Thursday Afternoon, November 12, 2009

MEMS and NEMS
Room: B3 - Session MN+IJ+TR-ThA

Multi-scale Interactions of Materials and Fabrication at the Micro- and Nano-scale I

Moderator: A.V. Sumant, Argonne National Laboratory

2:00pm **MN+IJ+TR-ThA1 Silicon Carbide Thin Film Technology for Microsystems in Harsh Environments**, ***C. Carraro***, University of California, Berkeley **INVITED**

Whereas silicon has been the dominant semiconducting material for the fabrication of mechanical and electronic elements of micro-/nanosystems, its materials properties impose limitations on its use in harsh environment and demanding applications (e.g., repetitive contact, high temperature, high humidity). Silicon carbide thin film technology offers an alternative that enables such applications, thanks to its wider bandgap, higher melting/sublimation temperature, elastic modulus, fracture toughness, hardness, chemical inertness, and thermal conductivity. In this talk, I will review those SiC surface properties that are most different from silicon. I will then highlight recent materials, process, and characterization advances that are enabling SiC micro/nano systems for harsh environment and demanding applications.

2:40pm **MN+IJ+TR-ThA3 Sidewall Tribometer and Quartz Crystal Microbalance Study of a Self-Assembled Monolayer Lubricant Reservoir Effect**, ***D.A. Hook***, *B.P. Miller*, North Carolina State University, *M.T. Dugger*, Sandia National Laboratories, *J. Krim*, North Carolina State University

Long hydrocarbon and fluorocarbon based monolayers have been widely used in MEMS applications to prevent release related stiction and adhesion.[1] These and similar monolayers, however, have proven ineffective as MEMS lubricants alone. Indeed, even the most robust of SAM layers fails to protect devices from tribological failure for either normal or sliding cyclic contact [2]. Alternate schemes, such as vapor phase lubrication, must therefore be developed if progress is to occur. [3] The vapor phase of pentanol has recently been reported by Asay et al to extend the lifetime of a MEMS device in a mixture of dry nitrogen and various concentrations of pentanol. [4] This method of lubrication poses it's own set of issues in applications where devices need to be operated in native environments outside of lubricating vapors. Namely, does the vapor adsorb onto the surface in such a way that it will continue to lubricate in the native environment. In this study we have used a quartz crystal microbalance (QCM) to measure the adsorption and mobility of ethanol onto a surface coated with a perfluorodecyltrichlorosilane self assembled monolayer and a bare silicone surface. We have also used a MEMS sidewall tribometer to measure lifetimes of a SAM coated and uncoated device dosed with ethanol vapor. QCM measurements show that the self assembled monolayer retains ethanol on the surface once the vapor is removed and the tribometer lasts two orders of magnitude longer with the self assembled monolayer present once the ethanol vapor is removed. This data provides strong evidence that the self assembled monolayer acts as a lubricant reservoir and allows the residual ethanol to flow back into the contact area lubricating for extended periods of time.

Work funded by the AFOSR Extreme Friction MURI

1 Srinivasan, U., Houston, M.R., Howe, R.T., Maboudian, R., "Alkyltrichlorosilane-Based Self-assembled Monolayer Films for Stiction Reduction in Silicon Micromachines", Journal of Microelectromechanical Systems 1998, 7, 252-260

2 Hook, D.A., Timpe, S.J., Dugger, M.T., Krim, J., "Tribological Degradation of Fluorocarbon Coated Silicon Microdevice Surfaces in Normal and Sliding Contact" Journal of Applied Physics, 104, 034303, (2008)

3 Krim, J., Abdelmaksoud, M., "Nanotribology of Vapor-Phase Lubricants" Tribology Issues and Opportunities in MEMS, B. Bhushan, ed. (Kluwer Academic, Dordrecht, 1998), pp. 273-284, invited

3:00pm **MN+IJ+TR-ThA4 *In situ* Reliability Studies of Interfacial Contact via a 2-axis MEMS Deflecting Cantilever Microinstrument**, ***F. Liu***, *I. Laboriante, C. Carraro, R. Maboudian*, University of California, Berkeley

Recent developments in the MEMS field have created a growing interest in the reliability of these miniaturized devices. Along with the reliability issues such as stiction, corrosion and friction, wear is an important failure mechanism in these microsystems. Repetitive contact between microelectromechanical systems (MEMS) surfaces can lead to device failure, making it highly desirable to develop a microfabricated instrument to study the effects of impact and wear in MEMS for a wide range of structural layers, contact mechanics, coatings, and ambients.

