Wednesday Afternoon, November 11, 2009

Plasma Science and Technology
Room: B2 - Session PS2+MN-WeA

High Aspect Ratio and Deep Etching for 3D Integration and Memory
Moderator: S. Hamaguchi, Osaka University, Japan

2:00pm **PS2+MN-WeA1 Advanced DRIE Via Etching**, ***F. Gao****, D. James, K. Kolari, J. Kiihamäki,* VTT Technical Research Centre of Finland, *M. Muggeridge*, Aviza Technology, Inc.

We present 3 different types of interconnection vias fabricated by deep reactive ion etching (DRIE) on silicon substrates. One type of vertical vias with 30µm diameter mask opening are etched through 400µm thick wafer by switched Bosch process, featured by very fast etch rate at about 6µm/min and over 12:1 aspect ratio. The other type of vertical vias are tested on smaller diameters ranging from 1-9µm and etched to 20-50µm deep. Those vias have the minimum undercut and smooth sidewalls achieved by non-switched etching. Another type of tapered vias with 75µm mask opening are etched isotropically in DRIE resulting in over 150µm deep vias with 70-80 degree tapering. Silicon etch selectivity against different mask materials are studied and compared for the vertical vias. Thick resist is thought to be better mask to minimize undercut and via top erosion by reflected ions. Tapered vias have the problem of sidewall roughness from the isotropic etch. Both plasma cleaning and argon annealing methods are tested to smooth the silicon sidewalls in the tapered vias.

2:20pm **PS2+MN-WeA2 The Generation and Removal of Heat during DRIE of High Aspect Ratio Structures in SOI with Buried Cavities**, ***J. Dekker****, F. Gao, J. Kyynäräinen, J. Kiihamäki*, VTT Microelectronics Research Center of Finland

This work examines the accumulation of heat and resulting increase in local temperature and loss of selectivity which may occur when etching high aspect ratio structures in SOI wafers with buried cavities. It is shown that unlike high-load, high-rate etches treated elsewhere, in the case of HAR etches the heat is generated mainly by ion-bombardment. Due to the presence of a cavity beneath the structures being released, which typically include a mass suspended by springs, the heat may only be conducted laterally away from the released structures to the surrounding device layer. During the final stages of DRIE etch and overetch, the heat flow from suspended masses is therefore restricted to occur along the springs which attach the mass to the surrounding device layer. The limited heat conductance of long meander springs in particular is unable to remove the heat generated in suspended structures. As a result, the temperature of the suspended structures increases resulting in reduced fluorocarbon depositon upon them. That, in turn, results in a locally increased etch rate of the oxide mask on the suspended structures. The effects of releasable thermal anchors, which provide additional paths for heat conduction away from the suspended mass and therefore aid in the removal of heat, is also discussed.

2:40pm **PS2+MN-WeA3 Ion Trajectory Prediction at High-Aspect-Ratio Hole Etching by the Combination of On-Wafer Monitoring and Sheath Modeling**, ***H. Ohtake****, S. Fukuda, B. Jinnai,* Tohoku University, Japan, *T. Tatsumi,* OKI Semiconductor Miyagi Co., Ltd., Japan, *S. Samukawa*, Tohoku University, Japan

The abnormal etching profiles, such as bowing, etch stop and twisting, have been reported at high-aspect-ratio hole etching. To avoid the failures, we have to predict the ion trajectory and etching profile precisely by analyzing the sheath area around the hole. In this presentation, we developed the ion-trajectory prediction system at high-aspect-ratio hole by combining the on-wafer monitoring technique and sheath modeling for explaining and predicting the etch stop and twisting. Since our developed on-wafer sensors provide the surface potential, the electron density/ temperature and side-wall resistance of the hole, we can simulate the distribution of electric field in the hole. This system revealed that the sidewall conductivity strongly affects the charge-up and ion trajectory in the high-aspect-ratio hole. It also predict the etch stop and twisting phenomena. Consequently, we believe this prediction system is an effective tool for developing the nano-scaled fabrication.

3:00pm **PS2+MN-WeA4 Enhancement Mechanism of Distortion and Twisting in Ultra High Aspect Ratio Dielectric Etching**, ***H. Mochiki,*** Tokyo Electron AT Ltd., Japan, *K. Yatsuda,* Tokyo Electron Ltd., Japan, *S. Okamoto, F. Inoue*, Tokyo Electron AT Ltd., Japan

It is required to fabricate capacitors with aspect ratio of from 40:1 to 60:1 for DRAM at hp 3x nm and beyond generation, and etching such ultra high aspect cylindrical shapes without distortion and/or twisting is the most difficult challenge. Recently, it has been reported that distortion and twisting were caused by electron shading effects, electrical potential difference between the top and bottom of dielectric during plasma etching. In this paper, we report how distortion and twisting are enhanced, and how they can be minimized.

First of all, electron shading effects are results of electrostatic charge on the surface of etched dielectric material – silicon dioxide, and organic capacitor mask is negatively charged where silicon dioxide surface is positively charged. At these generations, DRAM devices are so largely scaled that their capacitors need to be fabricated very close to each other. Consequently, incident positively charged ions in a cylinder, accelerated by plasma sheath, receive repulsive force from not only the cylinder surface itself but also neighboring cylinder surfaces. We confirmed that grad of distortion and twisting changed by altering the layout of capacitors.

On the other hand, we found that distortion and twisting could also be generated from the very beginning of etching at the low aspect ratio portion by observing the top view of a cylinder every 100 nm-deep from the wafer surface. Moreover, we affirmed that distortion strongly correlated with capacitor etch mask profile when varying it on purpose by changing mask etch conditions. Thus, we clarified that another enhancement mechanism of distortion existed apart from electron shading effects.

Therefore, there are several enhancement mechanisms of distortion and twisting, and it is necessary to address each solution. We divided the enhancement mechanisms of distortion and twisting into two modes, which are generated at low and high aspect ratio, and examined their solutions from the etching point of view, respectively. We conclude that the optimization of capacitor etch mask profile was the most effective solution at the low aspect ratio mode, and higher dissociation plasma with relatively higher plasma density and superimposed DC on CCP (capacitively coupled plasma) improved distortion and twisting at the high aspect ratio mode.

4:00pm **PS2+MN-WeA7 High Rate Deep Si Etching for TSV Applications**, ***I. Sakai****, N. Sakurai, T. Ohiwa*, Toshiba Corporation, Japan **INVITED**

Si etch process for etching deep and high-aspect ratio structures has been studied intensely for applications such as DRAM trench capacitors and MEMS devices. Recently, there is focus on Si etching for TSV (through Si via) applications for 3-D (three-dimensional) LSIs. Dimensions of the TSVs which are being investigated today vary widely, depending on its application and integration scheme. For example, TSV for 3-D packaging of logic devices may be sub-micron to a few microns in diameter and about 10 microns deep. On the other hand, TSVs used in stacking memory devices, the via diameter and depth would be several tens of microns, and, package for CMOS image sensors using TSVs may have via diameters and depths up to 100 microns.

For TSVs up to 10 microns in depth, the conventional Si deep trench etch process for DRAMs can be easily adapted to etching TSVs because of its similar dimensions. The typical etch rate is several microns per minute. On the contrary, etching of very deep holes of depths on the order of tens of microns and up to 100 microns is not within the experience of conventional front-end LSI fabrication processes. In this case, consequently, an extremely high Si etch rate becomes mandatory because of cost issues, especially for TSV applications which require via holes more than 20 microns deep.

To fulfill this requirement for TSV applications, the Si etch process was investigated focusing on the Si etch rate. First, a large via size of 40 microns was studied, and an etch rate of more than 50 µm/min was realized. It was found that the Si etch rate depended on fluorine radical density, so, high rate was obtained by creating a high fluorine radical density condition by using a high pressure condition of 350 mTorr, with a capacitively-coupled plasma (CCP) reactor with a Dipole-Ring Magnet (DRM) and SF_6 gas chemistry. Furthermore, the etch process for smaller holes of 8 microns was studied to realize high etch rates also. The etch process was modified to obtain a straight etch profile, then, via holes were etched to a depth of 60 microns at an etch rate of 24 µm/min.

High rate deep Si etching is realized for TSV application for holes more than 20 microns deep, using CCP RIE with SF_6–based gas chemistry.

4:40pm **PS2+MN-WeA9 Infinitely High Etch Selectivity and Variation of Line Edge Roughness during Etching of Hard-Mask Layer with Patterned Extreme Ultra-Violet**, ***B.S. Kwon***, *J.S. Kim, C.R. Jung, J.S. Park, W. Heo, N.-E. Lee,* Sungkyunkwan University, Korea, *S.K. Lee*, Hynix Semiconductor, Republic of Korea

In the nano-scale Si processing, patterning processes based on multilevel resist structures becoming more critical due to continuously decreasing resist thickness and feature size. In particular, highly selective etching of the first dielectric layer with resist patterns and control of critical dimension (CD) and ling edge roughness (LER) are of great importance. In this work, process window for the infinitely high etch selectivity of silicon oxynitride (SiON) layers to EUV resist and variation of LER of extreme ultra-violet (EUV) resist was investigated during etching of SiON/EUV resist in a $CH_2F_2/N_2/Ar$ and $CH_2F_2/N_2/O_2/Ar$ dual-frequency superimposed capacitive coupled plasma (DFS-CCP) by varying the process parameters, such as the CH_2F_2 and N_2 flow ratio, low-frequency source power (P_{LF}) and O_2 flow rate. It was found that the CH_2F_2/N_2 flow ratio was found to play a critical role in determining the process window for infinite SiON/EUV resist etch selectivity, due to the differences in change of the degree of polymerization on SiON and EUV resist. Control of N_2 flow ratio gave the possibility of obtaining the infinitely high etch selectivity by keeping the steady-state hydrofluorocarbon layer thickness thin on the SiON surface due to effective formation of HCN etch by-products and, in turn, in continuous SiON etching, while the hydrofluorocarbon layer is deposited on the EUV resist surface. On the other hand, CD size and LER tend to increase with increasing CH_2F_2/N_2 flow ratio.

Inkjet Technology: Printing, Materials Processing, and Microfluidics Fundamentals Topical Conference
Room: B3 - Session IJ+BI+MN+SE+AS-ThM

Inkjet Technology: Novel and Emerging Applications
Moderator: C. Klapperich, Boston University

8:00am **IJ+BI+MN+SE+AS-ThM1 An Overview of the Use of Ink-jet Technology for Non-traditional and Emerging Applications**, ***D.B. Wallace***, MicroFab Technologies, Inc. **INVITED**

In the last decade ink-jet printing technology has come to be viewed as a precision microdispensing tool. Today, this tool is being used in a wide range of manufacturing and instrument applications. Manufacturing applications include electrical (solders & nanometal conductors) & optical (microlenses & waveguides) interconnects; sensors (polymers & biologicals); medical diagnostic tests (DNA, proteins, cells); drug delivery (microspheres, patches, stents); scaffolds for tissue engineering; nanostructure materials deposition; and MEMS (Micro-Electrical-Mechanical) devices and packaging. Instrument applications using ink-jet technology have received less notice than manufacturing applications, but represent a growing class. Applications include protein identification (peptide mass fingerprinting, ion mass spectrometry tissue imaging) and structure analysis (protein crystallization); laser surgery and machining; medical diagnostic instruments; extreme ultra-violet (EUV) radiation generation; and explosive detector calibration. This paper illustrates some of the manufacturing and instrument applications of ink-jet technology.

