### Wednesday Afternoon, October 27, 1999

## The Science of Micro-Electro-Mechanical Systems Topical Conference

#### Room 620 - Session MM-WeA

#### **Micro-Science and Tribology**

Moderator: C. Zorman, Case Western University

## 2:00pm MM-WeA1 Micro-Science and Tribology, L. Lin, The University of Michigan INVITED

Microelectromechanical Systems (MEMS) have emerged as an interdisciplinary field in recent years and encompassed a wide range of scientific and engineering areas such as electrical engineering, mechanical engineering, material sciences, physics and chemistry. Tribological issues dealing with adhesion, abrasion, corrosion and erosion are generally encountered in macroscopic machinery and are inevitable problems for micro-science. The technological and economical impacts of tribological issues in MEMS demand fundamental understanding and characterization in material development, design, processing and testing of microstructures. Micro devices that are fabricated by IC (Integrated Circuit) processes represent very different surface features than those macro structures fabricated by conventional mechanical manufacturing processes. It is important to study the micro-science of tribology and investigate the tribological effects in the microscale for optimal design and processing of MEMS. This talk will present several important tribological effects, including surface roughness of the microstructures to the nucleation of micro thermal bubbles, to the mechanical properties and to the optical properties of microstructures. The causes and solutions of the surface force induced sticking failures in MEMS will be discussed. A review of MEMS micromachining processes describing key manufacturing steps is followed by practical engineering examples. Future research directions will be proposed in the conclusion.

# 2:40pm MM-WeA3 Deposition, Characterization and Degradation of Vacuum-deposited Fluorinated Alkylsiloxane Films, *T.M. Mayer*, *M.P. de Boer*, *N.D. Shinn*, *T.A. Michalske*, Sandia National Laboratories

We deposit monolayer films of fluorinated alkylsiloxanes by a chemical vapor deposition process using C@sub 8@F@sub 13@H@sub 4@SiCl@sub 3@ and H@sub 2@O. Films are formed under well controlled conditions of reactant exposure and temperature, from 25 - 300@super o@ C. Using insitu ellipsometry and quartz crystal microbalance techniques, we show that film thickness is self limiting at approximately one monolayer due to coverage dependent adsorption of precursors. Adhesion measurements of micromechanical structures coated with these films show typical adhesion energies of ~20 µJ/m@super 2@ at low humidity conditions. Exposure to high humidity environments for long periods results in degradation of the films and stronger adhesion. We postulate that this degradation is related to defects in the films, which are susceptible to hydrolysis by adsorbed water. We examine this postulate by measuring adhesion for varying film coverage, and by examining the morphology of freshly deposited and aged films by atomic force microscopy. Friction measurements correlated to film structure and history are examined as well in micromechanical test devices using structures coated with these films.

#### 3:00pm MM-WeA4 Vapor-Phase Lubricants: Nanometer-scale Mechanisms and Applications to Sub-micron Machinery, M. Abdelmaksoud, B. Borovsky, J. Krim, North Carolina State University

The concept of lubricating high temperature bearing surfaces with organic vapors which react with a surface to form a solid lubricating film has existed for at least forty years, with substantial efforts beginning in the 1980's and continuing to the present day. While vapor-phase lubricants have primarily been studied within the context of macroscopic system performance, they may well prove to be of critical importance to tribological performance in sub-micron mechanical systems as well: The vapor phase may ultimately prove to be the most effective, if not only, means to deliver and/or replenish a lubricant on account of the submicron scale of the device itself. In order to investigate the viability of vapor-phase lubrication for MEMS applications, we have studied the molecular scale properties of a number of known or proposed vapor-phase lubricants in controlled environments and well-defined contact geometries.A first study involves Auger Spectroscopy and Quartz Crystal Microweighing investigations of the known lubricant TBPP as it reacts with an iron film surface prepared in ultra-high vacuum conditions. Confirming prior conjecture, we observe that exposure of iron to TBPP vapors results in a rigidly adhering film with a graphitic carbon component which presumably

is the lubricating component. With the intent of modelling actual MEMS contacts, we have also constructed a simple nanomechanical system consisting of a Scanning Tunneling Microscope tip dragging on the surface of a Quartz Crystal Microbalance electrode. This system allows us to monitor lubricant performance in realistic sliding conditions. Of the systems which we have observed to date, those films which are associated with the greatest decreases in friction have also been the quickest to wear away due to the rubbing action of the STM/QCM combination. Work is now in progress to study the effect of these vapor-phase lubricants on actual MEMS devices, namely comb motors.

