, LA.

Using the structural and chemical changes induced in poly(vinylidene fluoride) or PVDF by X-ray irradiation, a novel method to fabricate X-ray masks is developed. Upon X-ray irradiation of PVDF, dehydrofluorination is exhibited and the polymer is directly etched without requiring any reactive chemicals. The irradiated region became carbon rich, and accordingly, the X-ray transmittance of the region increased by a factor of ~ 2.7 . By selectively irradiating the polymer to synchrotron X-rays (energy range: 2 keV to 16 keV), a contrasting pattern of X-ray transmission and absorption regions is created, which is used as an X-ray mask. To achieve selective irradiation, the gold absorber pattern, 1 to 5 micrometers in thickness, is formed directly on the PVDF (~ 500 micrometers thick) surface using optical lithography and either electroplating or wet etching methods. Upon irradiation, the ratio of power transmitted through the transmitting to the absorbing regions is calculated to be in the range of 2.8 (1 micrometer Au absorber and 1.5 GeV electron source) to 14.7 (5 micrometer Au absorber and 1.3 GeV electron source). These X-ray masks are used to produce 3-D relief images in different thicknesses of poly(methyl methacrylate), the standard X-ray resist and satisfactory results are obtained.

9:15 AM MM6.4

FRACTURE BEHAVIOR OF MICRO-SIZED Ni-P AMORPHOUS ALLOY SPECIMENS. K. Takashima, S. Maekawa, M. Shimojo, Y. Higo, Tokyo Institute of Technology, Precision and Intelligence Lab, Yokohama, JAPAN; M.V. Swain, Univ of Sydney, Dept of Mechanical Engineering, AUSTRALIA.

Fracture behavior of micro-sized materials is considered to be different from that of bulk materials, since the surface effect on fracture mechanisms is prominent in micro-sized materials. Therefore, the fracture behavior of micro-sized materials is extremely important to ensure the reliability of micro-sized materials when they are applied to MEMS devices. In this investigation, fracture tests have been performed for micro-sized Ni-P amorphous alloy specimens and the size effect on the fracture behavior has been examined. Cantilever beam type specimens with dimensions of $30 \times 10 \times 50 \mu\text{m}^3$ were prepared from an Ni-P amorphous thin film and notches with various depth from 0.25 to 0.75 a/W (a: notch depth and W: width of the specimen) were introduced by focused ion beam machining. Fracture tests were performed using a newly developed mechanical testing machine for micro-sized specimens. This testing machine is capable of applying static load to micro-sized specimens with a load resolution of 10 μN and a displacement resolution of 5 nm, respectively. Fracture of the specimens occurred in a brittle manner. The apparent fracture toughness values were calculated to be 9-15 $\text{MPam}^{1/2}$, and these values were dependent on the notch depth. The effects of specimen size and that of notch depth on the fracture behavior are discussed.

SESSION MM7: MEMS TRIBOLOGY

Chair: Maarten P. de Boer
Tuesday Morning, November 30, 1999
Room 313 (H)

10:00 AM *MM7.1

SUPPRESSION OF STICTION IN MEMS. C.H. Mastrangelo, Center for Integrated Microsystems, Department of Electrical Engineering and Computer Science, University of Michigan, Ann Arbor, MI.

Because microelectromechanical structures are built with thin films suspended a few micrometers off a substrate, these structures are very compliant and highly susceptible to sticking through surface forces which can catastrophically degrade the manufacturing yield. This paper reviews the physical mechanisms responsible for sticking failures and provides normalized elastic member dimension bounds for prevention of collapse and sticking. Some of the methods developed to suppress stiction failures in MEMS are also discussed.

10:30 AM MM7.2

INVESTIGATION OF WEAR OF MICROELECTROMECHANICAL CONTACTS. C. Cameron Abnet, Brian J. Gally, Stuart Brown, Exponent Inc., Natick, MA.

Microrelays and switches represent an area of increasing research and development. While device characteristics such as switching speed and contact resistance have been studied, relatively little attention has been directed to the fundamental roles played by surface morphology, contact pressure, and wear in device performance. A novel fixture has been developed which allows a microswitch to be actuated with calibrated forces from 1 to 1000 μN at frequencies from 1 to 50 kHz, while monitoring the contact resistance. The contacting surfaces, comprised of a sphere and a plane, can be separated and examined using SEM and interferometric techniques after a selected number of cycles. The geometry of the contacts provides a well characterized distribution of stresses. The fixture was used to make preliminary measurements of gold-gold contact. A steel sphere, 30 μm in diameter coated with 0.5 μm of sputtered gold cyclically contacted a planar silicon substrate with a 0.5 μm sputtered gold layer. Changes in contact resistance and surface morphology as a function of contact pressure and accumulated cycles are discussed.

10:45 AM MM7.3

INVESTIGATION OF THE DEPOSITION AND INTEGRATION OF PROTECTIVE HARD COATINGS FOR MOVING MEMS APPLICATIONS. Gouri Radhakrishnan, Ruby Robertson, Paul Adams and Robert Cole, Technology Operations, The Aerospace Corporation, CA.

