

# Monday Morning, October 29, 2012

## MEMS and NEMS

Room: 10 - Session MN+AS-MoM

### Characterization of Surfaces and Interfaces in MEMS and NEMS

Moderator: A.V. Sumant, Argonne National Laboratory

8:20am MN+AS-MoM1 Probing Dynamical Surface and Interfacial Effects in High-Speed Nanoelectromechanical Systems (NEMS), X.-L. Feng, Case Western Reserve University **INVITED**

Nanoelectromechanical systems (NEMS), especially vibrating or resonant-mode NEMS based upon advanced materials and new nanostructures, are emerging as attractive candidates for many nanoscale sensing and signal transduction technologies. Understanding and controlling various surface and interfacial effects in NEMS are important for engineering NEMS toward such goals. In this talk, we focus on using high-speed NEMS themselves as highly sensitive transducers for probing dynamical surface effects and interfacial behavior in these devices.

First, the behavior of physisorbed thin layers on solid surfaces is both interesting for fundamental studies and important for technological applications. For many solid-state devices, ranging from conventional commodity transducers to emerging miniaturized sensors, surface contaminants and adsorbates can be critical for the device performance. Recent advances in NEMS, particularly their excellent sensitivities, make it possible and to probe surface adsorbates and their behavior in the new regime – where a small number of adatoms can cause a detectable frequency shift for a NEMS resonator with a high quality factor ( $Q$ ); and random fluctuations in the sub-monolayer adsorbates may result in variations of the NEMS resonance. We experimentally measure the frequency noise induced by fluctuations of adsorbed xenon (Xe) atoms on the surface of a very high frequency (VHF, ~200MHz), high- $Q$ , SiC NEMS resonator. The measured adsorption spectrum and phase noise suggest interesting kinetics of Xe atoms on the surface. We further examine contributions from both surface diffusion and adsorption-desorption. The combined measurements and analyses not only demonstrate that surface diffusion dominates the measured noise in the experimental regime, but also reveal new power laws of noise processes that may be important in various low-dimensional nanosystems.

Second, in NEMS devices with contacts and contact-mode operations, a lot of studies have to date yielded good intuitive understanding and empirical laws. For many new devices with genuinely nanoscale contacts, it has been highly desired but very challenging to understand these nanocontacts with greater details and with quantitative information. By combining experimental measurements and modeling, we explore the detailed electronic and nanomechanical characteristics in contact-mode NEMS with high-speed operations, with a focus on NEMS based on SiC nanowires and nanocantilevers.

9:00am MN+AS-MoM3 Fabrication of Nanomechanical Switch Based on Ultrananocrystalline Diamond Nanowire, A.V. Sumant, Argonne National Laboratory, K.J. Pérez Quintero, University of Puerto Rico, D.A. Czaplewski, Argonne National Laboratory

Fabrication of nanomechanical switches using various materials is being actively pursued over conventional solid state switch technology because of advantages of zero leakage current, ultra low power consumption and reasonable switching speeds reaching to 100 ns. Diamond is an ideal candidate material for nanomechanical switches due to high Young's modulus, moderate electrical conductivity when doped with boron or incorporated with nitrogen, high thermal conductivity and chemically inert nature. Recently, fabrication of nanomechanical switches in single crystal diamond has been demonstrated. However, batch fabrication of nanomechanical switches and their integration with complementary metal oxide semiconductor (CMOS) technology in bulk diamond is not feasible.

Ultrananocrystalline diamond (UNCD), originally developed at Argonne National Laboratory is an excellent candidate material for nanomechanical switches due to its high Young's modulus (comparable to single crystal diamond), semi-metallic conductivity when doped with boron or incorporated with nitrogen and because it is the only diamond film that can be deposited at temperatures as low as 400 °C, at wafer scale, with demonstrated integration with CMOS electronics [1]. We have previously fabricated horizontally aligned N-incorporated UNCD nanowires by a top down approach using Electron Beam Lithography (EBL) patterning and Reactive Ion Etching (RIE) processes [2] with nanowire lengths of 50-100 um and widths as small as 30 nm.