8:40am **IJ+BI+MN+SE+AS-ThM3 Inkjet Printing for Bioengineering Applications**, ***T. Boland***, Clemson University **INVITED**

We will present the inkjeting of bioink, which may include active compounds such as drugs and living cells as well as non-active, scaffolding materials to build two- and three-dimensional constructs for medical treatment. The technology faces several limitations that present interesting engineering opportunities. The nature and scope of the problems will be discussed in the context of the fabrication of microvasculature. The current tissue-engineering paradigm is that successfully engineered thick tissues must include vasculature. As biological approaches alone such as VGEF have fallen short of their promises, one may look for an engineering approach to build microvasculature. Layer-by-layer approach for customized fabrication of cell/scaffold constructs have shown some potential in building complex 3D structures. With the advent of cell printing, one may be able to build precise human microvasculature with suitable bioink. Human Microvascular Endothelial Cells (HMEC) and fibrin were studied as bioink for microvasculature construction. Endothelial cells are the only cells to compose the human capillaries and also the major cells of blood vessel intima layer. Fibrin has been already widely recognized as tissue engineering scaffold for vasculature and other cells, including skeleton/smooth muscle cells and chondrocytes. In the study presented here, we precisely fabricated micron-sized fibrin channels using a drop-on-demand polymerization. This printing technique uses aqueous processes that have been shown to induce little, if any, damage to cells. When printing HMEC cells in conjunction with the fibrin, we found the cells aligned themselves inside the channels and proliferated to form confluent linings. Current studies to characterize the biology and functionality of these engineered microvascular structures will be presented. These data suggests that a combined simultaneous cell and scaffold printing can promote HMEC proliferation and microvasculature formation.

9:20am **IJ+BI+MN+SE+AS-ThM5 Inkjet Printing for MEMS Fabrication**, ***J.A. Kubby***, *O. Azucena*, University of California, Santa Cruz, *C.L. Goldsmith, D. Scarbrough*, MEMTronics Corporation, *A.S. Mangalam*, Tao of Systems Integration, Inc. **INVITED**

In this presentation we will review the use of inkjet printing to fabricate Micro-Electro-Mechanical Systems (MEMS). We are investigating the use of sintered silver nanoparticle inks for the structural layer and polymers for the sacrificial layer in printed MEMS fabrication. As an example, inkjet printing technology has been used to fabricate microwave transmission lines for an RF MEMS switch on a glass substrate (with MEMTronics Corporation). 50 mm resolution was obtained using 10 pL drop volumes on a Corning 7740 glass substrate. The conductivity of the sintered silver structures were 1/6 that of bulk silver after sintering at a temperature much lower than the melting point of bulk silver. A comparison of the DC resistance of the sintered silver shows that it can match the performance for electroplated and etched copper. Printed coplanar lines demonstrated losses of 1.62 dB/cm at 10 GHZ and 2.65 dB/cm at 20 GHz. We will also discuss printing MEMS hot-wire anemometer sensors for use in aeronautical applications (with Tao of Systems Integration).

10:40am **IJ+BI+MN+SE+AS-ThM9 Formation and Surface Characterisation of a Combinatorial Acrylate Polymer Microarray Produced by an Ink-Jet Printer**, ***A.L. Hook***, *J. Yang*, University of Nottingham, UK, *D.G. Anderson, R.S. Langer*, Massachusetts Institute of Technology, *M.C. Davies, M.R. Alexander*, University of Nottingham, UK

Polymer microarrays are emerging as a key enabling technology for the discovery of new biomaterials. This platform can readily be screened for properties of interest and for correlating surface chemistry with biological phenomenon. A method for forming polymer microarrays has been developed whereupon a contact printer is used to deposit nanolitre volumes of premixed acrylate monomer and initiator to defined locations of a glass slide with subsequent UV irradiation[1]. This results in polymerisation occurring on the slide, offering a useful high throughput materials discovery platform. The identification of relationships between cell response to these materials and surface properties is facilitated by high throughput analysis of this slide format[2,3]. Here, we have formed these polymer microarrays for the first time using ink-jet printing, to offer flexibility of slide production. Characterisation was achieved using a high throughput surface analysis approach, including the techniques of X-ray photoelectron spectroscopy, time-of-flight secondary ion mass spectroscopy and sessile drop water contact angle measurements[2]. Of particular interest were polymers containing ethylene glycol functionality that were investigated for their switchable properties under biologically relevant conditions.

[1] D. G. Anderson, S. Levenberg, R. Langer, *Nat.Biotechnol.* **2004**, *22*(7), 863.

[2] A. J. Urquhart, D. G. Anderson, M. Taylor, M. R. Alexander, R. Langer, M. C. Davies, *Adv.Mater.* **2007**, *19*(18), 2486.

[3] Y. Mei, S. Gerecht, M. Taylor, A. J. Urquhart, S. R. Bogatyrev, S. W. Cho, M. C. Davies, M. R. Alexander, R. S. Langer, D. G. Anderson, *Adv. Mater.* **2009**, 21(early view), doi:10.1002/adma.200803184.

11:00am **IJ+BI+MN+SE+AS-ThM10 Development of an Inkjet Printed Drug Formulation**, ***N. Scoutaris***, *C.J. Roberts, M.R. Alexander*, Nottingham University, UK, *P.R. Gellert*, AstraZeneca, UK

The potential application of ink-jet printing technology as a novel drug formulation technique is examined in this study. Since the inkjet printing technology offers high accuracy of fluids, a success implementation of the project can offer the capability to produce precise amounts of medicines, tailored for each patient.

Felodipine, an antihypertensive drug, was used as an example of an active pharmaceutical ingredient (API), and polyvinyl pirrolidone (PVP) as an excipient. These were dissolved at various ratios in a mixture of ethanol and DMSO (95/5). Using a piezoelectric driven dispenser, picolitre size droplets of the solutions were dispensed onto suitable hydrophobic substrates. The dried products were characterized using AFM, localized nano-thermal analysis and high resolution vibrational spectroscopy (ATR-IR and Raman). Results indicate intimate mixing of the micro-dot API and excipient mixtures. Specifically, ATR-IR confirmed the interaction of felodipine and PVP by means of hydrogen bonding. Nanothermal analysis indicates a single glass transition point which is lowered as the API concentration increases. Finally, confocal Raman microscopy mapping on single droplets allows the visualization of the homogeneous distribution of the drug. These results are a promising first step to ink jet printing of pharmaceuticals.

References

1. Peter A. Melındez, et al., Thermal inkjet application in the preparation of oral dosage forms: Dispensing of prednisolone solutions and polymorphic characterization by solid-state spectroscopic techniques. *Journal of Pharmaceutical Sciences*, 2007. **97**(7): p. 2619 - 2636.

2. Karavas, E., et al., Investigation of the release mechanism of a sparingly water-soluble drug from solid dispersions in hydrophilic carriers based on physical state of drug, particle size distribution and drug-polymer interactions. *European Journal of Pharmaceutics and Biopharmaceutics*, 2007. **66**(3): p. 334-347.

3. Karavas, E., et al., Combining SEM, TEM, and micro-Raman techniques to differentiate between the amorphous molecular level dispersions and nanodispersions of a poorly water-soluble drug within a polymer matrix. *International Journal of Pharmaceutics, 2007.* **340**(1-2): p. 76-83.

11:20am **IJ+BI+MN+SE+AS-ThM11 Fabrication of Plastic Biochips via *in situ* Inkjet Oligonucleotide Array Synthesis**, ***I. Saaem***, *K. Ma, J. Tian*, Duke University

With the foreseeable integration of microfluidics and microarrays, polymers stand to play a critical role. Generally, arrays are constructed on glass, silicon, membranes, or polyacrylamide matrices. The preference of such materials makes the marriage of arrays and microfluidics fraught with challenges such as developing low-cost manufacturing methods and simultaneously scaling rapidly for diverse applications and chip designs. In addition, deposition or synthesis of the requisite biomolecule reliably in defined surface geometries is a challenging task. We try to alleviate these problems by utilizing the steadily maturing art of inkjet printing on polymer substrates. Polymeric, or plastic, biochips have several advantages in cost, durability, the ability to scale to industrial techniques and possibly serve as disposable point-of-care devices. In our studies, we utilized an inkjet based *in situ* oligonucleotide synthesis platform that uses salvaged printheads from commercial printers. The platform utilizes standard four-step phosphoramidite chemistry with some modifications in order to synthesize oligonucleotides on functionalized substrates. A sensitive pressurization system is used to ensure print quality and an on-board vision system enables substrate registration and synthesis monitoring. Using this platform we synthesized oligonucleotides on prepatterned functionalized plastic slides. Such patterned substrates help in proper droplet formation and fluid mixing on the surface while mitigating satellite and irregular drops, which can lead to cumulative synthesis errors. Functional integrity of synthesized oligonucleotides was confirmed by hybridization with complementary strands. Being able to hot emboss microfluidic structures directly onto plastic slides in combination with the ability to generate arbitrary sequences provides diagnostic capabilities as well as the means to harvest pools of cheap oligonucleotides on demand. Importantly, our results show that the combination of technologies presented is a suitable strategy of fabricating plastic biochips at a cost-effective industrial scale.

11:40am **IJ+BI+MN+SE+AS-ThM12 Study on the Effects of Particle Size and Substrate Surface Properties on the Deposition Dynamics of Inkjet-Printed Colloidal Drops for Printable Photovoltaics Fabrication**, *S. Biswas*, ***Y. Sun***, Binghamton University

Using fluorescence microscopy, the inkjet deposition dynamics of monodispersed polystyrene particles in the size range of 0.02 to 1.1 µm have been studied on glass, Ar plasma cleaned glass, and PDMS coated glass substrates. The results show that the substrate properties play an important role in determining the final dried patterns formed by the colloidal particles. Our observations also reveal that particle size and contact angle formed by the solvent in the dispersion determine how close to the contact line the particles can be deposited. It is found that the diameter of the dried deposited features decrease with the increase in hydrophobicity of the substrates, irrespective of particle sizes. On Ar plasma treated glass ($\theta_A = 13°$), the smaller particles (0.02 & 0.2 µm) show larger depositions than the bigger 1.1 µm particles. Similar type of behavior of the dried deposited features are also observed on clean glass samples ($\theta_A = 36°$). In contrast, on PDMS coated glass ($\theta_A = 111°$), the behavior of the contact line diameter with the evaporation of the drop is similar for all types of particles. On an average, the diameters of the dried deposited features on PDMS coated glass substrates are independent of particle sizes. This study can serve as a realistic experimental model system for a number of fundamental queries on how the final deposition microstructure depends on the ink formulation and substrate properties. The knowledge obtained here can be explored further to optimize process parameters for the fabrication of hybrid solar cells with improved morphology and device properties.

