MEMS and NEMS
Room: 10 - Session MN+AS-MoM

Characterization of Surfaces and Interfaces in MEMS and NEMS

Moderator: A.V. Sumant, Argonne National Laboratory

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

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

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

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

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

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

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

We demonstrate a fabrication of UNCD nanowire based switch with a movable source anchored at both ends. An immobile drain electrode is separated from the center of the source beam by a narrow gap. Two electrically connected gate electrodes are separated from the source by the gate gap, which is larger than the drain gap [3]. A UNCD layer was deposited on top of a sacrificial SiO2 layer and covered with a SiO2 layer that served as a hard mask for the RIE process. The UNCD layer represents the mechanical layer of the switch, the switch contacts and the gate electrodes. We aim to fabricate a reliable switch with fast switching times and low actuation voltages.

References:

[1] Sumant *et al.* MRS Bulletin, 35, 281 (2010)

[2] Wang *et al.* Nanotechnology, 23, 075301 (2012)

[3] Czaplewski *et al.* Electronics Letters 45(11): 550 (2009)

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

We discuss a materials breakthrough for MEMS. In contrast with conventional electromechanical devices, whose constituents are chosen from a vast range materials and alloys to optimize fabrication, performance and cost, MEMS have largely been made using the same materials and methods as those used in the silicon-based microelectronics industry. In order to make MEMS out of a much richer suite of materials, including metals, semiconductors, and ceramics, we have developed a process termed carbon nanotube templated microfabrication (CNT-M). In CNT-M we employ patterned, vertically aligned carbon nanotube forests as a three-dimensional microfabrication scaffold to create precise high-aspect-ratio (up to 200:1) microstructures. The "as grown" CNT forests are very low density (at 0.009 g/cc the forest is ~1% car bon and 99% air) and not useful as mechanical materials because they are extremely fragile, due to their low density and weak intratube bonding. However, when we replace the air spaces between tubes in the forest with a filler material by atomistic deposition, the infiltrated CNT framework becomes a robust microstructure consisting mostly of the filler material. Thus, by patterning the CNT microstructure and limiting the deposition of the filler material, CNT-M gives us control over structural features on both the nano and microscales (nanoscale porosity and microscale structure). We have used chemical vapor deposition to infiltrate the CNT framework with semiconductors (Si) and ceramics (SiO_2 , SiN_x, and nanocrystalline carbon) for applications in microactuation, sensing, and chemical separations. But many potential MEMS applications would benefit from structures fabricated from functional metals. We now report on the fabrication of metal microstructures using the CNT-M process. We demonstrate the versatility of this fabrication approach by demonstrating both chemical vapor infiltration (making tungsten and molybdenum structures) and electrodeposition (making nickel structures) based metal CNT-M processes. These metals provide several desirable materials properties to high aspect ratio MEMS applications including high electrical and thermal conductivity, high melting temperatures, resistance to corrosion, low thermal expansion, high Young's modulus, hardness and yield strength. Electrical, mechanical, and structural characterization of the microfabricated metal structures will also be presented.

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

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

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

Use of the Center for Nanoscale Materials at Argonne National Lab was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

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

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

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

We have proposed two methods that can help to reduce this defect. The first consists in adding a low oxygen flow in the etch cycle, favouring a low additional passivation. The second technique consists in gradually increasing the SF_6 flow, in the etching steps, during the first minutes of the recipe. Consequently, the process starts with a low etch rate and a more efficient passivation, which helps to limit the extended scalloping. These two techniques efficiently reduce the defects but the profiles tend to be always positive. It seems impossible to get at the same time vertical sidewalls and low defects.

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

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

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

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

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

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

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

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

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

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

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

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

MEMS and NEMS
Room: 10 - Session MN-MoA

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

Moderator: M. Metzler, Cornell University

2:00pm **MN-MoA1 Acute Stress in Silicon Nitride**, ***J.M. Parpia***, *V.P. Adiga, B. Ilic, R.A. Barton, R. De Alba,* Cornell University, *I. Wilson-Rae,* Technische Universität München, Germany, *H.G. Craighead*, Cornell University **INVITED**

Mechanical structures fabricated from highly stressed silicon nitride films exhibit some of the highest Q (Quality factor) values observed in MEMS/NEMS structures at room temperatures. By varying the diameter and thickness of high stress silicon nitride circular "drum" structures, we observe that the dissipation follows generally predictable behaviors. Qualitatively we see that the fundamental out-of-plane resonance mode has the lowest Q in large structures (though it can still exceed 10^5). As higher modes with radial nodal lines (described as cake like modes) are added, the Q increases. Modes that add a nodal line at a constant radius also have higher Qs, but the Q improvement over the fundamental is not large, and can also lead to lowering of the Q in small structures. Generally thinner more uniform cross section structures reveal the higher Qs. As higher order resonances are excited, the product of the frequency times Q (*fQ*) tends to a constant. Many of these behaviors are consistent with recent models. This presentation will describe the results obtained as the diameter and thickness of the devices of these structures was varied.

2:40pm **MN-MoA3 *In Situ* SEM Micro Tension Tests on Nanoscale Single Crystal Metals and Nanocrystalline Metals**, ***M. Yilmaz***, *J.W. Kysar*, Columbia University

We developed a microscale uniaxial tension test MEMS device for in-situ SEM experimentation, with potential use for in-situ TEM experiments as well. We have characterized batch compatible integrated ultra-thin nanocrystalline gold nanoscale samples (~40nm thick), as well as externally integrated single crystal gold, and single crystal gold-silver thin-film nanoscale samples (~100nm thick) for their mechanical properties in-situ SEM.