We demonstrate a fabrication of UNCD nanowire based switch with a movable source anchored at both ends. An immobile drain electrode is separated from the center of the source beam by a narrow gap. Two electrically connected gate electrodes are separated from the source by the gate gap, which is larger than the drain gap [3]. A UNCD layer was deposited on top of a sacrificial SiO<sub>2</sub> layer and covered with a SiO<sub>2</sub> layer that served as a hard mask for the RIE process. The UNCD layer represents the mechanical layer of the switch, the switch contacts and the gate electrodes. We aim to fabricate a reliable switch with fast switching times and low actuation voltages.

References:

- [1] Sumant *et al.* MRS Bulletin, 35, 281 (2010)
- [2] Wang *et al.* Nanotechnology, 23, 075301 (2012)
- [3] Czaplewski *et al.* Electronics Letters 45(11): 550 (2009)

9:20am MN+AS-MoM4 Carbon Nanotube Templated MEMS: Three Dimensional Microstructures in Semiconductors, Ceramics, and Metals, R.C. Davis, L. Barrett, R. Hansen, A. Konneker, D.D. Allred, B.D. Jensen, R. VanFleet, Brigham Young University

We discuss a materials breakthrough for MEMS. In contrast with conventional electromechanical devices, whose constituents are chosen from a vast range materials and alloys to optimize fabrication, performance and cost, MEMS have largely been made using the same materials and methods as those used in the silicon-based microelectronics industry. In order to make MEMS out of a much richer suite of materials, including metals, semiconductors, and ceramics, we have developed a process termed carbon nanotube templated microfabrication (CNT-M). In CNT-M we employ patterned, vertically aligned carbon nanotube forests as a three-dimensional microfabrication scaffold to create precise high-aspect-ratio (up to 200:1) microstructures. The “as grown” CNT forests are very low density (at 0.009 g/cc the forest is ~1% carbon and 99% air) and not useful as mechanical materials because they are extremely fragile, due to their low density and weak intratube bonding. However, when we replace the air spaces between tubes in the forest with a filler material by atomistic deposition, the infiltrated CNT framework becomes a robust microstructure consisting mostly of the filler material. Thus, by patterning the CNT microstructure and limiting the deposition of the filler material, CNT-M gives us control over structural features on both the nano and microscale (nanoscale porosity and microscale structure). We have used chemical vapor deposition to infiltrate the CNT framework with semiconductors (Si) and ceramics (SiO<sub>2</sub>, SiN<sub>x</sub>, and nanocrystalline carbon) for applications in microactuation, sensing, and chemical separations. But many potential MEMS applications would benefit from structures fabricated from functional metals. We now report on the fabrication of metal microstructures using the CNT-M process. We demonstrate the versatility of this fabrication approach by demonstrating both chemical vapor infiltration (making tungsten and molybdenum structures) and electrodeposition (making nickel structures) based metal CNT-M processes. These metals provide several desirable materials properties to high aspect ratio MEMS applications including high electrical and thermal conductivity, high melting temperatures, resistance to corrosion, low thermal expansion, high Young's modulus, hardness and yield strength. Electrical, mechanical, and structural characterization of the microfabricated metal structures will also be presented.

