

# Monday Afternoon, October 20, 2008

## MEMS and NEMS

Room: 206 - Session MN+NC-MoA

### Fabrication at the Micro- and Nano- Scales for MEMS/NEMS

Moderator: A.V. Sumant, Argonne National Laboratory

#### 2:00pm MN+NC-MoA1 Large Area Nanofabrication for MEMS Applications, L.E. Ocola, A. Imre, Argonne National Laboratory INVITED

As microelectromechanical systems (MEMS) shrink towards nano scale dimensions (or NEMS) the need for novel fabrication techniques increases. In this paper we revisit the use of two well-known techniques and propose new ways they can contribute toward the fabrication of next generation NEMS devices. The two techniques are super-high aspect ratio patterning using high-voltage electron beam lithography, and large area nanofabrication using a focused ion beam tool (FIB). In this paper we report on hydrogen silsequioxane HSQ nanopatterned into super-high aspect ratio structures (aspect ratio > 10) using 100 KV e-beam lithography, along with development in aqueous TMAH solution and DI water rinse, both performed at elevated temperatures (60 °C). Hot development allows for the rapid removal of low molar mass uncrosslinked molecules from the exposed regions while preserving the mechanical integrity of the nanopatterned structures. Raising the water rinse temperature to 60 °C also has the benefit of reducing the water surface tension by about 10%. Preliminary results of 90 nm structures using 1.2 micron thick HSQ, i.e. aspect ratios of 12, have been obtained. Such structures have immediate application in MEMS, Fresnel zone plate fabrication, and nanophotonics among others. We also have explored the use of focused ion beam (FIB) lithography in similar fashion to that of electron beam lithography. Although FIB has been employed extensively as a single write-field exposure tool for small device tailoring, mask repair, and sample characterization, it is rarely used for nanoscale patterning on a large area. This requires a high precision stage, and additional lithography software to handle complex and large pattern designs. In general, neither the hardware nor the software of FIB instruments is prepared for this task. We configured a FEI Nova 600 Nanolab Dual Beam FIB system with a 100 nm resolution X-Y stage, a Raith Elphy Lithography software interface and a Raith 16-bit DAC pattern generator for the X and Y deflectors. This provides us with the capability to expose more than 65,000 pixels/axis in one write-field, and allows us reading layout designs from files in GDSII format. Both capabilities are typical and standard in any medium-level e-beam lithography tool, but not found until recently in FIB systems. We present various large-area patterns milled in silicon and diamond thin films, and evaluated for stitching accuracy.

#### 2:40pm MN+NC-MoA3 Large-scale Fabrication of Silicon Nanowire NEMS Devices Using a Top-down Approach, C.S. Roper, University of California, Berkeley, R.T. Howe, Stanford University, R. Maboudian, University of California, Berkeley

While bottom-up nanofabrication techniques, including vapor-liquid-solid (VLS) growth, can create single crystalline nanostructures,<sup>1</sup> the integration of a single nanowire into a functioning, addressable device is an extremely difficult task, and problematic on a large scale, owing to the stochastic nature of the growth process. The primary barriers to bottom-up integration are manifold. First, nanowire placement and alignment are difficult to control. VLS nanowire growth is catalyzed by molten eutectic alloy nanoclusters that wander erratically on the substrate upon heating, and often coalesce with one another, rendering even size control unacceptably loose. Furthermore, transmission of signals to and from a nanostructure is problematic due to difficulty forming either directly contacted or capacitively coupled electrodes. Electron-beam lithography can be used to pattern electrodes on a bottom-up grown nanostructure to create a device,<sup>2</sup> but its high cost and serial nature make it ineffectual for the realization of large arrays of interconnected devices. Unlike bottom-up techniques, top-down microfabrication techniques, including projection lithography, oxidation of silicon, chemical vapor deposition (CVD) of thin films, and plasma etching, readily lend themselves to precise placement, alignment, and ultra large scale integration. However, the minimum feature size and alignment error limitations of optical lithography preclude the direct patterning of nanoscale devices. We present a manufacturable fabrication process to realize large arrays of individually addressable silicon nanowire resonators using an entirely top-down approach that relies on optical projection lithography and multiple steps of controlled oxidation. Our fabrication process uses novel and elegant mask and process design to overcome the limitations of traditional top-down processes, yielding arrays

of precisely positioned, vertically aligned, and electrically connected silicon nanowires with diameters as small as 30 nm. With the aim of creating ultra-sensitive mass sensors, devices with a single vertically aligned silicon nanowire as the resonant mass and multiple electrodes spaced hundreds of nanometers from the nanowire are also fabricated with the top-down process.