This paper describes the design, and testing of a microinstrument that allows the surfaces of two microstructures to come into contact, after which the surfaces are separated sufficiently in the substrate plane to allow nondestructive surface analysis and then, for the first time, re-engagement of the contact. The device is designed to achieve large enough in-plane deflection for *in situ* analysis and controllable contact load. Using this microinstrument, the time-dependent assessment of the contacting surfaces is achieved by scanning probe microscopy, including atomic force microscopy (AFM) and Kelvin probe force microscopy (KPFM), as well as scanning Auger electron microscopy (SAEM) and electrical contact resistance measurements. The microinstrument design also allows for the study of a wide range of materials, coatings and environmental conditions under controlled loads. The contact resistance initially decreases during the first tens of millions of impacts and then increase gradually, a behavior attributed to the wear. The fracture of Si grains shows up at around 24 billion impacts and grows to 5-6 grains in diameter after about 100 billion impacts, associated with the interfacial oxidation. Based on these results, potential wear mechanisms at the microscale are proposed.

4:20pm **MN+IJ+TR-ThA8 Traceable Determination of Cantilever Spring Constants with a MEMS-based Sensor**, ***T. Dziomba***, *S. Gao, U. Brand, K. Herrmann, L. Koenders*, Physikalisch-Technische Bundesanstalt (PTB), Germany

Apart from accurate determination of dimensional, i. e. geometric, features of small objects such as nanostructures and semiconductor structures, the quantitative determination of small forces in the range from µN (10^{-6} Newton) down to several ten pN (10^{-12} N) is essential for many research tasks. Applications range from force spectroscopy in nanobiotechnology to the determination of the mechanical properties of nanomaterials, biological structures and organic molecules. Most of these measurements are performed with scanning force microscopes (SFM) and cantilevers with integrated nanometric tips as probing elements. However the comparability of experimental results lack under the knowledge of cantilever stiffness and traceability of small forces.

With the help of special Metrology-SFMs as reference instruments at National Metrology Institutes (NMIs), a large variety of transfer standards as well as guidelines for characterization & dimensional calibration of SFM, the length traceability to the SI-unit meter has been successfully established for SFM in the past few years.

However, a similar traceability chain for the measurement of small forces still needs to be realized. NMIs face the challenge to expand the traceability chain down to small forces by developing special nanoforce primary standards. A further challenge is the development of transfer standards and/or measurement procedures which allow the user to conveniently calibrate cantilevers used for SFM and scanning force spectroscopy. Besides the deflection-calibration a simultaneous force-calibration of the cantilever is necessary.

The contribution describes the properties of a MEMS (Micro-Electro-Mechanical-System) comb drive actor which can be used as a force sensor. Traceable calibration of its stiffness is done using a nanoforce calibration device based on a high resolution compensation balance. The sensor used has a force resolution in the nN-range, a measurement range of up to 1 mN, a translation range of 8 µm and was used to quantitatively determine the stiffness of SFM cantilevers. Preliminary experiments demonstrate that the long-term stability of the sensor is better than 3.7×10^{-3} N/m (1 sigma) over 1 hour. After careful traceable calibration of its stiffness, the MEMS sensor has the capability to determine the stiffness of a great variety of cantilever types (from 100 N/m down to 0.1 N/m) with high accuracy. Thus a new micro-force and stiffness transfer standard with nN force resolution is available for the traceable stiffness calibration of SFM cantilevers.