9:40am MN+AS-MoM5 Filling through Silicon vias with a Carbon Nanotube/Copper Matrix, M.B. Jordan, M. Rao, The University of Alabama, A.V. Sumant, R.S. Divan, Argonne National Laboratory, S.L. Burkett, The University of Alabama

The performance of through silicon vias (TSVs) depends on the material used to fill them. Copper and tungsten are two conventional metals used to fill TSVs. Recently carbon nanotubes (CNTs) have been considered as a filling material due to their superior material properties. CNT bundles can allow ballistic transport of electrons resulting in low resistivity and enabling them to carry a larger current density. CNT bundles also have a high Young's modulus, low coefficient of thermal expansion, and a high thermal conductivity. These properties make CNTs appealing for use as power delivery systems and as heat sinks. Protecting the CNTs after growth and making electrical contact to them remains a challenge. We have investigated a hybrid CNT/Cu TSV structure as a possible solution to these problems. Blind vias were formed using a cryogenic inductively coupled plasma (ICP) etch process. A copper seed layer was sputtered on the via base and along the sidewalls. The vias were filled using a periodic reverse pulse electroplating technique to reduce voids in the high-aspect ratio structures. The center region of the copper filled vias were then etched by ion milling. The growth of CNT bundles in the center of the copper filled

vias was done by thermal chemical vapor deposition (CVD). Electron-beam evaporated Fe serves as a catalyst for CNT growth.

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10:00am **MN+AS-MoM6 Optimization of STiGer Process used to Etch High Aspect Ratio Silicon Microstructures**, *T. Tillocher, P. Lefaucheur*, GREMI CNRS/Université d'Orléans, France, *J. Ladroue, M. Boufnichel*, ST Microelectronics, Tours, France, *P. Ranson, R. Dussart*, GREMI CNRS/Université d'Orléans, France

The STiGer process, which can be used in MEMS fabrication, is a time-multiplexed cryogenic process designed to etch deep anisotropic features in silicon: passivation and etching plasmas are cycled to get vertical structures. The passivation layer is a  $\text{SiO}_x\text{F}_y$  film which requires cryogenic substrate temperature conditions to grow. It desorbs and disappears when the substrate is heated back to room temperature. This is an advantage since no extra cleaning steps are required. Additionally, with the benefit of the periodic passivation cycles, this process is less sensitive to temperature or flow rate variations than standard cryoetching. This enhanced passivation helps to reduce undercut as well. Nevertheless, like in Bosch etching, the alternations induce a scalloping on the sidewalls.

We have already shown that trenches having critical aperture of about 0.8  $\mu\text{m}$  can be etched with high aspect ratios ( $> 40$ ). We have highlighted a defect called "extended scalloping", which is composed of anisotropic cavities developed on the feature sidewalls, just below the mask. It originates from ions scattered at the feature entrance that hit the top profile and remove locally the passivation layer. This defect is observed for aspect ratios higher than 10. Consequently, large structures, with openings larger than 100  $\mu\text{m}$ , etched to a few hundred of  $\mu\text{m}$  show no extended scalloping.

We have proposed two methods that can help to reduce this defect. The first consists in adding a low oxygen flow in the etch cycle, favouring a low additional passivation. The second technique consists in gradually increasing the  $\text{SF}_6$  flow, in the etching steps, during the first minutes of the recipe. Consequently, the process starts with a low etch rate and a more efficient passivation, which helps to limit the extended scalloping. These two techniques efficiently reduce the defects but the profiles tend to be always positive. It seems impossible to get at the same time vertical sidewalls and low defects.

We will present other ways to fix this problem. For example, we are currently investigating processes running at  $-50^\circ\text{C}$  instead of usual cryogenic temperatures ( $-100^\circ\text{C}$ ). This aims to have a more conformal passivation layer, which may prevent the initiation of the extended scalloping. Additionally, this range of substrate temperatures is of interest since it can be reached with chillers and thus, liquid nitrogen is no longer required.

Finally, we will present our results on downscaled structures. We have designed a mask with e-beam lithography comprising 200 nm to 800 nm wide trenches. It is used to evaluate the performances of the STiGer process on submicron structures.