The MEMS device is composed of an electrostatic comb-actuator, and two displacement sensors, with the purpose to mechanically characterize nanoscale samples that are located between the two displacement sensors. Sub-pixel resolution Digital Image Correlation (DIC) on SEM micrographs is used as displacement tracking technique in order to quantitatively characterize the displacement fields of the displacement sensors so as to obtain the force-elongation (hence, stress-strain) behavior of the tested samples. From the stress-strain behavior of the tested nanoscale specimens, we experimentally obtained fundamental material properties such as Young's modulus, and critical resolved shear stress for single crystal materials, and Young's modulus for nanocrystalline ultra-thin gold samples.

The method we apply in this study is the first in its field with the capability to integrate such small samples to MEMS with monolithic microfabrication. Although we worked with ultra-thin film gold, and thin-film gold and gold-silver alloy, the method can be adapted to other materials of interest, such as metals, carbon nanotubes, and graphene.

The results of the experiments are of interest to microelectronics industry, and materials research community.

3:40pm **MN-MoA6 Fabrication and Testing of Suspended Piezoelectric Nanocomposite Membranes**, ***J.R. Fox***, *S.B. Horowitz, J.P. Cortes, M.S. Allen, A.D. Mathias, L.A. Barkett,* Ducommun Miltec, *M. Sanghadasa*, U.S. Army Aviation and Missile Research Development and Engineering Center

The fabrication and testing of a novel piezoelectric microphone consisting of a suspended nanocomposite membrane of oriented hydrothermally-grown ZnO nanorods in a matrix of SU-8 photopolymer is detailed. High aspect ratio ZnO nanorods (0.5 – 1 microns long) were grown using a low temperature hydrothermal process on a patterned gold electrode on a wet-oxidized wafer. Then SU-8 photoresist was spin-coated over the rods to a thickness of approximately 1 micron and subsequently exposed and developed to reveal access to the bottom bond pads of the structure. A top electrode was deposited via sputtering and patterned on the SU-8 polymer. Finally a deep reactive ion etch (Bosch process) through-wafer silicon etch was used to release the nano-piezo-membranes in diameters of 50-400 microns. The infiltration of SU-8 photopolymer into the nanorods was observed to improve during long post-exposure bakes of the polymer as well as produce an improvement in vacuum compatibility of the polymer. Laser Doppler vibrometry (LDV) was used to characterize the actuation of the nano-piezo-membrane both under application of a driving potential as well as during acoustic load from a plane-wave tube.

4:00pm **MN-MoA7 Fabrication of Nanoelectromechanical Systems via the Integration of Glancing Angle Deposition Thin Films**, ***J.N. Westwood***, *V.T.K. Sauer, J.K. Kwan,* University of Alberta, Canada, *W.K. Hiebert,* National Institutute for Nanotechnology, Canada, *J.C. Sit*, University of Alberta, Canada

Nanoelectromechanical systems (NEMS) have been shown to be far more sensitive than microelectromechanical systems (MEMS). However, their smaller size also reduces the surface area of the device. This is problematic when scaling gas- and mass-sensing MEMS to the nanoscale regime because it reduces the area for analyte adsorption. Nanostructured thin films grown by glancing angle deposition (GLAD) provide a potential solution to this issue. GLAD thin films, deposited by evaporation at highly oblique angles between the source and the substrate, have extremely high surface area which can be used to counteract the decreased surface area of NEMS. The low density of GLAD films permits the increase in surface area without adding significant mass. Successful surface functionalization of GLAD films has also been demonstrated. These factors indicate that GLAD films are promising candidates for NEMS sensor applications. A major drawback, however, is that GLAD films are very difficult to pattern using lithography because they are incompatible with the wet processes required for photoresist development and removal. We have devised an alternative process that requires no lithographic patterning of the GLAD by depositing the films on patterned and released NEMS doubly clamped beams. The NEMS are fabricated from silicon-on-insulator wafers by etching away the oxide layer to give released silicon NEMS. Silicon dioxide GLAD films are then deposited. The GLAD films show good uniformity and limited edge effects. These GLAD-coated NEMS, or GLEMS, show significant potential for sensing applications. There are many parameters available for future optimization, including beam dimensions and GLAD film deposition parameters.

4:20pm **MN-MoA8 Electroactive Polymeric MEMS Actuators Fabricated by Thermal Imprinting of P(VDF-TrFE-CFE) and Poly(dimethylsiloxane) (PDMS)**, *J. Shkovsky,* ***L. Engel***, *A. Reuveny, Y. Sverdlov, Y. Shacham-Diamand, D. Schreiber, S. Krylov*, Tel Aviv University, Israel

The rapidly developing field of polymeric electronic and microelectromechanical (MEMS) devices has attracted much attention in recent years. Applications of polymeric MEMS devices include thin film transistors, waveguides for optical sensors, stretchable electronics as well as electroactive polymers (EAP) and dielectric elastomers actuators (DEAs). Polymeric actuators are distinguished by their very low fabrication cost, are often biocompatible, demonstrate large strain under small forces, exhibit fast response times, relatively large actuation forces and high efficiency. Because the electric fields required for the actuation of these devices are relatively high, of the order of tens or even hundreds of V/*m*m, reduction of the thickness of the polymeric layers is crucial for toreducing operational voltages. Thin layers of polymeric materials in MEMS devices are typically formed by spin-coating using diluted solutions of the uncured polymer. However, the spin-coating of polymers into micron scale films is challenging as there are strict requirements for film thickness, uniformity, process integration and defect density.