<sup>1</sup>J. Westwater et al., J. Vac. Sci. Technol. B., 15 (3) 554-557, 1997.

<sup>2</sup>H. T. Soh, et al. Appl. Phys. Lett. 75, 627-629, 1999.

#### 3:00pm MN+NC-MoA4 Fabrication of Nanopore and Nanochannel Structures through E-Beam Lithography and Atomic Layer Deposition Processes, S.W. Nam, IBM T.J. Watson Research Center & Seoul National University, Korea, M.J. Rooks, R. Sirdeshmukh, IBM T.J. Watson Research Center, K.B. Kim, IBM T.J. Watson Research Center & Seoul National University, Korea, S.M. Rossnagel, IBM T.J. Watson Research Center

The reliable and reproducible fabrication of nanopore and nanochannel structures is essential for building nano-fluidic systems, such as a device to control bio-molecules or DNA. Here, we report on CMOS-compatible fabrication methods to generate electrode-embedded nanopore and nanochannel structures as small as 10 nm feature size. To fabricate a small size fluidic channel, we adopt a combined process of e-beam lithography and atomic layer deposition (ALD). E-beam lithography is used for generating few tens of nanometer sized hole and line structures and ALD is employed for further shrinking down the features less than 10 nm. In the case of a pore fabrication, we used a membrane of SiO<sub>2</sub> (100 nm)/Si<sub>3</sub>N<sub>4</sub> (20 nm)/TiN (30 nm)/Si<sub>3</sub>N<sub>4</sub> (20 nm) on Si wafer. PMMA (300 nm thickness) hole structures on top of the membrane were patterned by e-beam lithography, which were then transferred to the membrane layers through two-step RIE process, consisting of sequential CHF<sub>3</sub>+O<sub>2</sub> and CH<sub>4</sub> steps to remove SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub>/TiN/Si<sub>3</sub>N<sub>4</sub>, respectively. This drilling process formed 50-80 nm diameter pore structures on membrane. In a parallel way, we fabricated trench structures by using a stack of Si<sub>3</sub>N<sub>4</sub> (5 nm)/a-Si (20 nm)/SiO<sub>2</sub> (100 nm) films on a bulk Si substrate. PMMA (200 nm thickness) line patterns fabricated by e-beam lithography were transferred to the Si<sub>3</sub>N<sub>4</sub>, a-Si and SiO<sub>2</sub> layers through a multi-step RIE process, which generated ~50 nm width and ~60 nm depth trench structures. Then, a selective isotropic etching of SiO<sub>2</sub> by a dilute HF(1:500) induced a round-shaped etch profile along the trench line. Alternatively, the a-Si layer could be oxidized by furnace oxidation to partially close the trench. For both pore (70-80 nm diameter) and trench (~50 nm width) structures, we used an amorphous, conformal ALD TiO<sub>2</sub> film to reduce the pore or trench dimensions down to less than 10 nm. ALD film provided a self-limiting process by remaining void along the pore and the trench, which will serve as fluidic channels less than 10 nm.