Microelectromechanical systems (MEMS) have been identified as a key technology for small-scale satellites, integrated sensors, and intelligent control systems. Using methods developed for highly integrated electronics, mechanical components are co-fabricated on planar wafers and subsequently etched free for mechanical movements in three dimensions. A major design limitation for these systems is their inability to withstand prolonged sliding surface contact. MEMS devices such as micro-gears and motors and permanently actively aligned micro-optics can be fabricated with the current technology, but they fail within minutes of operation. The fundamental problem is that the surface properties of silicon and poly-silicon, two of the most widely used materials for MEMS, are highly unsuitable for moving MEMS devices, resulting in high wear during operation. This work explores the feasibility and benefits of depositing thin, wear-resistant, low-friction coatings on silicon or poly-silicon. To achieve this goal, three-dimensional test silicon microstructures have been fabricated. The deposition of wear-resistant coatings on these test structures using a novel non-line-of-sight laser deposition process is under investigation. In parallel, the integration of the hard coating directly into the MEMS fabrication processes, and the compatibility of the coating with standard silicon processing sequences are being examined. This paper will address issues related to the patterning and fabrication of suitable test microstructures for MEMS, and potential methods to alleviate tribological problems in moving MEMS devices.

11:00 AM MM7.4

SURFACE PASSIVATION FOR REDUCED FRICTION AND WEAR IN SURFACE MICROMACHINED DEVICES. Michael T. Dugger, James A. Ohlhausen, Gerald C. Nelson and Greg A. Poulter, Sandia National Laboratories, Albuquerque, NM.

Micromachines in which normal operation requires intermittent contact of surfaces present unique challenges for surface modification. The surface treatment must result in low surface interaction energy so that adhesive interactions are minimal compared to available actuation forces. Beyond adhesion, long life devices require robust surface treatments that can yield reproducible friction forces combined with long operating life. Aspects of integration with fabrication or back-end-of-line processes impose additional constraints on applicable surface treatment methods. A critical aspect of understanding the role of surface chemistry in micromachine reliability is the need to understand how changes in surface properties affect device operation, i.e. how device performance evolves with time as the nature of the surface changes. In this work, we present results from a micromachined tribology tester designed for quantitative measurement of such phenomena, at a size scale relevant to MEMS. Alkoxysilane monolayers have received much attention as passivation layers for polycrystalline silicon micromachines. Measurements using

our micromachined tribology tester in controlled environments show that film durability is reduced in environments containing water vapor. Penetration of the film by water molecules at defect sites is the proposed mechanism for this degradation. Once the monolayer is damaged in a particular location, additional traction force at this site facilitates accumulation of additional damage and spreading to adjacent areas. These observations suggest that while strongly-bound passivation layers are desirable as release aids, more mobile or replenishable surface treatments are desired for long term operation.

11:15 AM MM7.5

FRICITION MEASUREMENT IN MEMS USING A NEW TEST STRUCTURE. Brian T. Crozier, David F. Bahr, Washington State University, Dept. of Mechanical and Materials Engineering, Pullman, WA; Maarten P. de Boer, Jim M. Redmond, Terry A. Michalske, Sandia National Laboratories, Albuquerque, NM.

Interfacial friction is important to the design, performance, and reliability of MEMS. The magnitude of friction and adhesion depends on device processing history, as well as the humidity of the operating environment. We have designed, fabricated, and initially tested a new MEMS test structure capable of measuring friction as a function of applied normal pressure from 0.2 to 50 MPa, and sliding velocity from 1nm to 1mm per second. Advantages over current friction measurement methods include a very compact device size (relative to comb driven devices), and surface contact characteristics that are more representative of a real device than the point contact methods employing AFM. With its compact size, this device has the potential to be placed on functional MEMS modules for use as an in-situ diagnostic tool. The structure is essentially a cantilever beam with an electrostatically actuated friction pad attached to its free end. Interferometry and laser-reflection techniques are used to quantify the deflection of the driver beam under applied electrostatic loading. Lateral displacements and frictional forces are then inferred from the deflections using modeling based on beam theory. Initial tests indicate that a frictional displacement of 40-50 nm has been achieved. This displacement is significantly greater than the 5 nm achieved by our previous designs, and is sufficient to allow multiple asperity point sliding on polysilicon surfaces. In this study, we employ the device to compare friction coefficients of MEMS structures that have undergone the super-critical CO₂ drying process resulting in hydrophilic surfaces, and structures that have silane monolayer coatings that give the surface a hydrophobic character. Furthermore, the effect of humidity on interfacial friction is evaluated by performing the friction test in an environmental chamber. With this research, we can begin to understand the relationship between processing and operating conditions, friction, and MEMS performance. 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.

11:30 AM MM7.6

SELECTIVE W FOR COATING AND RELEASING MEMS DEVICES. S.S. Mani, J.G. Fleming, J.J. Sniegowski and M.P. de Boer, Sandia National Laboratories, Albuquerque, NM.