In this work we report on a novel fabrication process based on thermal imprinting for the formation of micron-scale, freestanding, layers of two polymeric materials, the dielectric elastomer poly(dimethylsiloxane) (PDMS) and the electroactive relaxor P(VDF-TrFE-CFE). We have fabricated freestanding, smooth, defect-free membranes with thicknesses in the range of 0.4–4.8 μm and with diameters of several millimeters . Since the ability to detach the membrane from the chips after imprinting is critical for the production of freestanding layers, the adhesion between the polymers and the silicon (Si) stamp and the Si substrate is reduced by the deposition of a hydrophobic dodecyl-trichlorosilane monolayer on the chips prior to imprinting. We demonstrate the feasibility of patterning the devices at the time of imprinting to create freestanding patterned micron-scale structures. A simple device made up of a freestanding circular membrane with electrodes on the circumference demonstrating the application of the method is presented . The results of the device's electromechanical characterization revealed that a free-standing PDMS membrane 1 mm in diameter and 5.3 *m*m thick demonstrated displacements of 5 *m*m at an actuation voltage of 300 V.

Acknowledgements

This project was supported by Arkema/Piezotech . P(VDF-TrFE-CFE) materials were supplied by Piezotech S.A.S

4:40pm **MN-MoA9 A CMOS MEMS Gas Sensor Using Monolayer Protected Gold Nano-Clusters Coating on Three-Dimensional Interdigitated Electrodes**, *Y.C. Chen*, ***C.Y. Chang***, National Taiwan University, *H.L. Lu, C.-J. Lu,* National Taiwan Normal University, *W.-C. Tian*, National Taiwan University

In this work, a novel gas sensing platform using the TSMC 0.35μm CMOS-MEMS process was developed. Three-dimensional interdigitated sensing electrodes (3D IDEs) with a polysilicon microheater and a polysilicon thermometer were integrated in this CMOS-based platform. Compared to conventional 2D IDEs, our 3D IDEs not only extended the sensing surface area to the vertical sidewalls but also decreased the gap (the inter metal dielectric layer, IMD, thickness) between electrodes. The micoheater of 2.6kΩ resistance and the thermometer of 2kΩ resistance were designed to provide on-chip heating, which could facilitate the deposition and/or activation of the sensing material. The sensing material, monolayer protected gold nanocluster (AuC8), was coated onto the electrodes through the air brush spraying. The sensor performance was demonstrated with three compounds (Octane, Butanol, and Toluene) of concentrations in the range of 2000ppm to 5000ppm and manifested the good linearity and sensitivity.

The backend of CMOS processes for interconnects were utilized to provide microstructures which offer many potential advantages for sensors including low power consumption, low fabrication cost, high sensitivity and reliability. In the CMOS etching process, the design rules of a released (RLS) mask limit the minimum gap between electrodes to 3μm. Hence, 3D IDEs were used to decrease the gap between electrodes to 1μm (the IMD thickness) so as to increase the sensitivity of the designed sensor as well as to lower the resistance of the deposited sensing material. In addition, the consumption of the sensing material was reduced significantly.

When exposing to Toluene at different concentrations, the transient responses of the sensor were changed accordingly. The exceptional linearity of the sensor responses on targeted compounds at high concentrations was demonstrated. The great sensitivities, defined as the ratio of the impedance before and after exposing to the target gas, of the three compounds were obtained (Toluene: 3.66E-5/ppm, Octane: 3.30E-5/ppm, and Butanol: 5.71E-5/ppm). The differences in sensitivities are largely affected by the target gas and its affinity to AuC8 surface. These variations in sensitivity for different compounds can enhance the specificity of our CMOS-based gas sensor platform.

5:00pm **MN-MoA10 Integration of Functionalized Biological Nanostructures with Conventional Transducer Fabrication Schemes**, ***X. Fan***, *N. Siwak, A. Brown, J. Culver, R. Ghodssi*, University of Maryland

Nanoscale technologies have the potential to revolutionize a broad range of fields. Already, there have been a plethora of synthesis and fabrication techniques of nanostructures and devices utilizing both organic and inorganic materials. Biological molecules have transformed chem-bio detection, due to their innate ability to be tailored and engineered via genetics. These nanostructures can be versatile in their various binding properties making them attractive for a variety of applications. These biomolecules, often self-assembled or synthesized with a bottom-up approach, are used for scaffolding and functionalization purposes, while inorganic materials utilize conventional top-down lithographic techniques to pattern transducers. The integration of these two different technologies is challenging due to fabrication incompatibility. While inorganic materials are robust, biomolecules are highly sensitive to pH levels, temperature, and chemical compositions, making them incompatible with top-down techniques. Thus, their integration is often withheld until the final step of device fabrication. This prevents further backend processing once the biomolecules have been deposited, limiting the degree of integration of nanoscale platforms and restricting the full potential of nanotechnology.

Our team has developed a method to pattern a type of nano-biomolecules onto the active region of a photonic device using a hybrid top-down and bottom-up approach. An optical ring resonator, highly sensitive to refractive index changes, was fabricated using E-beam lithography to investigate the assembly of biomolecules on its surface. *Tobacco mosaic virus* (TMV) was then self-assembled onto the transducer's active area. TMV is a rod like structure with coat proteins that are genetically modified to allow for the self-assembly of viruses onto surfaces and the expression of functionalization on the outer surface. The TMV structure is stable in a pH 2-11 and temperature up to 60^{o}C that survives the conventional lift-off patterning process. This enables the patterning and alignment of biologically functionalized structures on lithographically fabricated transducers post self-assembly.

By integrating TMV structures onto the surface of the ring resonator, we will report on the optical properties of the TMV assembly, including its refractive index and optical loss, for sensing applications. This photonic platform with patterned TMV provides a compatible process to integrate biological nanostructures with conventionally fabricated transducers. This integration scheme we have developed will allow for an additional degree of control when developing nanoscale based hybrid platforms.