#### 3:20pm MN+NC-MoA5 Fabrication of Nickel-tipped Cantilevers for Magnetic Resonance Force Microscopy, S.A. Hickman, Cornell University, J. Van Delden, Eigenphase Technologies, L.E. Harrell, United States Military Academy, S.R. Garner, J.C. Ong, S. Kuenh, J.A. Marohn, Cornell University

Magnetic resonance force microscopy (MRFM) is a technique that may one day allow us to acquire magnetic resonance images of single molecules – an extremely exciting prospect. To date we have demonstrated that MRFM can achieve a sensitivity of ~10<sup>3</sup> proton spins, using a custom-fabricated silicon cantilever with a hand glued, 9 μm diameter magnet tip. By making improved magnetic tips and mitigating surface dissipation, it may be possible to achieve single-proton sensitivity, leading to such exciting prospects as structural determination of large biomolecules. Achieving the attonewton force sensitivity necessary to image single proton spins requires custom-fabricating cantilevers with extreme aspect ratios. In MRFM the force exerted on the cantilever, per spin, is proportional to the field gradient from the cantilever's magnetic tip. Achieving single proton sensitivity therefore also requires dramatically reducing magnet size. Unfortunately, all MRFM tips produced to date have been made by manually affixing magnets one-at-a-time to a cantilever. Even if the tips are ion-beam milled, it is difficult to see how they can be made small enough to detect a single proton. Likewise, ion-beam deposited tips have not yet reached the purity to produce the high magnetic field gradients desired for MRFM. We have developed an electron-beam-lithography (EBL) process for batch fabricating nanoscale tip magnets on ultrasensitive silicon cantilevers. Research by our group has shown that surface induced dissipation is a major source of noise. This surface dissipation results from charge in the cantilever interacting with electric field fluctuations in the sample. This surface dissipation can be minimized by fabricating the magnets overhanging the end of the silicon cantilever. We will present 50-600 nm wide nickel overhanging magnets fabricated by EBL and isotropic plasma etching. We will also present work on producing overhanging magnets via

anisotropic potassium hydroxide etching, and by fabrication of magnets over sacrificial oxide pillars. Our current challenge appears to be preventing the transformation of the magnet metal to metal silicide during the fabrication process. With our designed cantilever, we expect a sensitivity of better than  $10^5$  protons.

**4:00pm MN+NC-MoA7 A Process for Control of the Support Conditions of Nanomechanical Beam Resonators, and Evaluation of the Resulting Impact on Mechanical Dissipation, R. Barton, S.S. Verbridge, B.R. Ilic, H.G. Craighead, J.M. Parpia, Cornell University**

We have developed a fabrication process for suspended nanomechanical beam structures which allows precise control over the clamping conditions, particularly the extent of the overhanging support which results from the usual sacrificial release of such devices. We localize the sacrificial silicon dioxide material to specified locations using a process involving a chemical mechanical polishing step, followed by a deposition and patterning of the structural silicon nitride material. With proper alignment of the beam structures, we have succeeded in controlling or entirely eliminating the overhang which was previously thought to potentially impact the overall dissipation in these resonant systems. We will demonstrate that for a moderate overhang, on the order of ten percent of the resonator length, dissipation measured in terms of resonator quality factor,  $Q$ , is not significantly affected either in vacuum or ambient air. This finding demonstrates that neither the intrinsic material damping at low pressure, or the gas-related damping that is dominant at higher pressures are affected by the support overhang that inevitably results from typical release steps. We anticipate that the control over the clamping conditions we present could prove useful for more complex nanosystems, for example suspended fluid-containing nanochannels which integrate both optical and mechanical means of biomolecular detection.

**4:20pm MN+NC-MoA8 Dynamic-Mode Multidirectional UV Lithography with Liquid State Photoresist, J.K. Kim, Y.K. Yoon, University at Buffalo**