Two of the two major problems associated with Si-based MEMS devices are stiction and wear. Surface modifications are needed to reduce both adhesion and friction in micromechanical structures to solve these problems. In this paper, we will present a process used to selectively coat MEMS devices with tungsten using a CVD (Chemical Vapor Deposition) process. The selective W deposition process results in a very conformal coating and can potentially solve both stiction and wear problems confronting MEMS processing. The selective deposition of tungsten is accomplished through silicon reduction of WF₆, which results in a self-limiting reaction. The selective deposition of W only on polysilicon surfaces prevents electrical shorts. Further, the self-limiting nature of this selective W deposition process ensures the consistency necessary for process control. The selective tungsten is deposited after the removal of the sacrificial oxides to minimize process integration problems. This tungsten coating adheres well and is hard and conducting, requirements for device performance. Furthermore, since the deposited tungsten infiltrates under adhered silicon parts and the volume of W deposited is less than the amount of Si consumed, it appears to be possible to release stuck parts that are contacted over small areas such as dimples. Results from tungsten deposition on MEMS structures with dimples will be presented. The effect of wet and vapor phase cleans prior to the deposition will be discussed along with other process details. Endurance of the W coating is important, especially in applications where wear may occur. Tungsten CVD is used in the integrated-circuit industry, which makes this approach manufacturable. The United States Department of Energy under contract DE-AC04-94AL85000 supported this work. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy.

11:45 AM MM7.7

A NOVEL INTEGRATED MEMS PROCESS USING FLUOROCARBON FILMS DEPOSITED WITH A DEEP REACTIVE ION ETCHING (DRIE) TOOL. A.A. Ayon, D.-Z. Chen, R.A. Braff, R. Ghodssi, C.-C. Lin, M.A. Schmidt, Massachusetts Institute of Technology, Microsystems Technology Laboratories, Cambridge, MA; H.H. Sawin, Massachusetts Institute of Technology, Department of Chemical Engineering, Cambridge, MA.

Fluorocarbon films are useful as antistiction films for suspended structures [1], for orienting layers of liquid crystals [2], as corrosion resistant, biocompatible materials [3], and as sacrificial layers exploiting their low Young's modulus [4]. Furthermore, the recent introduction of copper interconnects in VLSI manufacturing has renewed the interest in fluorocarbon films due to their low dielectric constant. In the interest of building a complete database for these films, we report a designed experiment with a 4-variable matrix to fully characterize deposited fluorocarbon films using C4F8 as the feed gas. In contrast to the work of Man et al [1], rather than concentrating in a single set of deposition conditions, we generated and present response surfaces that permit us to optimize the characteristics of deposited films. Also, compared to the aforementioned group [1] and those of Limb et al [3] and of Jansen et al [4], we have utilized a high density plasma tool obtaining faster deposition rates which measurements show to be as high as 0.12 mm/min. Furthermore, we demonstrate the in-situ microfabrication of electrostatic actuators that exhibit the corresponding passivation film. We also demonstrate the utilization of these films as a masking material for MEMS applications. Additionally, to demonstrate the viability of these films as masking materials we measured the selectivity to silicon in DRIE, from 25:1 to 34:1, and tested a triple nested mask arrangement using silicon dioxide, fluorocarbon film and positive photoresist as depicted in figure 4. References. 1 P. F. Man, B. P. Gogoi and C. H. Mastrangelo, J-MEMS, 6(25), March 1997. 2 E. Kay, J. Coburn and A. Dilks, Topics in Current Chemistry, 94(1), 1980. 3 S. J. Limb, K. K. Gleason, D. J. Edell and E. F. Gleason, J. Vac. Sci. Technol. A 15(4), 1997. 4 H. V. Jansen, J. G. E. Gardeniers, J. Elders, H. A. C. Tilmans and M. Elwenspoek, Sensors and Actuators A, 41-42 (136) 1994.

SESSION MM8: NEW CHARACTERIZATION TECHNIQUES / MEMS DEVICES

Chair: Jeffrey J. Sniegowski
Tuesday Afternoon, November 30, 1999
Room 313 (H)

1:30 PM *MM8.1

COMPUTER MICROVISION FOR MEMS. Dennis M. Freeman, Massachusetts Institute of Technology, Electrical Engineering & Computer Science Department, Cambridge, MA.

We have developed a versatile instrument for in situ measurement of motions of MEMS. Images of MEMS are magnified with an optical microscope and projected onto a CCD camera. Stroboscopic illumination is used to obtain stop-action images of the moving structures. Stop-action images from multiple focal planes provide information about 3D structure and 3D motion. Image analysis algorithms determine motions of all visible structures with nanometer accuracy.

Hardware for the system includes the microscope, CCD camera and associated frame grabber, piezoelectric focusing element, and a modular stimulator that generates arbitrary periodic waveforms and synchronized stroboscopic illumination. These elements are controlled from a Pentium-based computer using a graphical user interface that guides the user through both data collection and data analysis. We demonstrate use of the system to obtain translation measurements with nanometer resolution, well below the wavelength of light.

