## Friday Morning, November 13, 2009

### MEMS and NEMS Room: B3 - Session MN-FrM

### Multi-scale Interactions of Materials and Fabrication at the Micro- and Nano-scale Moderator: M. Metzler, Cornell University

#### 8:20am MN-FrM1 Using Nonlinearity to enhance Micro/NanoSensor Performance, K. Turner, University of California, Santa Barbara INVITED

Resonant microelectromechanical systems are key building blocks for many microsensor applications, including mass detection, inertial detection and RF filters and timing oscillators. Especially in high-Quality-factor systems, often amplitudes are such that nonlinearities are present. In many applications, these nonlinearities can be significant, and need to be accounted for. In this talk, I will give an overview of a few applications where understanding and cleverly utilizing nonlinearity actually results in improved sensor performance.

Examples including mass sensors and resonant angular rate sensors (Coriolis force sensors) will be used to explain these concepts.

## 9:00am MN-FrM3 High-Q, In-plane Modes of Nanomechanical Resonators Operated in Air, P. Waggoner, C. Tan, L. Bellan, H. Craighead, Cornell University

Viscous damping is perhaps the greatest limitation on the applicability of nanomechanical resonant sensors, typically reducing device quality factors by several orders of magnitude when operated in air or liquid as compared to vacuum. In addition to degraded sensitivity due to lower quality factors, the viscous media also effectively adds mass to the sensors, shifting the resonant frequency and further decreasing sensitivity to added mass. In order to achieve real-time, ambient sensing of biological and chemical analytes, a solution to these problems must be achieved. We have fabricated arrays of 90 nm thick mechanical resonators, studied their resonance spectrum as a function of pressure, and found that some higher order resonant modes feature quality factors on the order of 2000 at atmospheric pressure, namely two symmetric, in-plane resonant modes. The side-to-side resonance of these trampoline-shaped resonators was confirmed using finite element analysis and by experimentally exciting device resonance nonuniformly. Even after deposition of a relatively thick polymer layer, the quality factor of the in plane mode in air only decreased slightly, suggesting that functional sensing layers can be used with devices operated in air. These encouraging results open the door for resonant micro- and nanoelectromechanical systems (NEMS & MEMS) to biosensor and chemical sensor applications at atmospheric pressure.

### 9:20am MN-FrM4 An Overview of a Simple Fabrication Method for Effective Piezoresistive Transduction of MEMS Resonators, J. Cross, B.R. Ilic, Cornell University, M. Zalalutdinov, Global Strategies Group, J. Baldwin, B. Houston, Naval Research Laboratory, H. Craighead, J.M. Parpia, Cornell University

We present an overview of studies on a piezoresistive transduction mechanism for detecting MEMS resonator motion. The transduction mechanism is based upon flexure of two fabricated stacked layers of polysilicon, separated by a thin dielectric material. We have used thermal silicon dioxide and LPCVD silicon nitride for dielectric layers. The dielectric material's resistivity can be reproducibly electrically tuned via breakdown to tailor a vertically-oriented piezoresistive transducer between the polysilicon layers. The transduction mechanism is presented analytically, along with examples of non-linear data used to determine the displacement of the resonators. We obtain a gauge factor of approximately 5 with silicon dioxide as the dielectric, which is adequate for direct detection of the resonator motion without amplification or impedance matching. Integrated resonator-transducer devices in various geometries, such as double-clamped beams and cantilevers, have been fabricated using this method and we report on the effectiveness of various geometric parameters as well as various thicknesses and resistances of dielectric layers. As the film stack is composed entirely of CMOS compatible materials, we discuss a fabrication recipe for integrating this transduction mechanism with a conventional CMOS fabrication process. This work was partially supported by the Office of Naval Research, DARPA, and fabrication was performed at the Cornell NanoScale Science and Technology Facility.

9:40am MN-FrM5 Determination of Young Modulus and Density of Thin Films using Nanomechanics, *B.R. Ilic*, Cornell University, *S.L. Krylov*, Tel Aviv University, Israel, *H. Craighead*, Cornell University

Material properties of atomic layer deposited (ALD) thin films are of considerable interest to proposed applications ranging from wear resistance to high k-dielectrics in electronic circuits. We demonstrate the ability to simultaneously measure Young's modulus (E) and density (p) of 212-215Å ALD hafnia, alumina and aluminum nitride ultrathin films from vibrations ofnanomechanical cantilever beams. The nanomechanical structures were fabricated from a 250nm thick single crystal silicon layer with with varying length and width ranging from 6 to 10µm and 40nm to 1µm, respectively. Our approach is based on an optical excitation and interferrometric detection of in-plane and out-of plane vibrational spectra of single crystal silicon cantilevers before and after a conformal deposition of an ALD thin film. Due to the high degree of conformality, uniform thickness and composition of ALD films, fundamental mode eigenvalues depending on uncertainties in geometrical parameters and clamping compliances of the nanomechanical structures were filtered out. In conjunction with threedimensional numerical finite element analysis, baseline measurements carried out prior the deposition revealed that while the influence of clamping compliances arising due to the undercut of the sacrificial layer is significant for wider beams, the effect is less pronounced for both, narrower cantilevers and the in-plane vibrational responses. Following the deposition, higher stiffness alumina films (E>E\_{Si}) showed an increase in the resonant frequency whereas lower stiffness (E<Esi) hafnia and aluminum nitride films decreased the natural frequency. From the measured spectral response, material properties were extracted using simple expressions for E and  $\rho$  in terms of measured in-plane and out-of-plane frequencies shifts were derived from a model based on an ideally clamped Euler-Bernoulli beam with effective bending stiffness and effective mass per unit length. In-plane and out-of-plane frequency measurements provided two equations required for the extraction of E and p without the necessity of knowing material density prior to experiment. Our theoretical and experimental results are in good agreement with the data available in literature and indicate that the suggested approach can be efficiently used for the in-situ material parameters extraction of very thin films incorporated in nano-scale oscillators as well as for combined stiffness-density based material identification and comparative quantitative characterization of the film quality.