MEMS and NEMS
Room: A8 - Session MN+GR-ThM

Graphene and Carbon Based MEMS/NEMS Devices
Moderator: B.R. Ilic, Cornell University

8:00am **MN+GR-ThM1 Graphene Mechanics, Tribology, and NEMS Resonators**, ***J. Hone***, Columbia University **INVITED**

This talk will describe our work toward fundamental understanding of the mechanical and and tribological properties of graphene, and its application in nano-electromechanical devices (NEMS). We have used nanoindentation to measure the elastic stiffness and ultimate strength of single graphene sheets[1]. These measurements show that graphene is the strongest material ever measured, with an ultimate strength of 130 GPa at an ultimate strain of over 25%. As such, it is the first material whose mechanical properties can be probed deep into the nonlinear elastic regime. We have also measured the frictional behavior of graphene and other related two-dimensional materials. These materials all show an unexpected strong dependence of the frictional force the number of atomic layers, with thinner samples demonstrating larger friction. Finally, we have demonstrated electronic readout of graphene nanomechanical resonators. These devices show highly tunable mechanical resonances in the range 20-300 MHz, with large output signal compared to other NEMS of comparable size. The quality factor of these resonators increases from ~100 at room temperature to ~10,000 at low temperature. The measured sensitivity of these resonators to applied mass shows their promise as multifunctional sensors.

[1] Changgu Lee, Xiaoding Wei, Jeffrey Kysar, J. Hone, “Measured elastic properties and ultimate strength of monolayer graphene,” *Science* **321**, 385 (2008)

8:40am **MN+GR-ThM3 Enhanced Stabilities in Resonant Response of Carbon Nanotube Network Reinforced Al Thin-Film Nanomechanical Resonators**, ***Y.D. Kim***, *J.H. Bak, J. Lee, S.R. Lee, K. Char, S. Hong, Y.D. Park*, Seoul National University, South Korea

With superior mechanical properties, single-walled carbon nanotubes (swCNT) are an attractive reinforcement component for nanoscale composites, based in either polymer or metal matrices [1]. Recently enhancements in mechanical properties of metallic thin-films reinforced by CNTs have been reported [2]. Self-consistent results from quasi-static and dynamic flexural measurements indicate the elastic modulus to nearly double with the inclusion of self-assembled swCNT network layer in Al thin-films [3]. Here, we present significant enhancements in resonant frequency stability of CNT network reinforced Al thin-film nanomechanical resonators. We characterize the stability in the resonant response either by long-cycle measurements or with applied stress. Long-cycle ($>10^{11}$) dynamic flexural measurements show suppression of anelastic effects, which limits the applicability of metallic thin-films nanomechanical resonators. Application of stress is accomplished by two differing methods. A tensile stress is applied by using a ‘chip-bending’ method. A compressive stress is applied through thermal-elastic effects from Joule heating. For both cases, a significant stability in CNT network reinforced Al thin-film resonant response is observed.

[1] W.A. Curtin and B.W. Sheldon, Materials Today 7, 44 (2004); J.N. Coleman et al., Advanced Materials 18, 689 (2006).

[2] Kang et al., Advanced Materials 19, 427 (2007).

[3] J.H. Bak, Y.D. Kim, et al., Nature Materials 7, 459 (2008).

9:00am **MN+GR-ThM4 Carbon Nanostructures on Microscrolls**, ***J.W. Choi***, Kyung Hee University, Republic of Korea

Thin bimetallic films on silicon substrates are formed in microscale scroll when the substrate temperature varies. To increase the functional properties of the bimetallic scroll, carbon nanostructures are directly integrated to the scroll. The formation of the bimetallic scroll and the direct integration of carbon nanostructures are studied for various apllications including NEMS, sensors, energy storage devices.

9:20am **MN+GR-ThM5 Thin Smooth Carbon Nanotube/Polymer Composite Membranes**, ***L. Pei***, *R. Vanfleet, M.R. Linford, R.C. Davis*, Brigham Young University

We have developed a new and straightforward method for fabricating freely suspended ultrathin carbon nanotube (CNT) membranes. A smooth transferrable CNT sheet was first made from vertically aligned carbon nanotube (VACNT) forests by placing mixed cellulose ester (MCE) filter paper on a VACNT forest and using a roller to both compress the forest and transfer the nanotubes to the filter paper. The compressed CNT film was then transferred to a solid substrate and the MCE was subsequently dissolved, leaving the CNT film on the substrate. Nanotube – polymer composite films were then fabricated by spin casting a polymer layer on top of the transferred CNT sheet. If the solid substrate was coated with a polymer film prior to CNT transfer, a polymer/CNT/polymer sandwich was created. The composite membranes were subsequently released from the substrate. Characterization of the films and membranes preformed by scanning electron microscopy, atomic force microscopy, and by strength testing will be presented.

9:40am **MN+GR-ThM6 Nanostructuring of Ultrananocrystalline Diamond (UNCD) Thin Films Via Block Copolymer Lithography**, ***M. Ramanathan***, *S.B. Darling, A.V. Sumant, O.H. Auciello*, Argonne National Laboratory

Diamond is in many ways an optimal material for numerous technological, industrial and biological applications because of its exceptional physical and chemical properties. In addition to high hardness, diamond is stiff, biocompatible and wear resistant. Nanopatterning of diamond surfaces is critical for the development of diamond-based MEMS/NEMS, such as resonators or switches. Micro/nano structuring of diamond materials is typically associated with conventional lithographies such as photolithography or electron beam lithography. In this paper, we demonstrate a simple process, known as block copolymer (BCP) lithography, of nanostructuring ultrananocrystalline diamond (UNCD) surfaces. In BCP lithography, nanoscale self-assembled polymeric domains serve as an etch mask for pattern transfer. We used thin films of a cylinder-forming organic–inorganic BCP, poly (styrene-*block*-ferrocenyldimethylsilane), PS-*b*-PFS, as an etch mask on UNCD. Orientational control of the etch masking cylindrical PFS blocks are achieved by manipulating the polymer film thickness in concert with the annealing treatment. For films much thinner than the equilibrium periodicity of the microdomains, the cylinders spontaneously orient themselves perpendicular to the substrate. On the other hand, films with thickness close to the equilibrium periodicity exhibit in-plane orientation. We have observed that surface roughness of UNCD plays an important role in transferring the pattern. Reactive ion etching (RIE) using oxygen gas was used to etch the exposed areas of UNCD. Arrays of both UNCD posts and wires have been created using the same starting polymeric materials as the etch mask.

10:40am **MN+GR-ThM9 Fabrication of a Reusable Template Based on Ultrananocrystalline Diamond for Electrodeposition of Metal and Semiconductor Micro/Nanowires**, ***D.B. Seley***, *D.A. Dissing,* University of Wisconsin - Stevens Point, *A.V. Sumant, R. Divan, S. Miller,* Argonne National Laboratory, *E.A. Terrell,* University of Wisconsin - Stevens Point, *O.H. Auciello,* Argonne National Laboratory, *M.P. Zach*, University of Wisconsin - Stevens Point

Electrodeposition is a versatile technique that has been used for the synthesis of nanowires. There are several methods available for the synthesis of nanowires, each requiring some form of template, which is not easily reusable. A recent report uses a combination of optical lithography, and the conductive edges of a metal for the deposition of nanowires, but the patterned metal is sacrificial, requiring a multi-step process to regenerate the electrode (1).

We demonstrate a top-down approach involving lithography and reactive ion-etching of ultrathin (150 nm) undoped and N-doped ultrananocrystalline diamond (UNCD) stack defining nanoelectrodes for subsequent electrodeposition of micro/nanowires of desired materials. Once this template consisting of arrays of nanoelectrodes of various shapes has been made, it is a permanent reusable template for synthesis of micro- and nanowires. Subsequent manufacture of nanowires becomes almost as simple as using a rubber stamp and ink. The multilayer diamond electrode provides low adhesion to the deposited materials which allows for easy transfer of the resulting electrodeposited micro- or nanostructures onto an adhesive polymer. Each set of structures is removed, regenerating a pristine electrode surface for multiple depositions without needing to repeat the difficult lithography steps for each batch of wires made. The combination of unique electrical and chemical properties of UNCD is promising to allow mass production of uniform patterned nanostructures. Materials electrodeposited until now include: Pb, Au, Cu, Pd, Pt, Co (non-aqueous), Te, CdTe, and CdS.

Acknowledgments

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Additional support was provided by UPDC Fund (UWSP), WiTAG and University of Wisconsin-System.

1. E. J. Menke, M. A. Thompson, C. Xiang, L. C. Yang and R. M. Penner, *Nature Materials*, **5**, 914 (2006).

11:00am **MN+GR-ThM10 Fabrication and Characterization of Ultrananocrystalline Diamond Nanowires for Developing Next Generation of Nanoelectronic Devices**, ***A.V. Sumant***, *L.E. Ocola,* Argonne National Laboratory, *X.P. Wang,* University of Puerto Rico, *D.O. Lopez, O.H. Auciello, D.C. Mancini*, Argonne National Laboratory

Recently, there is tremendous amount of interest in making diamond nanowires (DNWs) and diamond nano-rods (DNRs), due to their extraordinary mechanical, electrical, and optical properties as predicated by theory, however, synthesizing or fabricating these quasi1-dimensional sp^3 nanostructures is proved to be very challenging. To date, only few attempts have been reported either by etching single crystal diamond from top-down process to produce diamond nano-rods (DNRs) or by coating Si nanowires with nanocrystalline diamond. We report a method based on e-beam lithography and reactive ion etching of ultrananocrystalline diamond (UNCD), to produce UNCD nanowires (UNCDNWs) and UNCD nano-rods (UNCDNRs) with nanowire diameter as small as 20 nm. Since they are produced by lithographic approach, they can be fabricated almost at will in well defined position with nanometer scale precision. We have fabricated Nitrogen doped UNCDNWs and characterized them using Raman spectroscopy (UV and visible) and TEM microscopy. We will discuss about preliminary nanostructural studies of UNCDNWs and electrical measurements. The ability to fabricate UNCDNWs and UNCDNRs gives an opportunity to study fundamental mechanism of transport processes in diamond nanowires, which will enable new ideas and possibility of fabricating new functional nanoelectronic devices and sensors with increased sensitivity for a variety of applications in nanotechnology.

Thin Film
Room: B4 - Session TF-ThM

Nanostructuring Thin Films II
Moderator: P.D. Rack, University of Tennessee

8:00am **TF-ThM1 Nanostructure of Gold Film Over Vertically Aligned Carbon Nanofiber Surface Impact on Immobilization of Alkanethiols**, ***R. Desikan***, North Carolina State University, *T.E. McKnight,* Oak Ridge National Laboratory, *P.D. Rack,* University of Tennessee, *A.V. Melechko*, North Carolina State University

Chemical self-assembly has been widely used for several applications including surface modification and functionalization. The physics and chemistry of alkanethiol self-assembled monolayers (SAM) has been extensively studied. It is well known that SAM structures of alkanethiol are strongly influenced both by the surface structure of the underlying gold substrate. It has also been shown that the adsorption of alkanethiols onto gold surfaces having large, flat grains produces high-quality self-assembled monolayer. In this study we have investigated the effect of SAM formation over carbon nanofibers coated with gold thin film. The film nanostructure is strongly affected by the morphology of the surface on these high aspect ratio 3D features. The thiolated molecules are chosen due to the strong affinity of sulfur head groups with the gold surface of the carbon nanofibers. The difference in surface stress response alkanethiol of adsorption in vapor phase on large- and small-grained gold correlates well with differences in alkanethiol coverage and SAM structure on those substrates. We will present observations of how the surface structure of the underlying gold substrate influences the kinetics of SAM formation. It has been shown earlier that in the case of gold surfaces with small grain sizes (<100 nm), access to the high coverage, standing-up phase is inhibited. A comparison of annealed for increasing grained gold surfaces on the fibers to as deposited film will be discussed. The characterization of molecular coverage as a function of the gold grain size with an x-ray photoelectron spectroscopy will be presented.