5:20pm **MN-MoA11 A Novel Computational and Experimental Methodology for Development of Therapeutic Microdevices for Rapid Reconstitution**, *S. D'hers,* Buenos Aires Institute of Technology, Argentina, *A. Alexander-katz*, ***N.M. Elman***, Massachusetts Institute of Technology

Rapid Reconstitution Packages (RRPs) represents a breakthrough microfluidic platform to use pharmaceutical drugs in ambulatory settings without the need for refrigeration. RRPs were designed as microfluidic cartridges, keeping drugs in lyophilized form (powder) for years and perform on demand reconstitution in the order of milliseconds. The unique integration of a dual multi-scale computational and experimental model with nano-materials and microfluidics provides the scientific basis towards the development of an ultra-portable platform for long term storage and extremely rapid reconstitution. The device architecture consisted of mixing microstructures, fabricated with Stereo Lithography Apparatus (SLA) with biocompatible materials. Rapid prototyping provided a quick turnaround experimental model for validating computational models, rendering a unique methodology for optimization. Experimental setup was intended to emulate temperature fluctuations in ambulatory environments. Experiments were performed using standard analytical methods on RRPs containing drugs exposed to temperatures in the range of 25-65 C. High Performance Liquid Chromatography (HPLC) assays for quantifying reconstitution, and Enzyme-Linked Immunosorbent Assays (ELISA) for activity were performed. Several drugs were tested, including atropine for resuscitation, and tissue plasminogen activator (tPA) for treatment of thrombosis. The design was optimized in parametric software and tested for manufacturability and functionality using Computed Fluid Dynamics (CFD) analyses. Design optimization using the CFD models was performed with the goal of reducing drug retention in the device and tailoring drug concentration profiles during activation. A consistent fluidic representation was adopted to model drug dissolution and diffusion. The numerical scheme was validated through computational and laboratory tests for drug dose and concentration profile. Experimental results reveal the importance of the combined use of computational and experimental techniques. The convergence of these unique techniques allows exploitation of physical processes at the nanometer and micrometer scales to investigate the lyophilization and reconstitution processes, overall rendering a synergistic computational and experimental methodology for development of therapeutic microdevices. The use of RRPs will result in significant improvement in logistics for a number of civilian and military applications.

Graphene and Related Materials Focus Topic
Room: 13 - Session GR+AS+EM+MI+MN-TuM

Optical, Magnetic, Mechanical and Thermal Properties
Moderator: K.I. Bolotin, Vanderbilt University

8:00am **GR+AS+EM+MI+MN-TuM1 Characterization of Magnetically Tunable Iron Nanorod Coated Graphene Nanoplatelets**, ***S.D. Johnson***, *M.H. Gowda, S.-F. Cheng, N.Y. Garces, B. Feigelson, F.J. Kub, C.R. Eddy, Jr.*, U.S. Naval Research Laboratory

Composites made from iron coated graphene nanoplatelets (GNPs) show promise for applications such as, magnetic switches, electromagnetic interference shielding, and electromagnetic waveguides due to the large conductivity of GNPs combined with the magnetism of iron. Additionally, this composite can be easily formed into millimeter thick sheets making it a promising composite for other applications.

We report a novel method to synthesize iron oxide compound onto GNP using microwave hydrothermal synthesis at 60° C and reaction times between 10 and 120 minutes. Scanning electron microscopy imaging reveals iron oxide nanorods approximately 100 nm long adhered to the GNPs for reaction times as short as 10 minutes. X-ray photoemission spectroscopy reveals that the iron/carbon ratio remains constant across these reaction times. The resistivity of the composite increases with reaction time from 0.2 to 0.6 ohm-cm. Saturation magnetization and coercive field values follow a decreasing trend with increasing reaction time. From 10 to 120 minutes saturation magnetization decreases by 70% from 170 emu/g and coercive field decreases by 40% from 52 Oe. Remnant magnetization of around 0.7 memu/g remains constant throughout. We also report the temperature-dependent magnetic response of the compound across the Morin transition, which for submicron particles of α-Fe_2O_3 is near 250 K.

Preliminary results suggest that while the nanorod size and quantity remains constant with reaction time, the resistive and magnetic properties change. This may suggest that we are tuning the magnetism of the system by changing the iron structure between the ferromagnetic γ -Fe_2O_3 and the antiferromagnetic α -Fe_2O_3.

8:20am **GR+AS+EM+MI+MN-TuM2 Dynamical Origin of Blue Photoluminescence from Graphene Oxide**, ***A.L. Exarhos***, *M.E. Turk, P.M. Vora, J.M. Kikkawa*, University of Pennsylvania

The tunable broadband emission from graphene oxide (GO) has sparked significant interest in research regarding its potential for band gap engineering. Here, we use polarization sensitive time-resolved optical spectroscopy to study the spectral diffusion and sub-picosecond dynamics of the excited carriers in GO and photo-exposed GO, where photo-exposure has been demonstrated to constitute a reducing condition. In steady state measurements, a significant blueshifting of the photoluminescence (PL) is observed with photo-exposure. This blueshift correlates with a marked difference in the temporal behavior of the PL from GO and photo-exposed GO. The PL spectra are very similar at short delay times, but an increased non-radiative recombination rate in the exposed GO leads to a decreased lifetime in the material. Utilizing in-plane polarization memory measurements, we examine the electron-hole polarization in these systems which can probe excitonic effects and help to provide a better understanding of the role of the sp^2 graphene lattice in GO and exposed GO. We further discuss the relevance of our data to the origins of PL in these systems.

A.L.E. gratefully acknowledges the support of NSF DMR-0907226. M.E.T., P.M.V., and the construction of a Kerr gate system are supported by the Department of Energy Office of Basic Energy Sciences Award DE-SC0002158.