2:00 PM MM8.2

DYNAMICS OF MEMS MICROENGINES DETERMINED BY OPTO-ELECTRONIC LASER INTERFEROMETRY. Gordon C. Brown, Ryszard J. Pryputniewicz, Worcester Polytechnic Institute, Center for Holographic Studies and Laser Micro-MechaTronics, Worcester, MA; Maarten P. de Boer, Sandia National Laboratories, Intelligent Micromachines Department, Albuquerque, NM.

MEMS require advancements in dynamic test methodologies to enable refinement and optimization of designs. We have developed an optoelectronic laser interferometry methodology (OLIM) for rapid characterization of MEMS' motion dynamics. In this study, electrostatic comb drive actuated microengines comprised of a 64 μm diameter drivegear and a 250 μm outputgear (loadgear) were characterized dynamically. The actuators were driven by synchronized input signals generated by an arbitrary waveform generator. Two

different input signals were used: 1) a four-step square wave input and 2) an optimized input signal calculated from an analytical model. Out-of-plane wobble on rotating microengines was measured as a function of the input signals. The out-of-plane motions during the four-step input ranged from zero to ± 600 nm and from zero to ± 450 nm for the drive and output gears, respectively. For the optimum input, the out-of-plane motions ranged from zero to ± 700 nm and from zero to ± 300 nm for the drive and output gears, respectively. We believe that the smaller out-of-plane motions of the loadgear for the optimized signal are due to improved kinematics and kinetics leading to more continuous rather than impulsive forces (e. g., frictional, inertial) acting on the outputgear. Similar effects are believed to influence the dynamics of the drivegear. The wobble was also observed to depend on the angular position in the cycle in both cases. This can be related to the forces exerted on the drive gear by the pin during the rotational cycle. In detail, microengine wobble is seen to exhibit inconsistencies while under nominally the same operating conditions. This may be related to fabrication tolerances and to a clamping problem in the actuator in the present design. By understanding the details of MEMS performance in three-dimensions, we can make concrete suggestions for improvements in the designs of MEMS.

2:15 PM MM8.3

EFFECT OF BUFFER ELECTRODES IN CRYSTALLIZATION OF ZINC OXIDE THIN FILM FOR THIN FILM BULK ACOUSTIC WAVE RESONATOR. Y. Yoshino, N. Tsukai, K. Inoue, M. Takeuchi, T. Nomura, T. Makino and S. Arai, Murata Mfg. Co., Ltd., R&D Division, Shiga, Yasu, JAPAN, T. Hata, Graduate School of Natural Science & Technology, Kanazawa University, JAPAN.

A thin film bulk acoustic wave resonator (TBAR) has been fabricated using a ZnO thin film on a SiO₂ diaphragm by MEMS techniques. The ZnO/SiO₂ structure TBAR can be designed to cancel a temperature coefficient of frequency (TCF) by the ZnO/SiO₂ thickness ratio, because the TCF of ZnO is negative, and that of SiO₂ is positive. The ZnO thin film on the SiO₂ shows a c-axis orientation almost equivalent to that of the ZnO thin film on a glass substrate by RF sputtering. However, the crystallinity of the ZnO thin film is influenced by the crystallinity of the lower electrode that is formed on the SiO₂ diaphragm. ZnO thin films have been deposited on Au/Cr, Au/NiCr and Au/Ti. The Au/Ti/ZnO/Au/Ti/SiO₂ structure TBAR shows the best resonant characteristics in this experiment. The resonant characteristics of the TBAR depend on the crystallinity of the ZnO thin film. The resonant resistance of the TBAR at 200MHz using a Au/Ti lower electrode is improved by about 8% less than that using a Au/Cr electrode. The x-ray diffraction result shows that the crystallinity of ZnO is greatly influenced by the crystallinity of the lower electrode. The buffer layer has an influence on both the crystallinity of the ZnO thin film and the resonant characteristics of the TBAR through the Au electrode.

2:30 PM MM8.4

MEMS BASED THIN-FILM SCANNING MICROCALORIMETER FOR NANOLITER LIQUID CALORIMETRY. E. Olson, M.V. Efremov, S.L. Lai, A.T. Kwan, T. Wisleder, M. Zhang and L.H. Allen, Univ of Illinois at Urbana-Champaign, Dept of Materials Science, Urbana, IL.