8:40am **TF-ThM3 Spontaneous Growth of In-whiskers from In-Y Thin Films Prepared by Combinatorial Magnetron Sputtering Technique**, ***T. Takahashi***, *A. Abdulkadhim, D. Music, J.M. Schneider*, RWTH Aachen University, Germany

In-Y binary thin films with a composition gradient were prepared using a combinatorial magnetron sputtering technique. In-whiskers grow spontaneously from the film surface at room temperature upon exposure to air. Whisker morphology and population vary with the In to Y ratio. An appreciable amount of In-whiskers is formed at film compositions close to In-25 at.%Y. The In-whisker thickness ranges from a few hundreds nanometers to a few micrometers.

In order to identify the whisker growth mechanism, temporal changes of the film surface upon air exposure were captured using scanning electron microscopy. X-ray micro diffraction was employed for studying the structural evolution during the In-whisker growth in air. The results show that the In-whiskers grow not from the tip but from the root. The whisker growth rate was as high as 150 nm/s. The growth of In-whiskers is found to be related to the incorporation of oxygen into the film during air exposure. Correspondingly, the In concentration within the film decreases as In-whiskers grow. The mechanism of the spontaneous In-whisker growth presented here can be understood based on the stress-induced extrusion of In-whiskers due to the selective room temperature oxidation of Y in sputtered In-Y thin films.

9:00am **TF-ThM4 Photothermally Induced Microchemical Functionalization of Organic Monolayers**, ***A. Schroeter***, *B. Klingebiel, N.O. Hartmann*, University of Duisburg-Essen, Germany

Photochemical routines are widely recognized as a versatile means to fabricate multifunctional patterned organic monolayers with laterally alternating chemical terminations. The lateral resolution, in turn, usually is limited by optical diffraction, that is, the fabricated structures are not much smaller than the wavelength even when highly focusing optics are used. A simple routine for sub-wavelength nanopatterning of organic monolayers relies on photothermal processes, which take advantage of nonlinear effects

[1]. In photothermal processing a focused laser beam is used to locally heat the substrate and initiate thermal decomposition of the monolayer. Silane-based monolayers can be patterned from the micrometer-range down to the sub 100 nm range. In analogy to photochemical routines, it is tempting to explore photothermal procedures which allow to locally functionalize organic monolayers. Here we report on a simple photothermal procedure for local bromination of organic monolayers [2]. Surface-oxidized silicon samples are coated with alkylsiloxane monolayers. Local irradiation with a focused beam of an argon ion laser (λ = 514 nm) in gaseous bromine leads to bromination of the monolayer in a confined micrometer-sized reaction zone. While irradiation induces photolysis of bromine molecules in the gas phase, the local temperature rise of the substrate in the focal area allows for bromination of the alkyl chains. Thus locally brominated surface structures are fabricated. In conjunction with other chemical transformations this provides an efficient route to a broad variety of functional groups. This allows to build up functional surface architectures via directed self-assembly of nanoscopic building blocks such as noble metal nanoparticles and stimuli-responsive polymers. Mechanistic aspects and prospects of photothermal routines in micro- and nanofabrication of multifunctional organic monolayers are discussed.

1. D. Dahlhaus, S. Franzka, E. Hasselbrink, N. Hartmann, Nano Lett. 6 (2006) 2358.

2. B. Klingebiel, A. Schröter, S. Franzka, N. Hartmann, ChemPhysChem, in press.

9:20am **TF-ThM5 Composite Nanoparticles Produced Using Plasma-Enhanced Chemical Vapor Deposition of SiO_2 and Amine-Containing Films**, ***J.C. Shearer***, *E.R. Fisher*, Colorado State University

TiO_2 and Fe_2O_3 nanoparticles are of significant importance in both chemical and biological applications. TiO_2 nanoparticles are used in paint, coatings, food, solar technology and many other areas. Fe_2O_3, as well as other magnetic nanoparticles, are used in the biomedical industry in drug delivery schemes as well as for magnetic resonance imaging contrast agents. Silica-coated TiO_2 and Fe_2O_3 nanoparticles have distinct properties and enhanced functionality over those of uncoated nanoparticles. Plasma-enhanced chemical vapor deposition (PECVD) was employed to conformally coat TiO_2 and Fe_2O_3 nanoparticles with SiO_x and amine-containing films, thereby creating composite nanomaterials. Hexamethyldisiloxane (HMDSO)/O_2 plasmas were used to create SiO_2 and $SiO_xC_yH_z$–coated nanoparticles and pulsed hexylamine (HexAm) plasmas were used to create amine-containing hydrocarbon materials, all of which were analyzed using Fourier-transform infrared spectroscopy (FTIR), x-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and magnetic susceptibility. Films deposited on the nanomaterials showed little difference from those deposited on flat substrates. The performance of the TiO_2 nanoparticles was tested using UV-vis spectroscopy to determine dispersion characteristics of SiO_x-coated TiO_2 materials, which can give information about the agglomeration of the nanoparticles in solution. Notably, the coated materials stay dispersed longer in polar solvents, suggesting the coated nanoparticles may be better suited for applications involving colloidal suspensions. Magnetic susceptibility characterized the magnetic properties of the Fe_2O_3 nanoparticles before and after film deposition. Comparison of the mechanical and chemical properties of different composite nanomaterials will be discussed along with the influence of film composition on performance.

9:40am **TF-ThM6 Tailoring Local Conductivity by the Formation of Ag Nanoparticles in SiO_2 Xerogel Films**, ***M.F. Bertino***, *R.J. Caperton, A.A. Baski*, Virginia Commonwealth University

Thin SiO_2 xerogel films (~200 nm) were fabricated by dip coating and were doped with Ag^+ by adding $AgNO_3$ to the parent solution. Nanoparticles were then fabricated inside the pores of these films by either exposing them to ultraviolet (UV) illumination or by locally injecting charge using a conducting atomic force microscope (CAFM). In both cases, reduction of the Ag^+ ions to the metallic state and the subsequent formation of Ag nanoparticles was observed by optical absorption spectroscopy and X-ray diffraction. Surprisingly, the formation of these Ag nanoparticles was accompanied by a decrease in the electrical conductivity of the films. For exposed regions with nanoparticle formation, CAFM measurements demonstrated no measurable current (< 1 pA) at sample bias voltages above 10 V. We attribute this decreased conductivity to a change in the morphology of the conducting Ag species in the film. Before reduction, Ag^+ ions are attached to negatively charged pore walls in a comparatively dense packing and produce a conducting film. After reduction with UV exposure or CAFM charge injection, the silver metal agglomerates into conducting nanoparticles that no longer form a percolated network, leading to insulating behavior. Local modification of the conductivity on the nanometer-scale is possible by operating the CAFM with an applied voltage above 6 V and scanning a defined pattern. Subsequent imaging of the area at lower voltage then shows insulating behavior in the previously patterned regions. Larger-scale patterning on the micron-to-mm scale is possible by utilizing a mask when exposing the film to UV illumination. Extensions of this method to the fabrication of photonic and plasmonic devices is being explored.

10:40am **TF-ThM9 Synergistic Ag (111) and Cu (111) Texture Evolution in Phase Segregated $Cu_{1-x}Ag_x$ Magnetron Sputtered Composite Thin Films**, ***D.I. Filoti***, *A.R. Bedell, J.M.E. Harper*, University of New Hampshire

We investigated the texture and microstructure evolution of $Cu_{1-x}Ag_x$ composite thin films through x-ray diffraction pole figures as a function of composition for $x \leq 0.5$. As-codeposited at room temperature by magnetron sputtering, the fcc composite $Cu_{1-x}Ag_x$ emerge as a phase-segregated thin film, when the Ag volume fraction represents more than 15 at. % up to 50 at. %, or as a single phase thin film when Ag volume fraction represents less than 15 at. %. The texture evolution of Cu (111) and Ag (111) in phase-segregated Cu-Ag thin films proves to be synergistically enhanced when compared to pure copper or silver thin films. Not only is a stronger perpendicular (111) fiber texture obtained, but also an in-plane alignment of Ag (200) develops related to deposition direction and composition. By the use of transmission electron microscopy we observed a decrease in grain size in Cu-Ag composite films as compared with pure copper and silver films. These Cu-Ag thin films are being evaluated for antimicrobial applications.

11:00am **TF-ThM10 Plasmonic Phenomena in Indium Tin Oxide and ITO-Au Hybrid Films**, *S. Franzen, C. Rhodes, M. Cerruti, R.W. Gerber,* ***M. Losego***, *J.-P. Maria, D.E. Aspnes*, North Carolina State University

For more than 100 years the plasmonic periodic table has been dominated by two elements, Ag and Au. The change in the surface plasmon polariton (SPP) signal in Au thin films is currently one of the most widely used methods for detecting binding interactions in biological systems. Despite broad interest, there has been sparing fundamental research into new plasmonic materials. Here, we elucidate some equivalences regarding plasmonic phenomena in conducting metal oxides, specifically indium tin oxide (ITO), and Au. In contrast to Ag and Au, conducting metal oxides offer the possibility of tuning both the location of the metallic resonance and its width according to deposition conditions. We investigate properties of ITO and ITO/Au layers by reflectance spectra R_p and R_s obtained for light polarized parallel and perpendicular, respectively, to the plane of incidence. Data were obtained in the Kretschman configuration. These data reveal two distinct types of plasmonic phenomena, one due to a capacitive-type oscillation that can only occur for very thin conducting films and the second being the usual surface plasmon resonance. One or the other can be realized either by changing the ITO thickness or by depositing either metallic or nanostructured Au on the ITO to change boundary conditions. The results can be understood completely through a combination of the Drude model for free carriers in a metal, Bruggeman effective-medium theory, and the Fresnel equations. This provides a new dimension for engineering plasmonic phenomena for investigations of molecules adsorbed at interfaces.

11:20am **TF-ThM11 Micro and Nanostructured Interfaces for Therapeutic Delivery**, ***T.A. Desai***, University of California, San Francisco
INVITED

Efficient drug delivery remains an important challenge in medicine. Continuous release of therapeutic agents over extended time periods and in accordance to a pre-determined temporal profile; local delivery at a constant rate to overcome systemic toxicity; improved ease of administration, and increasing patient compliance are some of the unmet needs of the present drug delivery technology. This talk will discuss in vivo drug delivery strategies that capitalize on the strengths of micro and nanofabrication. By taking advantage of our ability to control topography and chemistry at submicron size scales, we have developed organic and inorganic interfaces which modulate cell function while at the same time allow for controlled drug release kinetics. Due to our ability to create monodisperse features (pores, wires, and hollow tubes)as small as several nanometers in a variety of non-planar biocompatible materials, these interfaces offer advantages in their reproducibility, stability, and their ability to intimately contact cell and tissue surfaces. Such nanoengineered interfaces may be optimized for biomolecular selectivity and surface bioactivity, leading to unique interfacial properties not achieved through existing drug delivery approaches. Nanotechnology can add functionality to current drug delivery platforms while becoming an enabling technology leading to new basic discoveries in the biological sciences.