Recently an advanced ultraviolet (UV) lithography scheme, where UV is applied to liquid state photoresist, has been introduced with several advantages such as short process time with quick develop/rinse, and in-situ surface modification.<sup>1</sup> The container with liquid photoresist remains in a horizontal position to vertically incident light, resulting in relatively simple micro structures. In this research, a multidirectional UV lithography process has been developed with liquid state photoresist for complex three dimensional (3-D) microstructures, where the UV source and a substrate containing liquid photoresist have an arbitrary angle each other. A leak free container has been devised to hold liquid photoresist and is capped with the photomask plate. The container set is attached to a computer controlled movable stage with tilting and rotational functionality.<sup>2</sup> A liquid state negative tone photopatternable polyurethane, LF55GN has been utilized for test since it shows fast realization of thick structures with high aspect ratio.<sup>3</sup> While the liquid state photoresist is exposed to UV, the stage is dynamically tilted and rotated in slow speed, at which the friction force should be greater than the inertia effect for the given viscosity of the photoresist and therefore the container and the enclosed liquid move as a single body in a quasi-static mode. This developed scheme provides all advantages of both the high aspect ratio complex 3-D micropatterning of multidirectional UV lithography with  $SU-8^2$  and the rapid process time of UV lithography with liquid state photoresist.<sup>1</sup> As the height of the container determines the height of the structure, several thousand micro meter thick film can be easily realized unlike the spin coating approach where the thickness is limited to several hundred micrometers. Thus complex 3-D structures which are more than 1000 micrometers tall structures have been successfully demonstrated in 30 minutes. Ray tracing with regard to the stage movement has been simulated. The shapes of the fabricated structures show good agreement with those of the simulated structures. A single vertical triangular slab and an array have been demonstrated as test structures.

<sup>1</sup>A. Sayah et al JMEMS 2007, vol.16, no.3, pp.564-570, June

<sup>2</sup>J.K. Kim et al MEMS 2008. Conference, vol., no., pp.399-402, Jan.

**4:40pm MN+NC-MoA9 Development of High Rate Etching for Deep Si Etching in Advanced NLD Plasma, T. Murayama, T. Morikawa, K. Suu, ULVAC, Inc., Japan**

In Si etching such as wafer level packaging technology for MEMS and the formation vertical interconnection of three-dimensional integration LSI, to establish high rate is important problem. Especially under 10um space pattern on 200-mm-diam wafer, it is desired to etch Si at high rates achieving anisotropic features. Commonly, it is essential to produce huge amount of F radicals in Si etching mainly dominated by radical reaction in Si surface, ion assist effect is important to achieve anisotropic feature.<sup>1</sup> Our experiments are conducted using NLD-Si etcher which we introduced sputtering system for sidewall passivation to NLD (magnetic Neutral Loop Discharge) etcher for quartz deep etching.<sup>2,3</sup> In this study, we tried to

improve etching rate using a novel antenna [Multi-Slits rf Antenna] for controlling both ion and F radical density. This antenna consisted of grid type structure in which several slits are set perpendicularly in same direction to RF current. In experiments we used 3 parallel slit antenna in other words 4 parallel current paths structure, expecting high-density plasma is generated because of ion and radical generation area extending in space compared with simple single-turn antenna. Next, for control anisotropic feature, we investigated the dependence of bias rf frequency in high-density NLD plasma using this multi slit rf antenna. We used SF<sub>6</sub> as process gas. Consequently, this antenna improved in etching rate more than two times compared with our conventional antenna. However etching feature shifted bowing. We considered that this result is caused by not only F radical increase but also sheath impedance decrease by high-density plasma. To overcome this sheath impedance decrease, we changed bias rf frequency to 2 MHz from 13.56 MHz. In low bias frequency we etched 4 um space pattern, then high rate and anisotropic feature were achieved etching rate is 10.5 um/min, selectivity (Si/SiO<sub>2</sub>) is 60 over. It is considered that for high rate and high quality Si etching it is very important that the control of incident ion energy distribution in addition to high-density radical produce.

<sup>1</sup> J. W. Coburn and H. F. Winters: J. Appl. Phys. 50 (1979) 3189.

<sup>2</sup> W. Chen, et al.; J. Vac. Sci. Technol., A 19 (2001) 2936.

<sup>3</sup> Y. Morikawa, et al.; Thin Solid Films 515 (2007) 4918.