Our group has developed a MEMS thin-film calorimeter, which can measure less than a nano-Joule of heat. Our group has used these thin film calorimeters to demonstrate melting point depression for nanometer-sized Al and Sn particles [1, 2]. While the behavior of nano-scale particles has recently received a great deal of interest, little attention has been paid to the behavior of fluids at small scales. In this paper we discuss a new design of an ultra-sensitive scanning thin-film calorimeter to investigate the thermal properties of liquids, specifically water. In this design, a nickel strip is used to simultaneously heat the sample via Joule-heating and measure the temperature. A silicon nitride membrane supports the sample electrically isolates the sample from the heater. The sample chamber is fabricated via anisotropic etching of Si. Here we present calorimetric measurements performed on ultra-small volumes of water. While traditional calorimetry requires sample sizes greater than 0.3 mL, this MEMS thin film calorimeter is used to investigate the heat of vaporization and specific heat of single droplets of water less than 100 nanoliters in volume. This device shows promise for materials science applications for investigations of material on the small scales as well as for calorimetric measurements for biology. References: [1] S. L. Lai, J.Y. Guo, V. Petrova, G. Ramanath and L.H. Allen, Size-dependent melting properties of small tin particles: Nanocalorimetry measurements, Phys. Rev. Lett. 77, 99 (1996) [2] S. L. Lai, J. Carlsson and L. H. Allen, Melting Point Depression of Al Clusters During the Early Stages of Film Growth: Nanocalorimetry Measurements, Appl. Phys. Lett. 72, 1098 (1998)

3:15 PM *MM9.1

MATERIALS ASPECTS OF MEMS PACKAGING.

Mehran Mehregany, Department of Electrical Engineering and Computer Science, Case Western Reserve University, Cleveland, OH.

Technical advances over the last decade have transformed the field of solid-state transducers (sensors and actuators) into what has become known as microelectromechanical systems (MEMS). In the most general form, MEMS consist of mechanical microstructures, microsensors, microactuators and electronics integrated in the same environment (i.e., on a silicon chip). While MEMS addresses the miniaturization of mechanical systems, it also opens a new paradigm for designing mechanical devices and systems. Miniaturization of mechanical systems promises unique opportunities for new directions in scientific and technological progress. Additionally, microfabrication technology enables integration of mechanical and electrical components which may individually perform simple tasks but in combination can accomplish complicated functions. A key challenge in application of MEMS technology is the packaging aspect. Packaging must provide for device protection from the environment, while allowing the device to interact with the environment for a desired purpose related to sensing and/or actuation. Accommodating these two often contradicting requirements while maintaining cost realism is at the core of the MEMS packaging challenge. An overview of MEMS packaging requirements and solutions will be presented with emphasis on materials issues.

3:45 PM MM9.2

LOW TEMPERATURE WAFER LEVEL GOLD-GOLD THERMOCOMPRESSION BONDING CHARACTERIZATION.

C.H. Tsau, Department of Materials Science and Engineering; M.A. Schmidt, Department of Electrical Engineering and Computer Science; S.M. Spearing, Department of Aeronautics and Astronautics, Massachusetts Institute of Technology, Cambridge, MA.

Low temperature, wafer level bonding allows the integration of multi-wafer MEMS structures at the end of a processing sequence, in which diffusion-controlled degradation of previously deposited materials may be of concern. Moreover, wafer level bonding can be used to protect delicate structures from post-processing handling damage such as die-sawing. One of the techniques being developed for this purpose is low temperature, gold to gold thermocompression bonding. In this study, gold is e-beam deposited onto patterned silicon wafers, which are subsequently aligned and bonded to create intermittently bonded parallel strips between the wafers. By orientating the bonded strips parallel to the direction of crack propagation, the reduced bonded area ensures crack propagation along the bond line. 8mm-wide specimens with a central notch through 60% of the thickness of one of the wafers are created by die-sawing. Specimens are loaded in 4-point bending until crack propagation occurs along the bond line. This technique allows the calculation of strain-energy release rate from the critical load for crack propagation, independent of the initial crack size in the specimen. The technique has been demonstrated to be reproducible for specimens bonded at 300°C, which have a measured fracture resistance of 31 J/m². This test method allows exploration of factors affecting bond strength, such as bonding temperature, surface roughness and cleaning protocols. These factors and details of the test technique and initial results will be presented.

4:00 PM MM9.3

ATOMISTIC ASPECTS OF WAFER BONDING: SIMULATIONS OF THE EFFECT OF MOISTURE ON INTERFACE FORMATION.

Stephen H. Garofalini, David A. Litton, Dept. of Ceramic and Materials Engineering, Rutgers University, Piscataway, NJ.

An oxide surface layer on silicon wafers enables hydrophilic bonding, in which hydrogen bonding is enhanced by the presence of surface hydroxyls (silanol). Exposing these surfaces to moisture is often used to maximize hydrogen bonding between wafers. Slight pressure is usually sufficient to cause enough H-bonding such that the wafers adhere well for handling. However, subsequent annealing is required to remove this moisture and form the strong siloxane bonds needed between wafer surfaces. Molecular Dynamics simulations have been used to evaluate the effect of water molecules and hydroxyls on the siloxane bond formation across the interface at different temperatures and surface separation distances. Results show the inhibiting effect of moisture on the early, low temperature formation of siloxane bonds and the beneficial effects of moisture at higher temperatures. The mechanisms of siloxane bond formation under these various conditions will also be presented.