BioMEMS Focus Topic
Room: A8 - Session BM+MN+MS+TF+BI-ThA

Advances in Microfluidics for BioMEMS
Moderator: G.W. Rubloff, University of Maryland

2:00pm **BM+MN+MS+TF+BI-ThA1 Advances towards Programmable Matter**, ***D. Erickson***, Cornell University **INVITED**

A dichotomy exists between the bottom-up self-assembly paradigm used to create regular structures at the nanoscale, and top-down approaches used to fabricate arbitrary structures serially at larger scales. The former of these enables rapid, highly parallel assembly but lacks critically important features of the latter such as the ability to arbitrarily direct the assembly location and perform error correction. We and our collaborators have recently proposed an alternative approach which combines these two based on dynamically programmable self-assembling materials, or ***programmable matter***. The uniqueness of our approach is that it uses dynamically-switchable affinities between assembling components facilitating the assembly of irregular structures. In this talk I present an overview of our approach and detail some of the analytical and experimental advances towards a programmable matter system we have recently made. These include: the development of a multi-chamber microfluidic chip for improved far-field assembly, the demonstration of near-field inter-tile affinity switching using a thermorheological assembly fluid and the ability to enhance assembly in three dimensions using unique fluid-structure interactions.

2:40pm **BM+MN+MS+TF+BI-ThA3 A Multilayered Microfluidic System with Buried Channels and Cell Compartmentalization for Engineering Heterogeneous Neural Networks**, *C. James, A. Greene, **A. Schiess**, G. Bachand,* Sandia National Laboratories*, M. Romero-Ortega*, University of Texas at Arlington

Current technology for engineering *in vitro* neural networks utilizes cell guidance cues that yield only temporary networks (< 1 month) as the cells rapidly diverge from their designed guidance cues during development of the culture. In addition, these engineered networks are typically comprised of homogeneous populations of neurons, thus the lack of multiple neuron types produces oversimplified networks that do not adequately represent in vivo networks. In addition, effective control over synaptic connections between different populations of neurons has not been demonstrated. Here, we describe a novel hybrid technology of multi-layered microfluidics with compartmentalized chambers containing multiple neuron types for engineering robust and complex neural networks with high resolution organization of synaptic connections. The device contains a first level of microfluidic channels etched 1-2 microns into the base glass substrate. These channels are fabricated with a novel process using a silicon nitride mask for hydrofluoric acid undercut etching to create buried microfluidic channels for robust containment and guidance of neurons. After the etching process, photoresist liftoff is performed to selectively adsorb poly-L-lysine (PLL) within the buried channels for improved neuron attachment and outgrowth at pre-defined locations. Polarity control of neurons is provided through a continuous set of guidance cues to promote axon development, while interrupted sets of guidance cues promote dendrite development. Current results show that axons and dendrites are positioned at predefined locations with a >65% accuracy. A second level of microfluidic channels and large (~mm) cell chambers are fabricated in polydimethylsiloxane (PDMS) from two-level SU-8 master molds. The base glass substrate and the PDMS substrate are aligned and bonded to create interconnects between channels in both substrates. These interconnects provide interaction regions for the development of synapses between growing neurites from cells in different chambers. We are currently applying this technology to engineer corticostriatal networks, an important region of the brain responsible for integrating multiple informational inputs crucial to complex decision-making in higher mammals. Specifically, we are using patch-clamp electrophysiology to track the development of synaptic memory in the form of long-term depression and potentiation (LTD/LTP) in these engineered networks.

3:00pm **BM+MN+MS+TF+BI-ThA4 Vesicle Production on a Microfluidic Platform using pH Sensitive Block Copolymers***, **L.E. Brown**,* The University of Sheffield, UK*, S.L. McArthur,* Swinburne University of Technology, Australia*, G. Battaglia, P.C. Wright*, The University of Sheffield, UK

The development of pH sensitive, biocompatible block copolymer vesicles has enabled the intracellular delivery of water soluble drugs and proteins. Improving the encapsulation efficiency of the vesicles is now a critical parameter. Transferring the production method to a microfluidic device creates the potential to vary the encapsulation conditions and improve this efficiency. In this work, a flow focussing microfluidic device is used. The self assembly of PMPC-b-PDPA block copolymer vesicles is induced within the device by changing the pH of the flows within the microchannels. The use of pH shift eliminates the need for organic solvents currently required for glass capillary production methods. This enables the biocompatibility of the block copolymers to be maintained, an essential factor for their application as molecular delivery vehicles.

The flow focussing microfluidic device was produced through standard soft lithography techniques. A three-channel flow system is used with the copolymer in solution at pH6 in the central channel and aqueous buffered solutions flowing in the channels either side. The laminar flow conditions within the microfluidic device result in a pH gradient at the interfaces where the three channels meet and where the block copolymers self-assemble into vesicles. These vesicle formation processes have been imaged using confocal microscopy via FRET with a block copolymer containing both rhodamine and fluorescein isothiocyanate groups. Dynamic light scattering and TEM were used to confirm vesicle formation.

With 50nm to 250nm vesicles continuously being produced within the device it was then possible to investigate whether higher encapsulation efficiencies can be achieved using the microfluidic device. The protein myoglobin was introduced through the central channel along with the copolymer. Spectrophotometric analysis indicated the overall the efficiency of the encapsulation process within the device is not a significant improvement on the standard bulk methods currently used, involving sonication of the vesicle solution containing the molecule to be encapsulated. Despite this, the continuous nature of microfluidic devices, as well as the lack of organic solvents being used in the production process indicates that the development of these devices offers a viable alternative production method for polymer vesicles that may enable the increases in encapsulation efficiency to be achieved. Work is ongoing to achieve this using the same pH shift mechanism within a glass capillary microfluidic device.

3:40pm **BM+MN+MS+TF+BI-ThA6 Integration of a Microfluidic Flow Cell Array with SPR Microscopy for In Situ Microarray Formation and Biomolecule Interaction Analysis***, J. Liu, M. Eddings,* University of Utah*, A. Miles*, Wasatch Microfluidics*, B. Gale, **J. Shumaker-Parry***, University of Utah

Analysis of biomolecule interactions based on surface plasmon resonance (SPR) microscopy provides a label-free approach to monitoring arrays of biomolecule interactions in real time. Typically the microarray sensing surface for these measurements is prepared ex situ and a single or few channel flow cell is used for the biomolecule interaction studies. The multiplexing nature then is derived from the microarray and the number of samples that can be run simultaneously is rather limited, diminishing the potential application for assays requiring a high-throughput approach due to a large number of samples. One example of this is the need to monitor for anti-drug antibodies from a large pool of patient samples during clinical trials of biotherapeutics. We demonstrate the capability of a multi-channel microfluidic flow cell array (MFCA) to expand the throughput capability when integrated with SPR microscopy. In addition, the MFCA provides an in situ approach to array fabrication that allows full characterization of the biomolecule immobilization process. We use the MFCA for delivery of sample solutions with continuous flow in 48 channels in parallel for rapid microarray creation and binding analysis while using SPR microscopy for real-time monitoring of these processes. Label-free measurement of antibody-antibody interactions demonstrates the capabilities of the integrated MFCA-SPR microscopy system and establishes the first steps of the development of a high-throughput, label-free immunogenicity assay. We demonstrate a limit of detection (LOD) of ~ 80 ng/ml for the particular antibody pair we studied. This LOD is ~6 times lower than the industry recommended immunogenicity assay detection limit. The high-throughput nature of the integrated system allows a large number of replicate experiments, including control experiments, to be performed simultaneously on the same sensor surface in a short time. The integrated system also will be applicable for more general high-throughput protein-array based analysis.

4:20pm **BM+MN+MS+TF+BI-ThA8 Nanochannel Stretching of Nucleic Acids: Towards Epigenetic Analysis**, *D.E. Streng, S.-F. Lim, A. Karpusenka, J. Pan, J.A. Hook, **R. Riehn***, NC State University

Nanochannels with a diameter of about 100nm2 are a novel method for stretching DNA for genomic investigations. Such devices are implemented through standard nanolithography in fused silica. The elongation of DNA results from an interplay of steric and entropic effects. Previous applications of nanochannel stretching included sizing, restriction mapping, and observation of transcription factor binding.

We show here that nanochannels can also be used to map the site-specific epigenetic state of DNA. In particular, we show here that the concept by nanoconfinement can be extended to chromatin, or DNA complexed to histones, and that the stretching is within the range expected from the de Gennes theory. We also demonstrate that the location-resolved cytidine methylation state of DNA can be mapped by specific fluorescent labeling. We will discuss the basic operation of these technique, and the application to artificial substrates with predefined epigenetic marks.

4:40pm **BM+MN+MS+TF+BI-ThA9 Microfluidic Models of Endothelial Cell Sprouting in Response to Biomechanical and Biochemical Microenvironments**, ***A.M. Shamloo***, *S.C. Heilshorn*, Stanford University

A novel microfluidic device was designed in order to generate stable, quantifiable concentration gradients of biomolecules in a cell culture chamber for 2-D and 3-D studies of shear-sensitive cell types such as endothelial cells. Endothelial cells form the inner lining of blood vessels and initiate a critical step in angiogenesis (the sprouting of new blood vessels) during wound healing and cancerous tumor growth. Therefore, a deeper understanding of the critical biomechanical and biochemical factors regulating endothelial cell sprouting can lead to improved clinical therapies for a multitude of diseases. Concentration distribution of soluble growth factors inside the microfluidic cell culture chamber was determined by simulation and experiment, and the stability of the gradient was verified over multiple hours. This device allows independent tuning of the matrix rigidity, the growth factor absolute concentration, and the growth factor concentration gradient steepness within a single experimental platform. Sprout formation of dermal microvascular endothelial cells was studied within collagen gels of varying density (0.3 - 2.7 mg/mL, corresponding to shear moduli of 8 – 800 Pa) that contained stable gradients of soluble vascular endothelial growth factor (VEGF). These experiments revealed that endothelial sprouting into multi-cellular, capillary-like structures is optimized at an intermediate collagen matrix density (G'~100 Pa). At lower matrix densities, cells were more likely to lose their coordinated motion and migrate as individual cells through the matrix; while at higher matrix densities, the cells formed broad cell clusters that rarely elongated into a sprout. Sprout thickness directly correlated with matrix rigidity, with thicker and less frequent sprouts present in gels with the highest shear moduli. Intriguingly, our 3D experiments also found that endothelial sprouts alter their sensitivity to VEGF depending on the matrix density, suggesting a complex interplay between biochemical and biomechanical factors. As matrix stiffness increases, steeper VEGF gradients and higher VEGF absolute concentrations are required to induce directional sprouting. In more compliant gels, endothelial sprouts that originally misaligned were able to turn and properly reorient parallel to the VEGF gradient; however, this turning phenomenon was only rarely observed in stiffer gels. These results demonstrate that matrix stiffness is an effective factor in stabilization and orientation of endothelial cells during sprouting and suggests new anti-angiogenic strategies for potential cancer treatment as well as pro-angiogenic strategies for regenerative medicine scaffolds.