11:00am **MN+AS-MoM9 The Effect of Back-action Force for the Electron Tunneling Transduction in MEMS Measurement**, *M.R. Kan*, University of Alberta, Canada, *Z. Diao*, National Institute for Nanotechnology, NRC Canada, *V.T.K. Sauer, M.R. Freeman*, University of Alberta, Canada, *W.K. Hiebert*, National Institute for Nanotechnology, NRC Canada

Nano-electromechanical systems (NEMS) have exciting potential for fields ranging from quantum measurement science to ultrasensitive mass detection. For many of these applications, a key challenge is implementing a fast, reliable, low-noise technique for translating small mechanical motion to electronic signals. Electron tunneling transduction based on quantum tunneling is a promising technique to measure small displacements, because the tunneling current is so sensitive to the change in distance between the probing tip and the sample surface (one angstrom distance change causes 7 times tunneling current change). With frequency downmixing, the bandwidth limitation associated with the large RC time constant in the circuits can be overcome; very high frequencies may become accessible, fundamentally limited only by the tunneling rate  $I_T/e$  in the GHz range.

Using electron tunneling to sense nanomechanical motion comes with an inherent risk of back-action of the sensing probe (STM tip) on the mechanical device. The local tip-sample energy gradients introduce spring forces that can produce sizable shifts in resonance frequencies and may also affect sample quality factors. Understanding these effects is important for reliable use of downmixed tunneling transduction. Controlling them will allow for novel methods of MEMS and NEMS tuning of both frequency and quality factor.

In this presentation, we will report our observation of back-action forces on MEMS devices during downmixed electron tunneling transduction. We explore differences in the magnitude of the back-action force for different flexural and torsional vibrational modes (with varying degrees of inherent stiffness). We also discuss the perturbation to device quality factors. Finally, the vibration of the back-action force as a function of tip-sample distance is investigated.

11:20am **MN+AS-MoM10 Electric-Stimulus-Responsive Pluronic Hydrogels as Actuators**, *L. Engel, I. Sokolov, O. Berkh*, Tel Aviv University, Israel, *K. Adesanya, E. Vanderleyden, P. Dubruel*, Ghent University, Belgium, *J. Shklovsky, I. Harari, Y. Shacham-Diamand, S. Krylov*, Tel Aviv University, Israel

Due to their unique mechanical and chemical characteristics, stimuli responsive hydrogels have garnered much interest in the field of biomedics. They perform dramatic volume transitions in response to external environmental stimuli such as pH and ionic strength of the solvent, temperature, and electrical field. Their soft elastomeric nature, serves to minimize mechanical and frictional irritation to the tissue bed, suggesting applications in artificial muscles and biomimetics, and their swelling capacity results in high permeabilities for certain drug molecules and metabolites making them ideal materials for drug delivery. Because the swelling rate of a hydrogel is inversely related to its size, MEMS offers a unique opportunity to exploit the capabilities of responsive hydrogels by minimizing actuator response time. While it is known that hydrogels with fixed charge groups deform when subjected to an externally applied electric field inside an electrolyte bath, the exact mechanism responsible for the deformation continues to be debated.

In this work, we have investigated the volume transformation of Pluronic based electroactive hydrogels immersed in a Krebs bathing solution under an applied electric field. The swelling characteristics of the crosslinked hydrogels were investigated and a model based on finite element analysis is proposed. Bias was applied via parallel Pt electrodes and the distance between the electrodes was varied as was the ionic concentration and pH of the solution inside the testing tank. The feasibility of using an array of interdigitated electrodes fabricated on a printed circuit board as a means of actuation hydrogel was demonstrated with the goal of downsizing the hydrogel electrical-stimulation system for the creation of MEMS electro-responsive hydrogel actuators.

11:40am **MN+AS-MoM11 CMOS MEMS Metal-based Tactile Sensors Development**, *Y.C. Lin, C.J. Hsieh, L.B. Wang, J.C. Liou, W.-C. Tian*, National Taiwan University, Taiwan, Republic of China

A CMOS MEMS tactile sensor using a pure metal-based structure by a special etchant (Silox Vapox III) to remove oxide sacrificial layers was developed. The tactile sensor was fabricated through a commercial 0.35mm 2 polysilicon and 4 metal CMOS technology followed by the self-developed post processes. In order to increase the effective gap between two electrodes, the tactile sensor used oxide as the sacrificial layer to replace the conventional metal sacrificial layer. Moreover, the CMOS MEMS-based tactile sensors provides the advantages such as lower cost, small size, compatible with the integrated circuits, and mass-production compared to other types of tactile sensors.