4:15 PM MM9.4

PACKAGING MEM SENSOR ARRAYS. T.F. Marinis, D.A. Fulginiti and H.G. Clausen, Draper Laboratory, Cambridge MA.

Many applications of MEM sensors require hermetic or vacuum packaging of sensor clusters. For example, multiple gyroscopes or accelerometers are fabricated on a single chip to improve alignment and stability of input axes or increase the dynamic range of instruments. Chemical sensors are fabricated as large arrays to both improve selectivity and increase the number of species that can be detected. Still larger arrays of sensors must be packaged for hydrophobic and bolometer imaging devices. All of these applications place a demanding combination of requirements on the sensor package. The electrical outputs of the sensor array must be well isolated from each other as well as power and excitation signals, while parasitic capacitance is minimized. The package must also be capable of being evacuated and sealed to achieve a pressure of 5 millitorr with a leakage rate below 10^{-11} [std cc sec⁻¹]. Finally, the package must be compact and low cost to realize these same attributes of the MEM sensor. This paper describes a packaging approach that is based on low temperature cofired ceramic materials. This technology meets the packaging requirements of sensor arrays and is well suited to the research environment in which the sensor design is continually evolving.

4:30 PM MM9.5

MESO-SCALE BONDING OF DISSIMILAR MATERIALS FOR THE PRODUCTION OF PACKAGED MICRO-ELECTRO-MECHANICAL SYSTEMS. K. Rajan, Rensselaer Polytechnic Institute, Troy, NY; M.A. Imam, Naval Research Laboratory and D.J. Nagel, The George Washington University, Washington, DC.

The production of MEMS often requires robust bonding of dissimilar materials, for example, between silicon and glasses or polymers. The packaging of MEMS of all types also requires bonding of the devices to polymeric materials or ceramics, resulting in interfaces tailored with appropriate mechanical, thermal and electrical properties. The sealing of MEMS within evacuated packages is necessary for operation resonant and other devices. This necessitates the bonding of package to substrates, sometimes at the wafer level for reasons of cost and reliability. In short, bonding of materials is central to the production and packaging of MEMS for both prototypes and mass production. The structure of the interface at the 'meso' or micro- structural scale has a profound effect on the resulting integrity of MEMS devices. Using a system that has been designed for cleaning and joining of dissimilar materials germane to MEMS and materials used to package MEMS, we present a microstructural analysis of some of the fundamental metrics controlling bonding at a meso scale in MEMS. These results are discussed in the broader context of reviewing the physical and chemical means, which have been employed for the preparation of surfaces and joining of dissimilar materials within MEMS devices and their packages.

4:45 PM MM9.6

FLUXLESS MICROJOINING BY Au-In-Ni ISOTHERMAL SOLIDIFICATION. Marc Waelti, Niklaus Schneeberger, Oliver Brand, Henry Baltes, ETH Zurich, Physical Electronics Laboratory, Zurich, SWITZERLAND.

This paper reports on a reliable, fluxless microjoining technique based on isothermal solidification. The microjoining technique has been applied to the assembly of a smart infrared sensor system. The bonding performed at temperatures below 200°C did withstand subsequent soldering cycles with peak temperatures of 235°C. The demand for smaller, lighter, faster, and smarter devices in microelectronics and microelectromechanical systems (MEMS) challenges the microjoining technologies. Especially smart MEMS often require a sequence of joining steps to build the mechanical systems and to provide the mechanical and electrical interconnections. However, reliable joining sequences are difficult to achieve using standard solder technologies. These challenges can be met using isothermal solidification. In this technology, a bond is formed between high and low melting metals. To achieve the bond, a temperature slightly higher than the lowest melting point of the components is applied. Metallic phases are formed by solid-liquid interdiffusion and the joint solidifies isothermally. As such a joint withstands higher temperatures as applied for bonding, subsequent bonding steps can be realized.

To demonstrate the potential if this microjoining scheme, smart thermoelectric infrared (IR) systems have been chosen. The fully CMOS-compatible sensors are manufactured using an industrial 1 μ m CMOS process. An optical silicon filter is directly bonded onto the sensor die by means of isothermal solidification. Further packaging steps include chip-on-board and SMT assembly. The Au-In-Ni system has been chosen for the isothermal solidification. Gold (Au) is often used for wafer bumping and nickel (Ni) is commonly applied for lead frame plating. Indium (In) offers a low melting point of 157°C. To

prepare for bonding, an Au frame has been electroplated onto the sensor die and a corresponding Ni/In pattern onto the filter. Bonding was then performed using different temperature and time schedules. Phase formations and transformations were analyzed using light and scanning electron microscopy. Long term stability was explored by thermal aging at 160°C for 1000 h. To investigate the resistance against subsequent SMT soldering, samples were subjected to reflow-soldering cycles.