5:00pm **BM+MN+MS+TF+BI-ThA10 Plasma Polymerisation of PDMS for Microfluidic Applications**, ***S. Forster***, *A.G. Pereira-Medrano, G. Battaglia, P.C. Wright,* University of Sheffield, UK*, S.L. McArthur*, Swinburne University of Technology, Australia

Polydimethylsiloxane (PDMS) has become the most popular material choice for a wide range of microfluidic bioengineering applications, including proteomics, protein separations and drug discovery and development. The reasons its popularity lie mainly in its highly advantageous fabrication requirements when compared to traditional materials such as glass and silicon. However, PDMS has some fundamental drawbacks, namely a lack of functionality present at the surface, high protein fouling and an inability to retain stable surface modification due to its motile hydrophobic monomer. These factors can lead to the loss of specificity and sensitivity in many bioassays. Due to this reason much work has been completed looking into surface modification of PDMS for such applications. Here an alternative method of stable surface modification of PDMS for many microfluidic applications through enhanced curing conditions and plasma polymerisation is shown. Stable and functional surface coatings have been achieved on bulk PDMS and within microfluidic channels. Bulk surfaces were characterised using a combination of XPS and ToF-SIMS, while coated micro-channels were tested using confocal microscopy and various assays. This methodology has been used in many applications and one area where it has proven extremely useful is in microfluidic proteomics where surface properties are of paramount importance due to the inherently small volumes and quantities associated with biological samples. Firstly, plasma polymer coated PDMS micro-channels were utilised for on-chip IEF protein separations (i.e. separating proteins bases on charge) and showed reduced electrosmotic flow (EOF) and protein adsorption within the device. Secondly, a µIMER (micro-immobilised enzyme reactor) was produced using plasma polymer coated PDMS devices. The µIMER was then used in 'shotgun' protein digestion applications in conjunction with Mass Spectrometry where it was shown to have numerous advantages over untreated PDMS devices, as well as comparing favorably to published work on other µIMER systems. The device was used to digest single and multiple protein samples as well as complex membrane protein samples. Finally, successful covalent bonding of plasma polymer coated devices has led to the completion of polymer vesicle immobilisation within a microfluidic channel. Initial work looking at the immobilisation of polymer vesicles with an encapsulated digestive enzyme has shown to increase proteomic digestion efficiency. This success opens up the possibility of translating this technique into many potential microfluidic applications through the extensive versatility of encapsulation within polymer vesicles.

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.

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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 275ºC 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.

Plasma Science and Technology
Room: A1 - Session PS1-ThA

Fundamentals of Plasma-Surface Interactions II
Moderator: D.J. Economou, University of Houston

2:00pm **PS1-ThA1 Negative Ion Surface Production in Low Pressure Plasma**, ***G. Cartry***, *L. Schiesko, J.M. Layet, M. Carrere*, PIIM, Aix - Marseille Université - CNRS, France **INVITED**

Negative ions in plasmas play a main role in the discharge kinetics. For instance they may be at the origin of plasma instability [1], or may be responsible for coalescence in the primary state of dust formation [2]. Plasma based negative ion sources can have many applications. They may be used to reduce surface charge during plasma etching and in the context of controlled fusion research, they are used to generate neutral beams to heat fusion plasma. Therefore controlling negative ion production and loss in plasmas is of primary interest in many research fields. Efficient negative ion sources use caesium deposited on surfaces to increase negative ion production. Indeed, due to its property of reducing the work function, caesium leads to a high negative ion surface production yield. Negative ions are also produced in plasma bulk through electron attachment on molecules. Up to now most of works focused on plasma bulk production and on caesiated-surface production. Few works deal with caesium free surface production while almost all low pressure plasma sources are running without caesium. The aim of the present paper is to study caesium free negative ion surface production in low pressure plasmas.

Our first study is concerned with H2/D2 plasmas and graphite material for fusion applications [3]. However, results obtained here can be extended to low pressure plasma sources used in microelectronic industry for instance, since carbon containing materials are often in interaction with H2 plasmas or even more electronegative plasmas such as oxygen, fluorine or chlorine containing plasmas.

We use a helicon reactor whether in capacitive or inductive mode. A mass spectrometer is placed in the diffusion chamber of the helicon reactor and faces a one square centimetre graphite sample. The sample is negatively biased with respect to the plasma. Positive ions (H+, H2+, H3+) bombards it and negative ions formed (H-) upon bombardment are repelled from the surface toward the plasma. Under low pressure considered here, they reach without any collision the mass spectrometer where they are analysed according to their energy. Study of negatve Ion Distribution Function (IDF) provides information on surface production mechanisms. In this talk we will discuss IDFs measurements, describe how we identify surface production mechanisms, show negative ion surface production yield dependency with positive ion flux and energy, and compare H2 and D2 plasmas.

The authors acknowledge ANR (project ITER-NIS BLAN08-2_310122)

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2:40pm **PS1-ThA3 Amorphous Hydrogenated Carbon Etching with a Low Energetic Plasma Jet**, ***T.A.R. Hansen***, *J.W. Weber, M.C.M. van de Sanden, R. Engeln*, Eindhoven University of Technology, The Netherlands

Structures in the chip industry are approaching the 32 nm half pitch, which requires radiation in the VUV and EUV range. Cracking of hydrocarbon impurities in the vacuum by the radiation causes C growth on the VUV and EUV optics. Each nm of deposited carbon reduces the reflectivity of the optics by 1%. Fast removal of these contamination layers without damage to the underlying optics is essential for the next generation of lithography devices.

Etching with a low energetic plasma jet can be used to selectively remove coatings such as hydrogenated amorphous carbon (*a*-C:H) without damage to the underlying structure. Real time, *in situ* spectroscopic ellipsometry measurements indicate that the highest etch rates are obtained for an Ar/H_2 plasma, rather than for a pure Ar or H_2 plasma.

Even though the etch rate of *a*-C:H thin films is dependent on both temperature and roughness, the highest roughness in absolute values is attained by the plasma with the lowest etch rate. At low temperatures, the etch rate deviates from an Arrhenius relation, while the activation energy is similar for both the H_2 and Ar/H_2 plasma at higher temperatures.

The two orders of magnitude higher etch rate for the Ar/H_2 plasma is due to chemical sputtering, which is a synergistic effect between atomic H and Ar^+ ions with an ion energy below the threshold of 58 eV for physical sputtering. Chemical sputtering has been observed by Hopf et al. for energies above 20 eV and an H to Ar^+ flux ratio over 100 [1]. In our plasma, however, the Ar^+ ion energy is only a few eV's and the estimated H to Ar^+ ratio is lower than 5.

The etch products, released from the surface, consist mainly of CH_4 and C_2H_y, as shown by residual gas analysis. Time resolved optical emission spectra of the Ar/H_2 plasma, from a few mm's in front of an *a*-C:H sample, indicates also the presence of C_2 and CH radicals. The CH radical is formed in the plasma phase through charge transfer between Ar^+ ions and these larger hydrocarbons, and dissociative recombination. Similar plasma chemical processes occur during the remote plasma deposition of *a*-C:H films. However, in contrast with deposition, the CH rotational temperature shows an overpopulation in the higher excited states, indicating that the (internal state of the) parent molecule is different for an etch plasma than for a deposition plasma.

Spatially resolved optical emission measurements are Abel inverted, by means of the numerical Barr method. While there is some CH production throughout the entire plasma jet, the highest CH production occurs in front of the *a*-C:H sample.

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3:00pm **PS1-ThA4 Investigation of Fluorocarbon PECVD During Processing of Si and ZrO_2 Surfaces**, ***M. Cuddy***, *E.R. Fisher*, Colorado State University

Films deposited from fluorocarbon (FC) plasmas exhibit low dielectric constants desirable for interlayers in ultra-large scale integrated circuits (ULSIs). The processing of ULSIs has involved the use of small monomer (CF_4, C_2F_6) FC precursors as an avenue for plasma-enhanced chemical vapor deposition (PECVD). To gain a broader understanding of both the FC plasma system and plasma-surface interactions, we have explored gas phase diagnostics and species-surface reactivity under varying plasma parameters. This presentation will reflect upon data obtained from optical emission spectroscopy (OES) concerning the role of excited state species present in FC plasmas. OES data show that during FC plasma treatment of Si and ZrO_2 wafers, CF_2* concentrations increase independent of feed gas and substrate type. The films deposited from such treatments do, indeed, consist of FC moieties and thus plasma-surface interactions are clearly influential in the overall process. We have studied the interaction of FC plasma species at the interface of depositing films using the imaging of radicals interacting with surfaces (IRIS) technique. IRIS data show that scatter probabilities for the CF_2 radical are greater than unity, indicating that CF_2 is produced from films *at the surface* during FC plasma processing of silicon. Furthermore, we have used quadrupole mass spectrometry to investigate mean ion energies of CF_2^+ in FC plasmas and have discovered that ion energies increase with increasing applied rf power. We have previously demonstrated that IRIS scatter coefficients for CF_2 produced from larger precursors (C_3F_8 and C_4F_8) correlate directly with ion energy. Thus, we will explore the role of this radical during processing of Si and ZrO_2 with small FC precursors as monitored by IRIS studies and compare these results with the respective ion energies for CF_2^+ in these systems.

3:40pm **PS1-ThA6 Studies of Chlorine-Oxygen Plasmas and Evidence for Heterogeneous Formation of ClO and ClO_2**, ***V.M. Donnelly***, *J. Guha*, University of Houston

Plasma and surface diagnostics of Cl_2/O_2 mixed-gas inductively coupled plasmas are reported. Using trace rare gas optical emission spectroscopy (TRG-OES) and Langmuir probe analysis, electron temperatures (T_e) and number densities for Cl atoms (n_{Cl}), electrons (n_e), and positive ions were measured as a function of percent O_2 in the feed gas and position in the plasma chamber. Adsorbates on, and products desorbing from a rotating anodized aluminum substrate exposed to the plasma were detected with an Auger electron spectrometer and a quadrupole mass spectrometer. T_e and n_e increased with increasing percent O_2 in the plasma, while n_{Cl} fell off with O_2 addition in a manner reflecting simple dilution. Cl atom recombination probabilities (γ_{Cl}) were measured and were found to be a nearly constant 0.036±0.007 over the range of Cl_2/O_2 mixing ratios and Cl coverage. Large yields of ClO and ClO_2 were found to desorb from the surface during exposure to the plasma, ascribed predominantly to Langmuir-Hinshelwood reactions between adsorbed O and Cl. In addition, the transient surface composition of an anodized aluminum surface was determined as the gas was switched from Cl_2 to O_2 and vice versa. When the surface was first conditioned in an O_2 plasma and then exposed to Cl_2 plasmas, a rapid uptake of Cl was found in the first tens of seconds, followed by a slow approach to a steady state value within ~5 minutes of plasma exposure. Conversely, when the surface was exposed to a Cl_2 plasma for a long time and then switched to an O_2 plasma, the anodized aluminum surface underwent a rapid de-chlorination in the first few seconds and then a slow approach to steady state over ~3 minutes. The buildup and decay of Cl coverage is well described by a stretched exponential function, reflecting a range of binding sites for Cl. Throughout these treatments, the coverages of Si (from erosion of the quartz discharge tube) and O was nearly constant.