### 3:20pm MM-WeA5 Adhesion Performance of Silane Coupling Agents at High Humidity Levels, *M.P. de Boer*, *T.M. Mayer*, *T.A. Michalske*, *R.W. Carpick*, Sandia National Laboratories; *R. Maboudian*, *U. Srinivasan*, University of California, Berkeley

We have measured the effect of humidity on autoadhesion of polycrystalline silicon cantilever beams fabricated by surface micromachining, and coated with silane coupling agents. To make the measurements, we designed and constructed an environmental microprobing station with interferometric capability, and automated the system to enable measurement of beam deflections in-situ. We quantified adhesion by applying a fracture mechanics equilibrium to each adhered beam. For both ODTS (C@sub 18@H@sub 37@SiCl@sub 3@) and FDTS (C@sub 8@F@sub 17@C@sub 2@H@sub 4@SiCl@sub 3@) coatings, the effect of relative humidity (RH) is negligible for RH up to approximately 80%. For ODTS coatings at 99% RH after a 40 hour exposure, adhesion increases only moderately by a factor of two. For FDTS coatings at 90% RH, adhesion increases dramatically by a factor of 100 after seven hours, with further subsequent increases at higher RH values. This is a surprising result, considering that FDTS has a higher contact angle with water than does ODTS, and exhibits lower adhesion at low RH. We believe that defect formation is responsible for the adhesion increase. To support this assertion, we conducted water absorption experiments and obtained atomic force microscopy images revealing agglomerated coupling agent on films exposed to high RH. ODTS is less susceptible to this mechanism than FDTS because of its greater chain length and smaller chain diameter. Our results contrast with experiments on fatty acid monolayers using the surface force apparatus, where uniform swelling of the film is responsible for a monotonic adhesion increase with RH.

3:40pm MM-WeA6 Adhesion Properties of Gold-on-Gold Microswitch Contacts, S. Majumder, N.E. McGruer, G.G. Adams, P.M. Zavracky, R.H. Morrison, Northeastern University; J. Krim, North Carolina State University Electrostatically actuated microswitches have been developed at Northeastern University. As part of this effort, gold-on-gold microswitch contacts have been studied on the basis of electrical measurements, surface analysis, and an analytical model of the contacts.@footnote 1@ Measurements show that the turn-off voltage of the switch is often substantially smaller than the turn-on voltage, an effect which is not predicted by an electromechanical model of the actuation mechanism. Also, contact stiction is a dominant mode of eventual switch failure. Motivated by these observations, we extend our study of contacts to include adhesive surface forces at the contact interface. We consider the applicability of the JKR and DMT surface force models.@footnote 2@ to our problem. We examine the validity of these models through various measurements. Under typical operating conditions, the contact force is approximately 40  $\mu$ N, the spring force which returns the switch to the off position is 150  $\mu$ N, and the adhesion force (minimum spring force required to turn off the switch) usually ranges from 10-50  $\mu N$  for a major portion of the switch lifetime (10@super 4@ - 10@super 6@ switching cycles). Failure by stiction is preceded by a gradual increase in the adhesion force. The adhesion force has a strong (inverse) correlation with the contact resistance, and some correlation with the maximum applied contact force during the on-cycle. Other results that are compared with the model include the variation of contact resistance with contact force during loading and unloading, the contact resistance when the switch just turns on and off, and the effect of loading history. @FootnoteText@ @footnote 1@ S. Majumder, N.E. McGruer, P.M. Zavracky, G. G. Adams, R. H. Morrison, J. Krim, Transducers '97, Chicago, IL (1997). @footnote 2@ M. D. Pashley, J. B. Pethica, D. Tabor, Wear, vol. 100, pp. 7-31, 1984. .

# 4:00pm MM-WeA7 Environmental Effects on the Tribological Behavior of Silane-Treated Micromachines@footnote 1@, M.T. Dugger, J.A. Ohlhausen, G.A. Poulter, Sandia National Laboratories

Reproducible performance of silicon surface micromachined devices having contacting surfaces in relative motion requires that contact surfaces

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maintain uniform friction coefficient over the useful life of the device. High yield fabrication of such structures also requires that the moving surfaces are physically free from other surfaces after the final manufacturing step. Several methods are available to produce hydrophobic surfaces on silica at the conclusion of manufacturing, so that capillary forces do not pull structural elements into contact. These may also favorably affect the friction coefficient and wear characteristics of the treated surfaces. However, these chemical surface terminations may be degraded by wear, and the degradation may be influenced by reactive species present in the gas phase. Polycrystalline silicon test structures have been used to determine the friction coefficient and durability of silane-based surface treatments in controlled environments. Water vapor present in the environment leads to changes in friction coefficient and device failure at fewer operating cycles than when water vapor is absent. Surface analysis and mechanistic aspects of interaction of the silane-treated surface with water vapor will be discussed. @FootnoteText@ @footnote 1@ 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.