8:40am **GR+AS+EM+MI+MN-TuM3 Spin-Transport and Magnetism in Graphene**, ***R. Kawakami***, University of California, Riverside **INVITED**

Graphene is an attractive material for spintronics due to its high mobility and the low intrinsic spin-orbit and hyperfine coupling, which should lead to excellent spin transport properties. In 2007, graphene became the first material to exhibit gate tunable spin transport and spin precession at room temperature. However, the spin injection efficiency was low and the spin lifetime was much shorter than predicted theoretically. In this talk, I will report on our progress in this area. The low spin injection efficiency into graphene is due to the conductivity mismatch between the ferromagnetic metal (Co) spin injector and the single layer graphene (SLG). To alleviate this problem and enhance the spin injection efficiency, we developed atomically smooth MgO tunnel barriers by utilizing a TiO_2 seed layer. With tunneling contacts, the non-local spin signal is found to be as high as 130 ohms at room temperature, with a spin injection efficiency of 30%. In addition to improving the spin injection efficiency, the tunneling contacts were found to improve the spin lifetime as well. This indicates that the short spin lifetimes reported before are due to the contact-induced spin relaxation from the ferromagnetic electrodes. Using tunneling contacts, we investigate spin relaxation in single layer graphene (SLG) and bilayer graphene (BLG). At low temperatures, contrasting behaviors of gate voltage dependence of the spin lifetime are observed between SLG and BLG, which suggest different mechanisms for spin relaxation in SLG and BLG. A final topic of interest is magnetism and the formation of magnetic moments in graphene. While there is substantial theoretical work on magnetic moments generated by hydrogen adatoms and lattice vacancies, the experimental situation is less clear. We have developed a new method for detecting magnetic moment formation based on scattering of pure spin currents in graphene spin valves. We will report the progress on our efforts to identify magnetism with this approach.

10:40am **GR+AS+EM+MI+MN-TuM9 A "How To" for Magnetic Carbon**, ***H. Ohldag***, SLAC National Accelerator Laboratory, *E. Arenholz, T. Tyliszczak*, Lawrence Berkeley National Laboratory, *D. Spemann, R. Hoehne, P. Esquinazi, M. Ungureneau, T. Butz*, University of Leipzig, Germany

While conventional wisdom says that magnetic materials have to contain some metallic atoms, the confirmation of intrinsic magnetic order in pure metal free carbon represents an ultimate and general scientific breakthrough because of the fundamental importance of carbon as an elemental building block of organic as well as inorganic matter. The common controversy raised across all disciplines is whether the magnetism of carbon is intrinsic or induced by other elements. We address this controversy by providing clear experimental evidence that metal free carbon can be ferromagnetic at room temperature using dichroism x-ray absorption spectro-microscopy. For this purpose we acquired soft x-ray microscopy images of magnetic structures on a thin carbon film that have been produced by irradiation with a focused 2.25MeV proton beam. Our element specific magnetic probe shows no indication of magnetically ordered Fe, Co or Ni impurities in these samples. In a second step we investigate the particular electronic states that are involved in carbon magnetism and find that the carbon p-states as well as C-H bonds show a magnetic moment, indicating that hydrogenation plays a crucial role in developing the ferromagnetic order. Our surface sensitive approach reveals that the magnetism at the surface of the irradiated graphite samples is much larger than in the bulk of the sample. We observe a surface magnetic moment similar to what is typically present in classical ferromagnetic 3d transition metals.

REFERENCES

P.Esquinazi et al., *Magnetic order in graphite: Experimental evidence, intrinsic and extrinsic difficulties*, Journal of Magnetism and Magnetic Materials, Vol 322, 1156 (2010).

H. Ohldag et al.,*p-Electron ferromagnetism in metal free carbon probed by soft x-ray dichroism*,Phys. Rev. Lett. 98, 187204 (2007) H. Ohldag et al., *The role of hydrogen in room temperature ferromagnetism at graphite surfaces*, *New J. Phys.* 12 123012 (2010)

11:00am **GR+AS+EM+MI+MN-TuM10 From Graphene to Amorphous Carbon by Sublimation and Condensation**, ***B. Steele***, *R. Perriot, V. Zhakhovsky, I.I. Oleynik*, University of South Florida

The mechanisms of the non-equilibrium melting process of graphene and the structure of the liquid phase of carbon was studied by molecular dynamics (MD). Graphene undergoes a non-equilibrium melting process at high temperature and low pressure as the carbon chains are formed out of the graphene sheet, thus making up a transient liquid phase of carbon. As the chains expand the material sublimates to a low dense gas of carbon chains. Under higher pressure the gas phase will condense to an intermediate porous phase of carbon with a significant sp^2 fraction of atoms, followed by the liquid phase, and finally an amorphous phase. Mechanisms of melting of graphene, including formation of topological and Stone Wales (SW) defects in two and three dimensions will be discussed.

MEMS and NEMS
Room: 10 - Session MN-TuM

Optomechanics and Photonic MEMS and NEMS
Moderator: W.K. Hiebert, University of Alberta and The National Institute for Nanotechnology

8:40am **MN-TuM3 Focused Ion Beam Fabrication for Nanophotonics and Microsystems Integration**, ***I.W. Jung***, Argonne National Laboratory **INVITED**

Focused ion beam (FIB) fabrication has become an invaluable tool to pattern nanoscale features on a vast range of materials otherwise not available due to the limitations on selectivity of chemical based reactions for etching. In addition, direct milling to create features allows precision patterning on a variety of surface topology. In this talk, we present work on using the FIB to pattern and integrate nanophotonic elements, e.g. photonic crystals, and microelectromechanical systems for novel device applications. In addition, we explore how the FIB can also be used for applications in integration of microsystems with plasmonic structures to achieve precision control of light-matter interactions at the nanoscale.