**5:00pm MN+NC-MoA10 XeF<sub>2</sub> Etching of Si and Mo for MEMS Manufacturing, J.-F. Veyan, Y.J. Chabal, University of Texas at Dallas, X.-M. Yan, A. Londergan, E. Gousev, Qualcomm**

XeF<sub>2</sub> is a widely used isotropic etchant in MEMS and NEMS fabrication because it selectively removes a large variety of pure compounds (e.g. Si, Ge, Mo, W) but not their stoichiometric oxides. Understanding the etching mechanisms is crucial to achieve highly selective etching. We have studied and compared the XeF<sub>2</sub> etching of Silicon oxide-Silicon and Molybdenum Oxide -Molybdenum systems, under typical pressure conditions used in industry (~ few Torr). Using in-situ Infrared Absorption Spectroscopy (IRAS) we have investigated the reaction kinetics and characterized the gas phase, surface and subsurface species after and during the etching process. To reduce side effects due to reaction with the reactor walls and sample holders, an all aluminum chamber has been designed, with Teflon gasket and Kalrez O-ring for sealing. The length of stainless steel tubing for gas transport has also been reduced. To minimize reactions with contaminants such as water and hydrocarbon, the reactor is pumped to  $10^{-5}$  Torr prior any XeF<sub>2</sub> introduction. The sample temperature prior and during XeF<sub>2</sub> exposure is controlled by heaters made with Ta filaments inserted in a ceramic frame imbedded inside the sample holders. A chromel-alumel thermocouple is placed directly in contact with the sample surface. With the sample out of the IR beam path the reaction products are monitored during and after etching process. Transmission is used to probe SiO<sub>2</sub>/Si, and grazing angle reflection to probe MoO<sub>3</sub>/Mo, and surface and subsurface species are detected/identified during and after etch reactions. XeF<sub>2</sub> induces a strongly exothermal reaction with both Si and Mo, producing gaseous SiF<sub>4</sub> (1030 cm<sup>-1</sup>) and MoF<sub>6</sub> (741 cm<sup>-1</sup>), and incorporating Fluorine species into the subsurface region. No reactions have been observed with stoichiometric Silicon Oxide and Molybdenum Oxide, but both oxides are completely removed when overlaying the pure material. The removal of the oxide overlayers is believed to be due to fluorine migration through the oxide and reaction with the elemental Si or Mo at the oxide-element interfaces.

**5:20pm MN+NC-MoA11 A Versatile, Bilayer Resist Method for Creating Silica Microstructures, B.R. Cipriany, B.R. Ilic, H.G. Craighead, Cornell University**

The rapid and widespread acceptance of polydimethylsiloxane (PDMS) in the microfabrication community illustrates the growing importance of versatile, simple, and inexpensive fabrication techniques, particularly for lab-on-a-chip applications. In this context, we demonstrate a new method for creating silica microfluidic networks over nanophotonic structures using a bilayer resist process involving Hydrogen silsesquioxane (HSQ) and a single photolithographic step. Ridge waveguides 180nm tall were formed in silicon nitride using electron beam lithography (JEOL9300FS) and reactive ion etching. A 630nm thick layer of HSQ was spun conformally over the structures and then exposed to oxygen plasma to cross-link a 10nm thin barrier layer. This barrier was robust against photoresist solvents, allowing a bilayer stack to be formed without altering the underlying HSQ bulk. Photoresist was then spun, patterned with optical lithography, and used as a mask layer. An HF based chemical etch was used to transfer the pattern into the barrier layer, followed by development to isotropically dissolve the HSQ bulk. Microfluidic networks formed with this developer-based transfer were self-terminated on the photonic structures without inducing structural damage. We studied the formation of HSQ channels with widths of 1.5-3.1 micrometers and heights of 80-520nm, respectively. Cross-sectional electron micrographs of these channels revealed a sponge-like film composition, which was compacted into a dense, amorphous silica film

during a subsequent high-temperature anneal. Post-anneal measurements with a variable-angle spectroscopic ellipsometer reveal a 17% reduction in film thickness and a negligible complex refractive index over the entire visible spectrum, relative to non-annealed films. Using an inverted microscope with photon counting modules, laser-induced autofluorescence of annealed HSQ was found to be over an order of magnitude less than PDMS, suitable for ultra-sensitive fluorescence spectroscopy. Unlike PDMS, annealed HSQ demonstrated chemical resistance in both aqueous solutions and common solvents. Within our sealed waveguide-microfluidics network, we directly observed flow of fluorophore-labeled deoxyribonucleic-acid (DNA) using fluorescence videomicroscopy. Future applications of this fabrication method include microfluidics integration with MEMS/NEMS, nanowire sensors, or other integrated optical elements.

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