4:20pm MM-WeA8 Selective Organophosphonate Chemical Sensors Using Self-Assembled Composite Monolayers and Adsorption-Induced Stresses in MEMS Devices, P.G. Datskos, H.M. Meyer, Oak Ridge National Laboratory; D. Karst, Virginia Tech; M.J. Sepaniak, University of Tennessee Recently there has been an increasing demand to perform real-time in-situ chemical detection of hazardous materials, contraband chemicals, and explosive chemicals. Currently, real-time chemical detection requires rather large analytical instrumentation that are expensive and complicated to use. The advent of inexpensive mass produced MEMS (micro-electromechanical systems) devices opens-up new possibilities for chemical detection. For example, microcantilevers were found to respond to chemical stimuli by undergoing changes in their bending and resonance frequency even when a small number of molecules adsorb on their surface. We describe a novel organophosphonate chemical sensor that is based on adsorption-induced stresses in MEMS (micro-electro-mechanical systems) and self-assembled monolayers. The MEMS microcantilever chemical sensor was found to exhibit high sensitivity, reversibility and chemical selectivity. Target molecules adsorbed on the surface of a microcantilever induce a differential surface stress causing changes in both the resonance frequency of the microcantilever and its bending. Measurable changes in the microcantilever bending always occur before any measurable resonance frequency shifts. Monitoring the bending of the microcantilever as molecules adsorb on its surface provides an extremely sensitive means of chemical sensing. In addition, monitoring of resonance frequency changes provides another sensing mechanism similar to the manner SAW and QCM devices operate. The chemical selectivity and reversibility of the present chemical sensor is based on the action of composite selfassembled monolayers. We will report on the response of microcantilevers with composite self-assembled monolayers to DIMP and DMMP. Our results show that such microcantilever chemical sensors exhibit rapid response times and high selectivity to organophosphonate compounds.

#### 4:40pm MM-WeA9 Nanofabrication and Electrostatic Operation of Singlecrystal Silicon Paddle Oscillators, D.W. Carr, S. Evoy, L. Sekaric, A. Olkhovets, J.M. Parpia, H.G. Craighead, Cornell University

Nanoelectromechanical systems (NEMS) are of interest from both scientific and technological standpoints. Such structures are being considered for use as sensors, force gauges and for various optomechanical and biomedical applications. Small resonant structures also open avenues for mesoscopic studies of the mechanical properties of materials. We have recently reported the fabrication and excitation of single wires with resonant frequencies as high as 380 MHz. Here we report the fabrication and characterization of paddle oscillators with nanometer-scale supporting rods. The devices are electrostatically driven and are detected at room temperature using an optical interferometric technique. The devices show two resonances in the f =1-10 MHz range. We have measured the frequency of both resonances for a series of devices of varying paddle length, d. A fit of data to a f = Kd^b power law reveals experimental power coefficients of b1=-0.5±0.1 and b2=-1.6±0.15 for the two resonances. These coefficients agree with the values expected for translational and torsional modes of motion, respectively. Our model of the torsional mode suggests that the external drive induces an angular dependent electrostatic torque, resulting in a modulation of the torsional constant. This results in a shift of the resonant frequency under the application of a DC bias. Dependence of this shift on the bias allows us to extract a mechanical

torsional constant of tau=  $4.21 \text{Å} \pm 0.04 \times 10$ -12 N.m. This modulation also results in parametric amplification effects that are under investigation. The translational motion shows non-linear behavior at low driving RF amplitudes. A model based on the mechanical stretching of the beams predicts the onset of non-linearity at such amplitudes. Finally, we will discuss the effects of material and surface properties on the dissipative processes in these structures. We are also looking at alternative geometries and potential chemical sensing applications.

5:00pm MM-WeA10 MEMS-Based Force Detected Nuclear Magnetic Resonance Spectrometer, *T. George*, *W. Tang*, *A. Chang-Chien*, *D.W. Elliott*, Jet Propulsion Laboratory; *L. Madsen*, *G. Leskowitz*, *D. Weitekamp*, California Institute of Technology

A novel nuclear magnetic resonance (NMR) spectrometer was recently demonstrated. In contrast to conventional NMR spectroscopy, which involves the detection of RF absorption, the force-detection technique works on the principle of using the RF to resonantly invert the magnetization of the sample of interest. The magnetization inversion is carried out at the mechanical resonance frequency of a microfabricated harmonic oscillator consisting of a silicon "diving board" on which a sensor magnet is mounted. The motion of the oscillator in response to the inversion of the sample magnetization is detected using fiber-optic interferometry. A two pronged approach was undertaken to develop the MEMS-based instrument. Microfabrication techniques including deep reactive etching of silicon and micro-electroplating of Fe-Ni alloys are being developed for the 2 mm diameter MEMS instrument. In parallel, a 25 mm diameter, conventionally machined magnet array mounted on a microfabricated silicon "diving board" has been used successfully to demonstrate the proof-of-concept. NMR spectroscopy has been conducted using this device, on millimeter-sized water droplets. Spin-echo experiments have also been undertaken to reduce the linewidth of the NMR peaks to below <1 Hz. The results of these experiments and the fabrication process will be described in detail. Applications of FDNMR spectroscopy in planetary exploration will also be discussed.

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