9:20am **MN-TuM5 Nanomechanical Resonator Detection using Racetrack Resonator Structures for Use in Mass Sensing**, ***V.T.K. Sauer***, *Z. Diao, M.R. Freeman, W.K. Hiebert*, University of Alberta and The National Institute for Nanotechnology, Canada

Nano-optomechanical systems have been demonstrated as an excellent mechanism for detecting the motion of nanomechanical resonators. They have very high displacement sensitivities and also very large frequency detection bandwidths. These properties make nano-optomechanical systems a promising platform for on-chip inertial mass sensing. By mass loading a resonating mechanical device, the mass of the analyte can be determined by measuring the frequency change this addition of mass causes. The high displacement sensitivities and large operational bandwidth allow for smaller mechanical resonators to be measured which allows for smaller masses to be detectable. Both cantilevers and doubly clamped beams have been detected using the interaction of the evanescent fields from photonic structures such as waveguides, ring/racetrack resonators and torroid structures. Many of these devices incorporate the mechanical resonator directly into the photonic element, but for optimal use in a mass sensing system it is preferable that any added mass not interact directly with the photonic modes. This can cause losses or other uncontrollable effects that negatively impact the operation of the mass sensor. To avoid this, the mechanical element should interact with, but still be external to, the optical cavity structure. Here, cantilever beams 0.5 to 5 μm long and doubly clamped beams 3 to 10 μm long are fabricated 70 to 170 nm from a ring resonator optical cavity. As a beam oscillates in the plane of the wafer, toward and away from a ring resonator, it modulates the ring's effective index. This causes a phase shift in the ring which is detected by a probe laser. The beams are actuated using a power modulated pump laser which uses an optical gradient force to pull the beams toward the optical structure. To increase their mass sensitivity, the devices are implemented into a phase-locked loop and their frequency stabilities are measured.

9:40am **MN-TuM6 Fabrication and Characterization of Ultra-Fast Electrostatically-Actuated Surface Micro-Machined Aluminum Mirrors**, ***J.R. Fox***, *A.D. Mathias, J.P. Cortes, M.S. Allen, S.B. Horowitz*, Ducommun Miltec, *M.G. Temmen, M. Sanghadasa*, U.S. Army Aviation and Missile Research Development and Engineering Center

The design, optimization, fabrication, and characterization of an electrostatically-actuated, surface micro-machined aluminum, torsional-beam micro-mirror is presented. The design was optimized to produce a 5 degree tilt of the 20 x 20 micron mirrors with a settle time of less than 6 microseconds with a 190 V electric potential across a 3 micron gap. The design is repeated in 25 x 25 arrays for high-speed deflection of incident light as an optical shutter. Utilization of the COMSOL Multiphysics finite element analysis environment for parameterization of geometries is described and the resulting optimized micro-mirror design is detailed. Fabrication of micro-mirrors via argon ion-mill patterning of aluminum over sacrificial resists and their subsequent dry release with oxygen plasma will be described. The micro-mirrors were then subjected to scanning electron microscopic examination, and laser-Doppler vibrometry was used to examine micro-mirror actuation performance.

10:40am **MN-TuM9 A Comparison of Different Releasing Methods in Fabricating Nano-Optomechanical Devices**, ***Z. Diao***, National Institute for Nanotechnology, NRC Canada and University of Alberta, Canada, *V.T.K. Sauer, J.E. Losby, M.R. Kan, M.R. Freeman*, University of Alberta and The National Institute for Nanotechnology, Canada, *W.K. Hiebert*, National Institute for Nanotechnology, NRC Canada and University of Alberta, Canada

Nano-optomechanical systems (NOMS), in which guided light is utilized to actuate and transduce the motion of nanomechanical resonators, have received intense attention in recent years [1, 2]. This actuation and transduction scheme offers unprecedented displacement sensitivity and ultrahigh bandwidth, which is also able to be fully integrated with state-of-the-art opto-electronic and semiconductor technology. It can be envisioned that NOMS will see a large variety of applications in mass sensing, gradiometry, and high precision frequency counting.

Strong optical forces and large evanescent field gradient, both critical factors in defining the actuation efficiency and the motion transduction sensitivity in a nano-optomechanical system, only exist in a distance smaller than the wavelength of light from the nanophotonic waveguide. This requires the nanomechanical resonator in NOMS to be brought in close proximity to adjacent nanophotonic structures (normally in the range of 100 – 300 nm). A well known device failure mechanism in this case is stiction of released structures due to attractive forces with adjacent surfaces. A critical point drying process is so far conventionally utilized in NOMS fabrication to remedy this issue [1, 2].

In this work we report on our attempt in utilizing alternative device releasing protocols in fabricating NOMS structures. The test structure selected is a several tens of micrometers long doubly clamped beam embedded in a race-track nanophotonic resonator. The entire device was fabricated on a silicon-on-insulator substrate with deep-UV lithography. The race-track resonator possesses an optical quality factor of a few tens of thousands and a finesse of ~ 20. The large finesse of the optical resonator allows sensitive motion transduction in which thermomechanical noise of a ~ 10 μm long device was able to be detected. Device releasing methods tested include sublimation drying with dichlorobenzene and cyclohexane, and hard masked hydrofluoric acid vapour etching. Finally, we also discuss the influence of different device releasing methods on the photonic properties of the system and the undercut profile.

[1] M. Li et al., Nature Photon. **3**, 464 (2009).

[2] J. Roels et al., Nature Nanotech. **4**, 510 (2009).

11:00am **MN-TuM10 Optomechanical Experiments with Large Area Graphene Membranes**, ***V.P. Adiga***, *R.A. Barton, I.R. Storch, B.R. Ilic, C.B. Wallin, P.L. McEuen, J.M. Parpia, H.G. Craighead*, Cornell University

Large area, ultra-thin membranes are useful as mechanical resonators whose mechanical degree of freedom can be easily controlled using light due to low spring constants and resonator mass. In this regard, there are advantages associated with using two dimensional materials like graphene and ultrathin silicon nitride. However, achieving large area suspended devices with high mechanical quality (Q) factors in these high surface-to-volume-ratio resonators has been a challenge. Recently it has been observed that the Q of these membranes can be significantly improved by a combination of tensile stress, resonator geometry and optimized fabrication techniques. Here we fabricate CVD grown, electrostatically tunable graphene drums of diameter up to 100 mm and measure high quality factors (up to 4000) at room temperature. We then use lasers to control the amplitude of mechanical vibrations using the back action provided by the photothermal effect. We can effectively cool (increase the effective damping) or heat (decrease the effective damping leading to self oscillation) the graphene membrane in a Fabry-Perot cavity formed by the membrane suspended over prefabricated trench, with cavity detuning provided by a highly reflective movable mirror. The strong optomechanical coupling1 observed in these membranes is due to the low mass and relatively strong absorption in the atomic monolayer.