10:00am MN-FrM6 Parametric Excitation of Microstructures by Direct Mechanical Stiffness Modulation, S.L. Krylov, Y. Gerson, Tel Aviv University, Israel, T. Nachmias, U. Keren, Microsystems Design Center, RAFAEL LTD, Israel

In this work, we report on theoretical and experimental investigation of resonant behavior of a parametrically excited microstructure actuated by a time-varying electrostatic force. Parametric devices typically described by

Mathieu-type differential equations with time-dependent coefficients are attractive for a broad variety of applications such as mass sensors, dynamic electromechanical amplifiers or inertial sensors due to the ability to generate resonant responses in relatively wide band of excitation frequencies as well as sharp transition between low-amplitude to large-amplitudes responses. In electrostatically actuated MEMS devices, parametric

excitation arises mainly as a result of nonlinearity of electrostatic forces combined with periodic time dependencies of the actuation voltage as well as for geometrical reasons or due to kinematic excitation.

In this work we implement direct mechanical stiffness modulation by means of a periodic tensile force applied along suspension flexures. The frametype structure is realized as a pair of cantilever (clamped-guided) beams connected at their ends by a rigid link. The electrostatic actuation force applied to the rigid link by a parallel-plate electrode connected to an AC voltage source results in a periodic tensile force within the beams and consequently in periodic structural stiffness modulation and mechanical parametric excitation of the structure. Combination of compliant cantilevertype suspension with lateral motion in the direction parallel to the electrode results in large resonant amplitudes and higher quality factors while high axial stiffness in the direction of the force application prevents undesirable pull-in instabilities. The devices were fabricated from single crystal silicon using silicon on insulator (SOI) substrates and deep reactive ion etching (DRIE) process. The devices were excited electrostatically in the vicinity of the 2:1 subharmonic (parametric) as well as primary resonances and large resonant responses were registered. The lumped model of the device consists of a rigid link undergoing electrostatic loading as well suspension flexures modeled as geometrically nonlinear massless beams. Experimental resonant curves as well as stability regions boundaries built by means of video acquisition and image processing are in good agreement with the results provided by the model. Theoretical and experimental results indicate that the suggested actuation approach have clear functional advantages and could be efficiently used for excitation of various types of microdevices where resonant operation combined with robustness and large vibrational amplitudes are desirable.

# 10:20am MN-FrM7 Fabrication of Overhanging Magnet-Tipped Cantilevers for Nanoscale Scanned-Probe Magnetic Resonance, J.G. Longenecker, S.A. Hickman, Cornell University, L.E. Harrell, United States Military Academy, J.A. Marohn, Cornell University

Mechanical detection of magnetic resonance opens up exciting possibilities for characterizing soft materials at nanometer-scale, and potentially atomicscale, resolution. Scanned-probe detection of single-spin electron paramagnetic resonance has been demonstrated. Proton images exhibiting 4 nm resolution have recently been acquired via magnetic resonance force microscopy (MRFM), albeit in an experiment with the sample glued to the cantilever. With the goal of pushing proton imaging resolution beyond 4 nm in a true scanned-probe experiment capable of imaging potentially any thinfilm sample, we have taken up the challenge of fabricating attonewtonsensitivity cantilevers with integrated nanorod magnetic tips.

Since the force exerted on the cantilever, per spin, is proportional to the field gradient from the magnetic tip, achieving single proton sensitivity requires reducing the magnetic nanorod diameter to below 50 nm. In the most sensitive scanned-probe magnetic resonance measurements to date, a magnetic particle was manually affixed to the cantilever and the particle diameter reduced to  $\sim 150$  nm by focused-ion-beam (FIB) milling. Unfortunately, FIB is a serial process and it is difficult to see how FIB milling can be used to make MRFM tips smaller than  $\sim 150$  nm due to ion-beam damage limitations.