SESSION MM10/V5: JOINT SESSION: THIN FILMS FOR APPLICATIONS IN MEMS

Chair: Richard Vinci
Wednesday Morning, December 1, 1999
Room 306 (H)

8:30 AM MM10.1/V5.1

WAFER SCALE TESTING OF MEMS STRUCTURAL FILMS. Brian J. Gally, C. Cameron Abnet, Stuart Brown, Exponent, Inc., Natick, MA.

Biaxial modulus and residual stress of silicon nitride and polysilicon films on silicon substrates were measured at multiple (discrete) locations across individual wafer surfaces using a versatile bulge testing method. An array of 0.5 μ m thick silicon-rich silicon-nitride membranes were fabricated across the surface of 100 mm diameter wafers. In-plane dimensions of the membranes were 1 mm square. Some of these wafers were coated with an additional 2 μ m thick layer of polysilicon, forming a composite membrane. Material parameters of the films were extracted by measuring the deflection of the silicon-nitride and composite membranes under controlled pressure. Pressures from 0 to 30 psi were applied across the membranes while the deflected shapes of the membranes were measured using a white-light interferometer. Numerical analysis of the pressure-deflection behavior of the silicon-nitride and composite membranes enabled the biaxial modulus and residual stress of the films to be mapped over the wafer surface with a sensitivity of better than $\pm 5\%$. Results from wafers fabricated at three foundries are presented and compared.

8:45 AM MM10.2/V5.2

FATIGUE BEHAVIOR OF THIN SILVER FILMS INVESTIGATED BY DYNAMIC MICROBEAM DEFLECTION. R. Schwaiger, O. Kraft, Max-Planck-Institut f. Metallforschung, and Institut f. Metallkunde, University of Stuttgart, GERMANY.

It is now well-known that the mechanical behavior of thin films differs from that of their bulk counterparts. For instance, it has been found both experimentally and by modeling that the flow stress of thin films is higher and varies inversely with the film thickness and the grain size. This can be explained by both dimensional and microstructural constraints on dislocation movement, which might also affect the fatigue behavior of thin film materials. In this paper we describe a new method that allows the investigation of high cycle fatigue behavior of materials with small dimensions. In particular, fatigue properties of thin Ag films of varying thicknesses were investigated by dynamic microbeam deflection utilizing a commercial nanoindentation system. Silicon dioxide microbeams were fabricated by conventional integrated circuit techniques and a silver film was sputter-deposited onto the patterned wafer. The microbeams were cyclically deformed and the changes in mechanical behavior monitored. Sudden decreases in beam stiffness were observed during the fatigue experiments. The stiffness decrease was related to damage formation in the thin film, including voids, cracks, and extrusions. Several microscopic techniques were applied to characterize the microstructure of the beam specimens. The extrusions appeared as narrow ribbons of squeezed-out material located in the interior of single grains. The height of the extrusions was in the range of the film thickness. Voids were found to extend from the film-substrate interface towards the surface. Based on these observations, we suggest a qualitative explanation of extrusion growth in terms of dislocation glide and annihilation associated with the production of point defects.

9:00 AM MM10.3/V5.3

BENDING RESPONSE OF A 100 NANOMETER THICK FREE STANDING ALUMINUM CANTILEVER BEAM. M.A. Haque and T.A. Saif, Dept. of Mechanical & Industrial Engineering, University of Illinois at Urbana-Champaign, IL.

This study investigates the behavior of a free standing thin metal film under bending loads using a micro-electro-mechanical (MEMS) systems device. A 2.1 micron wide, 11.3 micron long and 100 nanometer thick cantilever beam specimen was fabricated from 99.99% pure evaporated Aluminum. The MEMS device is a comb drive actuator fabricated separately from the specimen. The actuator has a force resolution of 1 nano-Newton and has a probe that can deflect

the specimen up to 10 micron by point loading. Two cycles of loading and unloading were carried out. The experiment was observed in-situ under an optical microscope and was video taped for data acquisition. Plastic deformation was observed in both the loading cycles. The yield stress estimated from the load displacement profile is 841 MPa which is 49 times higher than the published data for pure bulk Aluminum. To the best of our knowledge, this is the first study to report a bending test of a 100 nanometer thick free standing film showing a significantly large yield stress compared to its bulk counterpart.

9:15 AM MM10.4/V5.4

FILM STRESS INFLUENCE OF BILAYER METALLIZATION ON THE STRUCTURE OF RF MEMS SWITCHES. R.E. Strawser, R. Cortez, Air Force Research Laboratory; M.J. O'Keefe, University of Missouri-Rolla, Dept. of Metallurgical Engineering; K.D. Leedy, J.L. Ebel, Air Force Research Laboratory, Wright-Patterson AFB, OH; H.T. Henderson, University of Cincinnati, Dept of Electrical Engineering, Cincinnati, OH.