1) *Cavity Optomechanics with Graphene Resonators,* R. A. Barton et al, Submitted.

11:20am **MN-TuM11 Optomechanics of Graphene Resonators**, ***R.A. Barton***, *I.R. Storch, V.P. Adiga, R. Sakakibara, B.R. Cipriany, B.R. Ilic, S. Wang, P. Ong, P.L. McEuen, J.M. Parpia, H.G. Craighead*, Cornell University

By virtue of their low mass and stiffness, atomically thin mechanical resonators are attractive candidates for use in optomechanics. Graphene, in particular, is an ideal material to investigate as it possesses excellent electrical and mechanical properties as well as a strong interaction with

light over the entire visible range. Here, we demonstrate photothermal back-action in a graphene mechanical resonator comprising one end of a Fabry-Perot cavity. As a demonstration of the utility of this effect, we show that a continuous wave laser can be used to cool a graphene vibrational mode or to power a graphene-based tunable-frequency oscillator. In addition to enabling studies of fundamental physics, the remarkable sensitivity of graphene optomechanical resonators and their ability to operate over a broad range of wavelengths and mechanical frequencies makes them attractive for applications.

MEMS and NEMS
Room: Central Hall - Session MN-TuP

MEMS and NEMS Poster Session

MN-TuP1 Tangential Momentum Accommodation Coefficients in Coated Microtubes, *M. Hadj Nacer, I. Graur, P. Perrier, J.G. Méolans,* Aix-Marseille Université, Ecole Polytechnique Universitaire de Marseille, France, ***M. Wüest***, INFICON Ltd, Liechtenstein

The experimental setup based on the constant volume technique is developed to measure the mass flow rate through microtubes under isothermal stationary flow conditions. Four different working gases (helium, nitrogen, argon and carbon dioxide), and two surface materials (Stainless Steel and Sulfinert) are considered.

The Knudsen number calculated for the experimental conditions varies from 0.0001 (hydrodynamic regime) to 0.1 (slip regime). In this range the approach based on the analytical solution of the Stokes equation subjected to the first order velocity slip boundary condition is used. The velocity slip coefficient and the Tangential Momentum Accommodation Coefficient (TMAC) are extracted from the experimental data of the mass flow rate using their analytical expressions.

The results are summarized in the tables representing the accommodation coefficients for the corresponding gas-surface material combinations. The influence of the molecular mass on the tangential momentum accommodation coefficient is discussed.

MN-TuP3 Development of Deposition and Etching Processes of Thick ZnS Films for Pixel Level Packaging of Infrared Focal Plane Arrays, ***B. Glück***, ST Microelectronics, France, *G. Rodriguez, G. Dumont, S. Barnola*, CEA, LETI, MINATEC Campus, France

Because of its transparency in the medium and long wave infrared light (MWIR and LWIR) zinc sulphide (ZnS) is an attractive material to make optical windows for infrared devices. Moreover, its relatively low optical index can be used advantageously in association with high index material such as Germanium (Ge) to create an anti-reflecting coating.In our application a thick ZnS film of about 1.2µm is deposited on top of the Ge to create the infrared window of a micro packaging structure for micro-bolometer devices in 200mm. This window has to be opened at the end of the process flow to realise the contacts. This study focuses on the deposition of ZnS by electron beam evaporation and its reactive ion etching to form the first layer of the IR-window. The integration of these processes in the fabrication of infrared focal plane arrays is presented in this work.In particular structure and morphology of the deposited ZnS films were investigated by X-ray diffraction, X-ray reflection, atomic force microscopy and scanning electron microscopy. Spectroscopic ellipsometry measurements were done to determine the optical properties. Concerning ZnS etching we developed a HBr based etch process that is also applicable to etch the Ge layer underneath using the same mask. The main process trends were investigated to maximise the etch rate and the selectivity to photoresist.

MN-TuP4 Low Damage Etching Process for Fabricating Micro Electro Mechanical Systems (MEMS) Devices using Neutral Beam, ***K. Miwa***, *Y. Nishimori, S. Ueki,* BEANS Laboratory, Japan, *M. Sugiyama,* The University of Tokyo, Japan, *T. Kubota, S. Samukawa*, Tohoku University, Japan