4:00pm **PS1-ThA7 Etching of Silicon and Silicon Oxide in a Pulsed Inductively Coupled Plasma with Chlorine**, ***C. Petit-Etienne***, LTM/UJF, France*, L. Vallier, E. Pargon, O. Joubert*, LTM/CNRS, France

For the next technological generations of integrated circuits, the traditional challenges faced by etch plasmas (profile control, selectivity, critical dimensions, uniformity, defects, ...) become more and more difficult, intensified by the use of new materials, the limitations of lithography, and the recent introduction of new device structures and integration schemes. Chemical plasma composition can be changed by modifying the gas mixture, ion flux can be partly controlled by source power, and ion energy can be chosen thanks to the bias voltage applied to the substrate. However, these control parameters are not always sufficient to reach all required etching characteristics and new control parameters are needed. Pulsing the plasma source power or the substrate bias offers new operating parameters (pulse frequency, duty cycle). The main advantages of a pulsed etching process are the improvement of etch selectivity and the reduction of charge-up damages and defects by reducing the electron activity and controlling the dissociation of radicals in the plasma.

Studies are being conducted on the etching characteristics of silicon and silicon dioxide in a 300 mm industrial inductively coupled plasma etching chamber having pulsed plasma discharge capability from Applied Materials. The reactor has been modified to be connected to an Angle-Resolved X Ray Photoelectron spectroscopy analyzer by a robotized vacuum chamber. Hence after an etching process, XPS spectra were recorded as function of take-off angle and the integrated intensities of the core-level peaks were used to obtain chlorine concentration and chemical state information from different depths of the sample, thereby permitting non-destructive characterization of chlorine profile in thin silicon oxide films. Material etch rates were measured in real time by in situ multi-wavelength ellipsometry.

When the plasma is pulsed, two parameters can be adjusted, namely the frequency of the pulse and the duty cycle. While the frequency has only a small influence on the etch rates in the investigated frequency range, our results demonstrate that a low duty cycle clearly modifies etch rate and can considerably improve the etch selectivity between silicon and silicon oxide. When a thin silicon gate oxide layer is exposed to very low energy etching conditions, a first step of chlorine incorporation is observed before etching. Preferential accumulation near the SiO_2/Si interface is observed and chlorine is shown to bond to both silicon and oxygen in multiple distinct chemical states.

4:20pm **PS1-ThA8 Fully Atomistic Profile Evolution Simulation of Nanometer-scale Si Trench Etching by Energetic F, Cl, and Br Beams**, ***H. Tsuda****, T. Nagaoka, K. Eriguchi, K. Ono,* Kyoto University, Japan*, H. Ohta*, University of California, Santa Barbara

An atomic-scale understanding of interactions between chemically reactive plasmas and surfaces is required to establish nanometer-scale processing technologies. Various numerical studies based on molecular dynamics (MD) simulation have been reported so far, but these were limited to simulations of the simple blanket etching to estimate microscopic etching properties [1,2]. Here, we first report a fully atomistic silicon feature profile simulation using classical MD simulations. The potential form can be found in our previous papers [2,3,4]. F, Cl, and Br beams with a translational energy of 100 eV were used as reactive species. The surface area of Si (100) substrates was about 163×22 Å^2, where 3840 silicon atoms were initially located in the structure of diamond lattice. M ask patterns were introduced in the direction parallel to the short axis with periodic condition, in order to reproduce the trench etching feature. Then, the area without mask was 50×22 Å^2. By using our new atomistic profile evolution simulation, we investigated halogen plasma-surface interactions at sidewalls and bottom surfaces of nanometer-scale Si trench in detail. It was found that specific feature profiles with different gaseous species appear not only at the sub-micrometer-scale but also at the nanometer-scale etching, and the difference of surface reaction layer formation strongly affects the feature profile evolution during etching. For instance, fluorine beam etching showed that fluoride layer is formed on the entire surfaces containing sidewalls and bottom surfaces, thus giving isotropic etching. Chloride layer was thicker than fluoride and bromide layers, to give feature profiles of sidewall tapering. Bromide layer on bottom surfaces was thinnest among the three, and so the etching rate was lowest. So, it was cleared that the surface reaction layer formation strongly affects the feature profile evolution during etching. Our approach is essential as a reference for macroscopic or empirical profile simulation, where simulation sizes have been reduced recently. We also show some comparison between MD-based profile simulation and our empirical profile simulation (atomic-scale cellular model [5]).

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4:40pm **PS1-ThA9 Molecular Dynamics Simulations of Oxygen-Containing Polymer Sputtering and the Ohnishi Parameter**, ***G.K. Choudhary****, J.J. Végh, D.B. Graves*, University of California, Berkeley

The effects of ion bombardment on polymer surfaces can be profound, with implications for all plasma-based pattern transfer processes that involve the use of polymer etch masks in lithography and etching. It is known that Ar$^+$ bombardment of various polymers results in the formation of a 1-2 nm deep cross-linked region at the exposed surface, and that virgin polymer sputtering yields can be several orders of magnitude higher than steady state yields after ion bombardment. [1]

In this talk, we report results from molecular dynamics (MD) simulations of Ar+ (~ 40 - 150 eV) sputtering of oxygen-containing polymers. The MD data are compared to available experimental results, with special focus on the so-called Ohnishi parameter, which has been shown to correlate with sputtering yields for many O-containing polymers. [2] The MD simulations match the published correlations well, and we present a quantitative model of sputtering for these polymers that shows why the Ohnishi parameter (a function of the polymer composition) is proportional to the steady state sputtering yield.

However, we also show that the Ohnishi parameter does not correlate with yields for other polymers, including polyfluoroethylene and polyethylene. The MD simulations show that the validity of this parameterization is dependent on whether or not the sputtering of the polymer transitions between ion-induced scissioning to cross-linking at steady-state.

Finally, we discuss the implications of the dynamics of ion-induced surface cross-linking for synergistic photoresist roughening that occurs in plasmas, especially in the presence of vacuum ultraviolet photon and/or beaming electron exposure.

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5:00pm **PS1-ThA10 Charge Trapping and Valence-band Structure of VUV-Irradiated BEOL Dielectrics**, ***J.L. Lauer****, J.L. Shohet,* University of Wisconsin-Madison*, Y. Nishi,* Stanford University*, A. Antonelli*, Novellus Corporatiion

The minimum spacing between conductive lines in advanced integrated circuits (ICs) continues to decrease with each generation of technology. As a result, the long-term reliability of ICs is becoming increasingly dependent on the reliability of the intermetal dielectrics which often become damaged during back-end-of-the-line (BEOL) processing. Dielectrics used in BEOL structures are often irradiated with photons of various energies during plasma processing, charge annealing, and curing of porous materials. In particular, processing plasmas produce significant amounts of vacuum ultraviolet (VUV) radiation which are, among other processes, capable of creating electron-hole pairs within dielectrics. As a result, VUV radiation has an impact on the electrical conductivity of dielectrics during plasma processing which can either contribute to or mitigate trapped charge within dielectrics. We compare the charging response of 50, 250, and 450 nm of SiOCH, SiN, SiCO, SiCN, and SiC dielectrics on Si substrates after irradiation to vacuum ultraviolet (VUV) radiation. We choose to irradiate the dielectric layers to a photon energy of 9.5 eV because photons with this energy are often emitted from processing plasmas that contain oxygen, i.e. ashing and etching plasmas. The charging response of the dielectrics was evaluated by measuring the surface potential on the dielectrics with a Kelvin probe after irradiation with several doses of 9.5 eV photons. The surface potential on all of the dielectrics after VUV irradiation was positive due to the accumulation of positive charge by traps located within the dielectrics. By comparing the surface potential on several thicknesses of dielectrics after VUV irradiation we can estimate the location within the dielectric the charge is trapped. The surface potential on SiOCH layers of varying k-values after VUV irradiation indicates the presence of both negative- and positive-charged traps. From VUV-spectroscopy, we determined the SiOCH layers have electron traps located 0.8 eV below the conduction-band edge and hole traps located 1.4 eV above the valence-band edge.

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Thursday Afternoon Poster Sessions

MEMS and NEMS
Room: Hall 3 - Session MN-ThP

MEMS and NEMS Poster Session

MN-ThP1 Imprinting of Guide Structures to Weave Nylon Fibers, ***H. Mekaru***, *M. Takahashi*, AIST, Japan

We are developing a large-area display and a wearable health checker by weaving fibers with an electrical circuit on their surface. In this technique, a guide structure that determines the position to fix fibers was processed on the fiber by a thermal nanoimprint technology. We used two kinds of molds with guide structures with different cross-sectional shapes (rectangle and arc). Micropoles to connect the fiber were arranged in the bottom side of the guide structures. In the case of guide structure with a rectangular pattern, the multilayer structure was formed on a Si substrate using MEMS technology; and Ni mold was made by electroforming. Fifteen convex rectangles with their length, width, and height as 14.4 mm, 100 µm and 50 µm were arranged in a 1-mm pitch formation. And, 10-µm deep column holes with the diameters of 5, 10, and 20 µm, were fabricated on the upper side of the guide structure. The other kind of guide structure with an arc pattern was processed by precision machining. A 150-µm-thick Ni-P layer was electroless-plated on an Inconel-600 alloy substrate and the layer was then coarse-processed by dicing. The finish processing employed a 20-µm-diameter diamond endmill and a Robonano α-0iB (FANUC Ltd.). Thirteen arc guide structures with 20-µm-diameter holes were processed for 4 hrs. The length, bottom-width, and height of the individual guide structure were 15 mm, 160 µm, and 100 µm. Hemispherical holes with 20 µm diameter and a maximum depth of 10 µm were processed on the upper side of the guide structure. The size of each type of mold was the same 20 mm square. The guide structures from the two kinds of molds were imprinted on a 90-µm-diameter nylon fiber (Amilan, Toray Industries, Inc). In the imprinting experiments, a desktop thermal nanoimprint system NI-273 (Nano Craft Technologies Co.) was used. Molding conditions were: heating temperatures = 100 °C, cooling temperature = 70 °C, loading force = 100 N, and holding time = 1 s. The guide structure and micropoles were successfully transcribed from the mold onto the nylon fiber. The side-view of the guide structure was examined with an optical microscope and the pressed depth was measured as 21 µm, regardless of the kind of mold used. After the imprinting, the weaving of the fiber was carried out with tweezers under an optical microscope. Each guide was confirmed to be connected to each fiber. It was easy to weave the arc guide structure processed with machining because the guide sidewall was curved. In future, weaving of fiber with variety of electric circuit patterns will be presented.

MN-ThP2 Test Instrument for the Tensile Strength of Micro-Nano Materials, ***A. Kasahara***, *H. Suzuki, M. Goto, H. Araki, P. Yuriy, M. Tosa*, NIMS, Japan

There is considerable research at present on the performance and properties of nanosheets, nanofibers and other functional nanomaterials such as fullerenes and nanotubes. This is particularly true of carbon nanotube, made from carbon atoms, where many research projects throughout the world are looking at measurement techniques for evaluating electrical and electronic characteristics with a view to developing electronic device applications such as high-intensity field-emitted electron sources and ultra-fast transistors. However, we have not yet to see a genuine, flexible methodology for evaluating the key characteristic of mechanical strength essential to micro-nano structural materials development — the nanoscale equivalent of tensile strength testers for ordinary materials. This is due to the inherent difficulties associated with the manipulation and transportation of materials at the micro-nano scale level. Here, we will discuss our recent results on mechanical strength measurement of micro-nano wires in diameter several nm through several thousand nm and in length several mm by means of prepared micro-nano tensile strength tester device.