Two different capacitive-based tactile sensor designs, parallel-plate type and vertical-comb-drive type, were proposed in this work. A boss-structure was implemented to provide the uniformity of the membrane displacement during the device operation. The dynamic range of the sensor detection was targeted from 0 to 200 mmHg according to the human vessel pressure. The capacitance variation was measured and analyzed via an integrated circuit board, the arduino board, and an A/D IC, AD7746. The readout circuit module reduced the noise and improved the sensor accuracy to 4fF and the resolution down to 4 aF. The sensitivity of the parallel-plate type is measured to be 1.39 fF/mmHg which is suitable for the blood flow monitoring. More characterizations on the vertical-comb-drive type sensors will be presented.

# Authors Index

**Bold page numbers indicate the presenter**

## — A —

Adesanya, K.: MN+AS-MoM10, 2  
Allred, D.D.: MN+AS-MoM4, 1

## — B —

Barrett, L.: MN+AS-MoM4, 1  
Berkh, O.: MN+AS-MoM10, 2  
Boufnichel, M.: MN+AS-MoM6, 2  
Burkett, S.L.: MN+AS-MoM5, 1

## — C —

Czaplewski, D.A.: MN+AS-MoM3, 1

## — D —

Davis, R.C.: MN+AS-MoM4, **1**  
Diao, Z.: MN+AS-MoM9, 2  
Divan, R.S.: MN+AS-MoM5, 1  
Dubruel, P.: MN+AS-MoM10, 2  
Dussart, R.: MN+AS-MoM6, 2

## — E —

Engel, L.: MN+AS-MoM10, 2

## — F —

Feng, X.-L.: MN+AS-MoM1, **1**

Freeman, M.R.: MN+AS-MoM9, 2

## — H —

Hansen, R.: MN+AS-MoM4, 1  
Harari, I.: MN+AS-MoM10, 2  
Hiebert, W.K.: MN+AS-MoM9, 2  
Hsieh, C.J.: MN+AS-MoM11, 2

## — J —

Jensen, B.D.: MN+AS-MoM4, 1  
Jordan, M.B.: MN+AS-MoM5, **1**

## — K —

Kan, M.R.: MN+AS-MoM9, 2  
Konneker, A.: MN+AS-MoM4, 1  
Krylov, S.: MN+AS-MoM10, 2

## — L —

Ladroue, J.: MN+AS-MoM6, 2  
Lefaucheux, P.: MN+AS-MoM6, 2  
Lin, Y.C.: MN+AS-MoM11, 2  
Liou, J.C.: MN+AS-MoM11, 2

## — P —

Pérez Quintero, K.J.: MN+AS-MoM3, 1

## — R —

Ranson, P.: MN+AS-MoM6, 2  
Rao, M.: MN+AS-MoM5, 1

## — S —

Sauer, V.T.K.: MN+AS-MoM9, 2  
Shacham-Diamand, Y.: MN+AS-MoM10, 2  
Shklovsky, J.: MN+AS-MoM10, 2  
Sokolov, I.: MN+AS-MoM10, 2  
Sumant, A.V.: MN+AS-MoM3, **1**; MN+AS-MoM5, 1

## — T —

Tian, W.-C.: MN+AS-MoM11, 2  
Tillocher, T.: MN+AS-MoM6, 2

## — V —

Vanderleyden, E.: MN+AS-MoM10, 2  
VanFleet, R.: MN+AS-MoM4, 1

## — W —

Wang, L.B.: MN+AS-MoM11, 2