The performance of microelectromechanical switches (MEMS) is highly dependent on the switches' constituent materials. The switch material must be able to provide both structural integrity and high electrical conductivity. Cantilever beams, microbridges, and membranes represent typical MEMS structures used in microwave/millimeter wave applications. In this study, cantilever and bridge microswitches were fabricated on GaAs substrates using bilayers of titanium and gold metallization in with the total thickness was held constant at 1.5 μm but the thickness of gold varied from 0.5 μm to 1.5 μm . The lengths of the cantilevers were varied from 300 to 500 μm and the bridge lengths were varied from 600 to 800 μm while the cantilever and bridge beam width was fixed at 50 μm . The switch metals were deposited via either e-beam evaporation or electroplating. Qualitative comparisons of the topography and the resulting 'stiffness' of the released switches were made using focused ion beam/scanning electron microscopy. Thin film stress measurements using laser reflectometry of the various ratios of titanium/gold metallization films deposited on bare wafers revealed an increasing intrinsic tensile stress as the gold ratio increased. The upward deflection of the gold dominated cantilever beams corroborated this trend of increased tensile stress. A discussion of the observed microswitch structure within the context of the measured film stresses will be presented and recommendations for future metallization studies will be made.

9:30 AM MM10.5/V5.5

STUDY OF STRESS IN Pt/PZT/Pt/Ta ON SILICON WAFER FOR FABRICATION OF PZT MEMS PRESSURE SENSOR. Eugene Zakar, Madan Dubey, Brett Piekarski, Richard Piekarz, John Conrad, and Robert Widuta, US Army Research Lab, Sensors and Electron Devices Directorate, Adelphi, MD.

Stress was studied in piezoelectric PZT ($\text{Pb}(\text{Zr,Ti})\text{O}_3$) films using a commercial stress analyzer which measures warpage with a laser-reflection system. PECVD oxide, nitride, and oxide/nitride/oxide (47 to 255 nm thick films) were deposited at 250°C on silicon (100) wafers (3-inch diameter). The oxide and nitride films were heat treated in furnace of the stress analyzer under nitrogen from RT-860°C-RT at a rate of 25 and 30°C/min with in-situ stress measurement. For as deposited oxide layers (90 and 255 nm thick), measured stress was compressive (-351 and -579 MPa respectively), and changed from -579 to -186 MPa after heat treatment. Similarly stress for (47 and 165 nm thick) nitride was compressive (-78 and -90 MPa respectively) which changed from -78 to +520 MPa after heat treatment. Ta (20 nm) and Pt (170 nm) were sputter deposited on the above oxide and nitride films at 100°C with (Ta-160/Pt-240) nm/min deposition rate for bottom electrodes. The average stress in as deposited Ta/Pt on nitride was -570 MPa, which changed to average $+3.5 \times 10^6$ MPa after RTA annealing at 700°C for 60 sec. Stress due to the (Ta/Pt) films, deposited on 90 and 255-nm thick oxide, were -576 and -785 MPa respectively. The RTA treatment further changed the stress from -576 to $+2.8 \times 10^6$ MPa and -785 to $+2.2 \times 10^6$ MPa. When (Ta/Pt) films were deposited on a sandwich of (Oxide-213/Nitride-203/Oxide-200 nm) films, the measured stress was -1089 MPa which changed to $+2.7 \times 10^6$ MPa after similar RTA treatment. Sol-gel deposited PZT thin (500-nm) film on Ta/Pt electrodes created an average +580 MPa stress. Several PZT MEMS static pressure sensors were fabricated using dry etching process. Performance of the sensors was measured by capacitance method, values varied from 423 to 907 pF. The effect of the stress on capacitance values was also studied.

9:45 AM MM10.6/V5.6

THICKNESS EFFECTS ON MICROSTRUCTURE AND TRANSFORMATION BEHAVIOR OF COBALT THIN FILMS. Heiko Hesemann¹, Peter Müllner², and Eduard Arzt¹;

¹Max-Planck-Institut für Metallforschung and Institut für

Metallkunde, Universität Stuttgart, Stuttgart, GERMANY; ²Institut für Angewandte Physik, ETH-Zürich, SWITZERLAND.

Martensitic transformations are important mechanisms with respect to shape memory alloys, which are used, e.g., as thin films in microactuators. In order to understand the influence of film thickness on the martensitic transformation, we study the transformation behavior in cobalt thin films. Cobalt is particularly useful for this purpose owing to the simple crystallography of its martensitic transformation. The austenite and martensite phases are face centered cubic and hexagonal close packed, respectively, and the habit plane is the close packed plane in both phases. The martensitic phase does not contain any internal structure such as twins. Co-films of 0.2 μm to 3.0 μm thickness have been sputter deposited on Si substrates. The films have been characterized by electron backscattered diffraction (EBSD), X-ray diffraction and wafer curvature measurement. Upon ongoing thermocycling, the martensitic transformation is repeatedly found in 3.0 μm thick films. In these films, the microstructure changes during cycling and also during isothermal annealing from a strong fiber texture to a ring texture. A stress drop in heating as well as in cooling accompanies the martensitic transformation. Neither transformation nor texture change occur at film thickness 0.2 μm . It is concluded that the thickness as critical size parameter strongly affects the mutual interaction of structural evolution and martensitic transformation in thin films.