We demonstrate a method for batch-fabricating attonewton-sensitivity silicon cantilevers with integrated nickel tips having critical dimensions of 70 nm. The magnets are patterned by electron-beam lithography and can therefore potentially be made even smaller. The overall fabrication protocol involves thirty-eight carefully-integrated processing steps, including three electron beam lithography steps and two isotropic etching steps. A crucial feature of our cantilever design is that their narrow magnetic tip overhangs the leading edge of the cantilever by up to 400 nm, which minimizes extraneous force and frequency noise in the MRFM experiment known to arise from interactions of the cantilever charge with fluctuating electric fields and field gradients in the sample. Cantilever magnetometry indicates that the tips are nearly fully magnetized. We will detail ongoing work to develop cobalt tips, to push magnet critical dimensions to less than 50 nm, and to study the chemical structure of the tips using high-resolution transmission electron microscopy.

# 10:40am MN-FrM8 Nanoscale Resonant Mass Sensors Containing Nanofluidic Channels, R.A. Barton, B.R. Ilic, S.S. Verbridge, H. Craighead, J.M. Parpia, Cornell University

The ability of nanomechanical resonators to sense mass in a liquid environment is compromised by a large dissipation of energy to the surrounding liquid. One way to overcome this problem is to deliver analytes in solution to the resonator via an embedded fluidic channel, while surrounding the resonator itself with vacuum. Previously, this technique has been applied to detect mass from solution with roughly femtogram precision, but it has never been applied with the sensitivity required to detect single biomolecules. In order to enable studies in the regime of attogram analyte mass, we have designed and fabricated resonators that contain nanofluidic channels. We optically actuate and detect mechanical resonance of the channels and estimate from their frequency and quality factor that they will be able to detect mass with sub-attogram precision. We anticipate that these devices will be useful for sensing and for studies of single large biomolecules.

11:00am MN-FrM9 Spectroscopic Investigations of XeF<sub>2</sub> Chemistry with Si and Mo Layers on Al Substrate, J.-F. Veyan, K. Roodenko, Y. Gogte, University of Texas at Dallas, X.-M. Yan, Qualcomm MEMS Technologies, Inc., Y. Chabal, University of Texas at Dallas

Etching sacrificial material during MENS and NEMS manufacturing constitute an important processing step.  $XeF_2$  is a commonly used chemical etchant because of its selective interaction with pure compounds and their oxides. Practically, realistic systems are characterized by multicomponent films with interfaces, and side reactions have to be taken into account in the study of the complex chemistry taking place during the etching process. We have studied XeF<sub>2</sub> etching of pure Silicon, pure Molybdenum, and Molybdenum deposited on Silicon oxide and Al substrates, in pressures consistent with industrial conditions ( $\sim$  Torr range) using in-situ time-resolved IR reflection spectroscopy and ex-situ XPS.

We find that Si and Mo react with XeF2 in very different ways. For Si, F atoms penetrate deep inside the Si lattice, for Mo they stay at the surface.

These differences greatly affect the etching kinetics, involving bulkcontrolled etching process for Si, and a surface controlled etching for Mo.

After  $XeF_2$  etching and removal of Mo and Si films deposited on Al substrates, the surfaces exhibit residual molybdenum (oxy) fluoride and silicon oxide layers. F1s core level spectra indicated excess fluorine atoms on all surfaces.  $CF_x$  contaminations have been found on Si/Al and on Mo/Al surfaces after etching, but not on bare Al substrates, even after  $XeF_2$  exposures. The stability of etched surfaces is also studied.

### 11:20am MN-FrM10 Cryogenic Inductively Coupled Plasma Etching for Fabrication of Tapered Through-Silicon Vias, A. Kamto, The University of Alabama, R. Divan, A.V. Sumant, Argonne National Laboratory, S.L. Burkett, The University of Alabama

Vertical interconnects pose an interesting method for heterogeneous integration of electronic technologies allowing three-dimensional (3D) stacking of Microelectromechanical systems and integrated circuit device components [1, 2]. The vertical interconnects, referred to as through-silicon vias (TSVs), begin with formation of blind vias in silicon that are eventually exposed by mechanically lapping and polishing the wafer backside. Inductively coupled plasma (ICP) etching using SF<sub>6</sub>/O<sub>2</sub> gas chemistry at cryogenic temperatures has been investigated as a way to form vias with a tapered sidewall. The point in creating a controlled taper is so that subsequent thin films can be deposited along the sloped sidewall lining the via with insulation, barrier, and seed films. This tapering is necessary if the via lining processes do not provide adequate conformal coverage, a common problem for conventional low temperature deposition processes. In our process for lining vias, plasma enhanced chemical vapor deposited (PECVD) silicon dioxide is used to insulate vias from the surrounding silicon. After insulation, thin films of Ti and Cu are sputter deposited. Ti provides protection from copper migration while the Cu acts as a seed layer for the electrodeposition step. After etching and lining, the vias are filled by reverse pulse plating of Cu. Vias are 20 - 25 µm in diameter and etched using a photoresist mask. The effect of changing gas flow rates, chamber pressure, RF forward power, ICP power, and substrate temperature on etch rate, via profile, and sidewall morphology will be presented. These parameters are critical in optimization of an etch process for vias of specific dimensions to be used in 3D integration.

This work is supported by the College of Engineering at the University of Alabama. Use of the Center for Nanoscale Materials was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

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