4:40pm **MN+IJ+TR-ThA9 Improvement in Mechanical Contact Reliability with ALD TiO_2 Coating**, ***V. Pott***, *H. Kam, J. Jeon, T.-J. King Liu*, UC Berkeley

Introduction: In order to overcome energy limits of CMOS, micro-electro-mechanical relays are now being investigated. High endurance is required for relay-based ICs to be viable, and has been a challenge due to stiction and wear. In this work, we demonstrate that a mechanical contact can be made to be very reliable if the surfaces of the conductive electrodes are coated with an ultra-thin layer of titanium dioxide (TiO_2) by atomic layer deposition (ALD).

Device structure: A 3-terminal (3-T) relay design was used: an electrically conductive mechanical beam (source) is actuated electrostatically by applying a voltage to an underlying electrode (gate) separated from the beam by an air gap. If the applied bias is above a threshold voltage (V_{TH}), the tip of the beam is deflected to bring it into contact with a fixed electrode (drain).

Device fabrication: First, tungsten gate and source electrodes were formed on top of a thermally oxidized Si wafer using sputter deposition. Then, a sacrificial low-temperature oxide (LTO) layer was deposited and patterned. The top W electrode was then sputter deposited and etched. A heavily doped polycrystalline silicon-germanium (poly-SiGe) structural layer was then deposited and patterned. The top W electrode is attached to the bottom of the poly-SiGe beam. The beam was then released in HF vapor. Immediately afterwards, the relay was coated with ALD TiO_2 at 275°C using titanium tetrachloride ($TiCl_4$) as the precursor material. One ALD cycle consists of one pulse of $TiCl_4$ followed by Ti oxidation, and deposits ~0.25A of TiO_2. TiO_2 – W is a moderate potential barrier for electron in the ON state.

Results: W contacts were coated with either 3, 6 or 12 cycles of ALD TiO_2. 3 cycles coated contacts have poor stability and degrade with time. This is attributed to tungsten native oxide growth. Devices were characterized by applying an actuation bias V_{GS}=12V and source-drain bias V_{DS}=50mV. The estimated force in the contact region is 9μN. Measurements are done after 100 ON/OFF switching cycles, to stabilize the contact resistance. Linear I_{DS}-V_{DS} characteristics have been measured for both 6 and 12 cycles of ALD TiO_2. Reported contact resistances are 85.2kΩ and 1.47MΩ, for a contact area of 15μm^2. No stiction or contact degradation is observed. If properly biased, 6 and 12 ALD TiO_2 cycles have an excellent yield and a good reliability (max. number of switching cycles tested thus far = 500).

Conclusion: We have found that coating of tungsten with ALD TiO_2 is an efficient way to reduce contacts ageing, stiction, and W oxidation. A contact resistance of 85.2kΩ has been measured and suggests the use of W-W contacts for relay-based ICs.

5:00pm **MN+IJ+TR-ThA10 Mass Producable, Multiple Stack, Integrated Micro Gas Chromatography System**, *K. Stacey, A. Knobloch, N. Chen,* GE Global Research, ***W.C. Tian***, National Taiwan University, *M. Shannon, R. Masel*, University of Illinois at Urbana-Champaign

This paper presents the novel wafer level processing, assembly, and characterizations of key components of a micro Gas Chromatography system (uGC). We demonstrated ways to perform wafer level low temperature bonding of silicon substrates and methods of patterning Metal Organic Frameworks (MOFs) within micropreconcentrators in a uGC. We also showed a polyimide membrane transfer process that is integral to the assembly of an electrostatic microvalve within our uGC. The overall device consists of multiple microvalves and a micropreconcentrator which are fabricated using a multiple wafer stack process and assembled using wafer level bonding. The entire process involves more than two hundred process steps including over tens of deep reactive ion silicon etching steps and multiple wafer bonds. We have succesfully fabricated functional preconcentrators and microvalves and have realized yields as high as 88%. In addition, full wafer microvalve assembly will improve device assembly time by 15-20x vs. die level assembly. The initial characterizations of micropreconcentrators and microvalves will be presented. In the future, the entire assembly of the system will be implemented.

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