MN-ThP3 Morphology and Mechanical Properties of Block Copolymer Films for Bone Regeneration Applications, *B. Bhushan*, ***M. Palacio***, *S. Schricker*, The Ohio State University

Biocompatible polymers act as scaffolds for the regeneration and growth of bones. In dentistry, these can be used to treat diseases with accompanying bone loss, such as aggressive periodontitis. Surface morphology, specifically the presence of nanostructures, is expected to affect the adhesion of the cells adsorbed on the surface, which should be optimized for successful cell growth. Block copolymers are of interest as scaffold materials because a number of them are biocompatible, and their nanostructure is easily tunable with synthetic techniques. In this investigation, atomic force microscopy (AFM) studies were conducted for two block copolymers, namely, poly(methyl methacrylate-*b*-acrylic acid) and poly(methyl methacrylate-*b*-hydroxyethyl methacrylate). The topography, stiffness, phase angle, and friction maps were obtained in dry and aqueous environments in order to study the morphology, elasticity, viscoelasticity, and friction properties, respectively. Results of AFM imaging identified the presence of polymer domains corresponding to the copolymer components. Images taken in an aqueous medium reveal greater contrast as a consequence of the differential water absorption between the copolymer components.

MN-ThP4 Mechanically Durable Superhydrophobic, Self-Cleaning, and Low-Drag Surfaces with Hierarchical Structure, ***Y.C. Jung***, *B. Bhushan*, The Ohio State University

Superhydrophobic surfaces exhibit extreme water-repellent properties. These surfaces with high contact angle and low contact angle hysteresis also exhibit a self-cleaning effect and low drag for fluid flow[1-4]. For the development of superhydrophobic surfaces, which is important for various applications such as glass windows and solar panels, alternative materials and fabrication methods need to be explored to improve durability[5]. It is necessary to perform durability studies on these surfaces in order to identify fabrication techniques and materials that can best withstand real world applications. Micro-, nano-, and hierarchical structures which would lead to superhydrophobicity and self-cleaning are prepared using different fabrication methods. In order to compare the durability of the various fabricated surfaces, friction and wear studies are performed at the microscale using a pin-on-disk test, where a stationary pin applies a constant normal load while sliding on the sample surface. Waterfall and waterjet tests are also conducted to determine the loss of superhydrophobicity by changing the flow volume and pressure conditions, respectively. The changes in the surface morphology and structure and the wettability are examined by SEM and AFM imaging and contact angle measurements, respectively .

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MN-ThP5 Tripod Honeycomb Shape Scaffold for Retina Cell Culture by Dynamic Mode Multidirectional UV Lithography, ***J. Kim***, *J. Yang, M.M. Slaughter, G. Kim, Y.-K. Yoon*, University at Buffalo, the State University of New York

Since the cytoskeletal properties of the cells cultured in two-dimensional (2D) culturing environment are different from those of the real biological cells constituting three-dimensional (3D) organs, the *in-vitro* cytokinetics from a 2D scaffold cannot be applied to *in-vivo* 3D cell culturing study. In this research, a 3D tripod honeycomb shape scaffold array fabricated using automated dynamic mode multidirectional ultra violet (UV) lithography [1] has been demonstrated for efficient 3D cell culture. A unit element of the scaffold consists of honeycomb shape confinement on the top, three posts at the bottom, and tapered openings in the side walls. **This architecture provides advantageous properties for 3D cell culture: (1) sufficient nutrient supply paths through the openings in the side walls, (2) mechanically stable tripod structure, (3) moldable 3D geometry useful for mass production with various material selection, and (4) structural flexibility amenable to further 3D macro shaping.** SU-8 (negative tone photoresist) mold masters made by multidirectional lithography, replicas with biodegradable polymer (poly lactic-co-glycolic acid: PLGA) after micromolding, and macroscopically deformed scaffolds are successfully demonstrated. As a test vehicle, retinal cells [2] are successfully cultured on the fabricated PLGA scaffold.

Polydimethylsiloxane (PDMS) has been used to make a negative form of the mold master. A broad range of materials can be used for the final polymeric structure. In this research, PLGA has been cast to form a final scaffold.

The structural compliance associated with the tapered sidewall provides macroscopic flexibility, one of the unique merits of this architecture. A rounded scaffold for the potential usage of artificial blood vessels or other implant devices is demonstrated. A unit scaffold layer has a height of 300mm and multilayer scaffolds can be implemented for much thicker 3D cell culturing by stacking multiple layers.

[1] J.K. Kim et al, “Automated dynamic mode multidirectional UV lithography for complex 3-D microstructures,” Proceedings of IEEE Micro Electro Mechanical Systems, Jan. 13-17, 2008, Tucson, AZ, pp. 399 – 402.

[2] X Luo et al, “Susceptibilities to and Mechanisms of Excitotoxic Cell Death of Adult Mouse Inner Retinal Neurons in Dissociated Culture,” Invest Ophthalmol Vis Sci., 45 (2004), pp. 4576–4582.

MN-ThP6 Micro Accelerometer with Mechanically Nonlinear Self-Limited Bistable Suspension, ***E.M. Amir***, *S.L. Krylov*, Tel Aviv University, Israel

We report on operational principle, modeling and design of an electrostatically actuated accelerometric device with mechanically nonlinear suspension element. The device incorporates a proof mass actuated by a parallel-plate electrode and attached to a substrate by initially curved beams in such a way that both electrostatic and inertial forces are directed along the beam. In accordance with the exact extensible *elastica* and approximate reduced order models of the beam used for the analysis, the deformation of an initially curved slender beam subjected to an end force can be subdivided into two stages - the "bending" stage associated mainly with the straightening of the beam and the "tension" stage corresponding to elongation of the almost straight beam. Since the stiffness of the beam at the first stage is significantly lower than at the second stage, the force–displacement dependence of this kind of suspension is of self-limiting type and the beam can be viewed, in a sense, as one directional constraint. Application of nonlinear electrostatic force results in electrostatic (pull-in) instability followed by the steep increase in the straightened beam stiffness preventing contact with the electrode and resulting in appearance of an additional stable configuration and bistability of the beam.

In this research we present two operational principles for measuring the acceleration - the pull-in voltage monitoring and the resonance frequency shift monitoring. The pull-in voltage approach is based on the (found to be close to linear) dependence between the pull-in voltage and the acceleration, while the self-limiting characteristic of the suspension prevents undesirable from the reliability point of view contact between the proof mass and the actuation electrode. The resonance frequency approach is based on the monitoring of the resonant frequency shift appearing due to acceleration and significantly enhanced in the vicinity of the pull-in instability points. Model results show that using suggested approach significant improvement, comparing to conventional designs with linear flexures, in the device performance could be achieved and μg resolution combined with extended dynamic range are feasible for relatively simple architecture and well established silicon on insulator (SOI) based fabrication process.

MN-ThP7 Creation of Co-planar Oxide Pillars for Fabricating Overhanging Metal Structures, ***S.A. Hickman***, *E.C. VanWerven, J.C. Ong, J.A. Marohn*, Cornell University

Magnetic resonance force microscopy (MRFM) combines the nanoscale resolution of scanned probe microscopy with the three-dimensional, isotopically specific imaging capabilities of magnetic resonance imaging. The ultimate goal of MRFM is to achieve single-proton imaging resolution and create an atomic-resolution three-dimensional image of a individual molecule. At this level, the technique could achieve such feats as the structural determination of a single copy of a protein or macromolecular complex, making it a fantastic tool for biological study.

The key technology for MRFM is extremely sensitive, magnet-tipped cantilevers. While extensive effort has gone into fabricating such cantilevers, thermally-limited cantilever sensitivity is seldom achieved in practice because of surface-induced dissipation. The design of our cantilever minimizes this noise by extending the magnet past the cantilever tip. In our current cantilever fabrication scheme, we deposit the magnet on the device layer of a silicon-on-insulator wafer, and then create an overhanging magnet by using an isotropic sulfur hexafluoride etch to partially remove the silicon under the magnet. While successful, this process raises concerns over possible damage to the magnet from the etch species, and there is some degree of variability in the length of the overhang because of the very high silicon etch rate. To mitigate these issues, we have developed an alternative process in which the magnet is deposited over a pillar of silicon oxide extending through the device silicon layer and conformal with the top of this layer. This innovation removes the plasma etching step of our previous approach, and the length of overhang can be controlled by changing the lithographic placement of the magnet relative to the edge of this pillar. The pillar is created by localized oxidation of the device silicon, followed by chemical mechanical planarization. In this poster we will present our progress on this work, as well as present ideas for uses of our innovation well beyond cantilevers for MRFM.

MN-ThP9 Fabrication of a Vibratory Gyroscope Based on Piezoelectric Actuators and Sensors using MEMS Technology, ***V. Rincon***, *H. Nampoori, A.L. Highsmith, S. Kotru*, University of Alabama

Piezoelectric materials are commonly used in micro electro mechanical systems (MEMS) due to the self-generating sensing, large actuation amplitude with low voltage, and compatibility to integrated circuit process. In this work we have used Nb-doped $Pb(Zr_{20},Ti_{80})O_3$ (PZT) films for fabricating actuators and sensors for a micromachined vibratory gyroscope. PZT films exhibit higher values of effective transverse piezoelectric coefficient ($e_{31,f}$) and effective longitudinal piezoelectric coefficient ($d_{33,f}$) compared to any other available piezoelectric materials.

The complete gyroscope device consists of one active wafer, two handler wafers and a Si post bonded together. The active and handle wafers were fabricated using MEMS technology. Entire processing was done in a clean room environment using the state of art micro fabrication facility (MFF) at the University of Alabama . First, PNZT was deposited on both side of the active wafer resulting in $PNZT/Pt/TiO_2/SiO_2/Si/SiO_2/TiO_2$ /Pt/PNZT stack. This step was followed by $SiO_2/Ti/Au$ deposition using an e-beam evaporator. Processing the active wafer involves five masks and twenty four photolithography steps. Sine the device is on both sides of the wafers, one side was always protected with photoresist while the other side of the wafer was being processed. The final structures were released by etching away various film layers using a combination of dry and wet etching techniques. This includes three different etching techniques viz., ion mill, oxide etcher, and wet etch.

For the handle wafers Cr films were deposited on Si wafers using an e-beam evaporator. A photo-defineable polyimide was then spun on these wafers and patterned. These patterns were transferred using two masks and two photolithography steps. The active and handle wafers were then bonded together and the Si post attached to it to form the complete device. Details of the fabrication process of the device and its evaluation will be presented.

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 non-uniformly. 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 (ρ) 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 three-dimensional 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 ($E<E_{Si}$) hafnia and aluminum nitride films decreased the natural frequency. From the measured spectral response, material properties were extracted using simple expressions for E and ρ 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 ρ 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 frame-type 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 cantilever-type 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 atomic-scale, 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 thin-film sample, we have taken up the challenge of fabricating attonewton-sensitivity 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 ~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 ~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_2 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_2 etching of pure Silicon, pure Molybdenum, and Molybdenum deposited on Silicon oxide and Al substrates, in pressures consistent with industrial conditions (~ 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 bulk-controlled 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_6/O_2 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.

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