We have developed low damage etching process suitable for fabricating micro electromechanical systems (MEMS) devices based on silicon by using neutral beam. For cutting edge three-dimensional (3D) MEMS devices, high aspect ratio structures are indispensable. In addition, the surface of the silicon device is required to be smooth enough to achieve excellent properties as electromechanical or optical devices. However, conventional processes using reactive ion enhanced etching (RIE) of silicon are likely to have rough surfaces called scallops or ripples on sidewalls. Furthermore, conventional plasmas used for etching process cause damages on the etched surfaces by ion and electron fluxes (charges) or vacuum ultraviolet/ultraviolet (VUV/UV) light emission from etching plasmas. In turn, neutral beam etching technology is able to achieve ultra-low damage etching and to obtain atomically flat silicon surfaces. Therefore, by using neutral beam we have developed novel dry process to fabricate silicon MEMS devices of which surface is smooth enough and have damage-less surfaces. The neutral beam was produced from an inductively coupled plasma (ICP) of pure Cl_2 gas in an etching tool. The ICP was generated by radio frequency (RF) wave (13.56 MHz) and the RF was time modulated at 10 kHz and the duty ratio was 50% (50µs ON/ 50µs OFF). Ions in the ICP were accelerated toward a carbon aperture and neutralized by colliding onto the aperture which was biased by applying 450 kHz alternating voltage. Two types of apertures are used for the experiment. The apertures have many small holes of its aspect ratio is approximately 10 and 20, respectively. We have found that the etched silicon trench profile by the neutral beam depend on bias voltage and aspect ratio (AR) of the aperture. In addition, mask material can change the trench shape.By optimizing these conditions to produce neutral beam, we have obtained silicon trenches which have perpendicular trench profile of its width is around 200 nm. No defects or damaged layer are seen in transmission electron microscope (TEM) observation of the trench sidewalls. The study was supported by new energy development organization (NEDO). Authors would like to thank to NEDO and project members in BEANS Laboratory.

MN-TuP5 Deveropment of Test Instrument for the Mechanical Strength of Micro-nano Wires, ***A. Kasahara***, *M. Sasaki, H. Suzuki, M. Goto, M. Tosa*, National Institute for Materials Science (NIMS), Japan

Recent nano-technology researches have created various advanced micro-nano materials.

In particular, there have been many reports on nano-meter-scale tubes and wires such as carbon nanotubes

and silicon wires.

We have prepared long crystal silicon wires with a diameter of several tens of nano meters at a temperature lower than 523k by using the low-pressure low-temperature CVD method too. To use these as materials for application to micro-nano electromechanical system, we need to fully understand their electric, chemical and mechanical properties.

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 nano scale equivalent of mechanical strength testers for ordinary materials.This time, we are developing the device which could support a bending or shearing test.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 mechanical strength measurement device.

MN-TuP6 Tin-Oxide Nanostructured Arrays Based Integrated MEMS Device for Low Temperature Hydrogen Detection, ***R.N. McCormack***, University of Central Florida, *N. Shirato,* University of Tennessee, *U. Singh, S. Das, A. Kumar, H.J. Cho,* University of Central Florida, *R. Kalyanaraman,* University of Tennessee, *S.S. Seal*, University of Central Florida

In the pursuit of an alternative fuel source, hydrogen gas appears to have the best potential. All hydrogen gas related processes require accurate monitoring for leaks during the storage, transportation and usage. The problem that arises with the use of hydrogen is its tendency to leak along with being highly explosive at 4-vol%. Most of the current metal oxide based chemi-resitors in use as detectors operate at elevated temperature (above 100 degree Celsius) in order to aid their sensor's response kinetics. This becomes a safety concern due to its proximity to the highly explosive hydrogen gas. The search for low temperature sensitive hydrogen sensing device is at the forefront of our research endeavor.

SnO2 was deposited on SiO2/Si substrates through the method of pulse laser deposition (PLD) to form thin film. Through the process of nanosecond pulse laser interference irradiation of the thin film, successfully architectured SnO2 nanoarrays were developed. These nanowire-like SnO2 structures fabricated were uniformly distributed along the surface of the substrate. Dimensions of the nanostructure were obtained through Atomic Force Microscopy (AFM) and Scanning Electron Microscopy. Results obtained illustrate that the nanoarray's nanowires were ~8 nm in cross-sectional height and tens of microns in length. Both thin film and nanoarray were then incorporated into MEMS device. Tests of chemi-resistors were conducted at room temperature within the concentration limits of 300 to 9000 ppm under dynamic condition, simulating the actual environments of exposure. In comparison to SnO2 thin film, the nanoarray illustrates a significantly larger electrical response upon exposure to concentrations as minimal as 600 ppm. Nanoarray exhibited a (drop in resistances by 2 orders of magnitude) 150 fold increase in electrical response in comparison to that of the thin film.

SnO2 nanoarray incorporation into the MEMs platform has successfully produced a low temperature hydrogen sensor. The performance of the nanoarray showed promising applicability due to it fast response time, high electrical response and its robustness. Theoretical models of the depletion layer and the diffusive characteristic within SnO2 were developed in order

to exemplify the combined sensing mechanism due to the nanoarray's geometry. This research endeavor therefore combines aspect of interdisciplinary materials design and integration alongside MEMS design, experimental conduction and modeling of device mechanism in the development a gas detector.

MN-TuP7 Nanoimprint Block Co-polymer Enhanced Nanostructure Lithography, ***J. Zendejas***, *B. Wong, S. Franz, R. Candler*, UCLA

As the demand for higher densities in microelectronic devices increase, the strain on current lithographic technologies becomes great. To achieve greater performance, smaller feature sizes are necessary and will require innovative lithographic technologies. One technique, called Nanoimprint Lithography (NIL) differs from traditional lithography in the exposure and development process, which is replaced by a process in which a resist on a substrate is imprinted by a patterned mold. The nano-scale reproducible patterns allow for a high-throughput technique that saves much processing time and cost. At the UCLA NRF, we are developing baseline recipes for producing nanoscale pattern transfers using PMMA and mr-I (Micro Resist Technologies) polymers. The ability to mass produce nanoscale patterns (C.D.<100 nm) will have a great impact on projects ranging from microelectronics to bioengineering. Using the NIL techniques learned at the UCLA NRF, two target applications have emerged. The cost effective means to produce nanoscale

patterns has made it possible to readily investigate; block copolymer (BCP) lithography and nanoimprint assisted DNA sequencing.

MN-TuP9 Electrostatic Deposition of a Micro Solder Particle Using a Single Probe by Applying a Single Rectangular Pulse, ***D. Nakabayashi***, *K. Sawai, P. Hemthavy, K. Takahashi, S. Saito*, Tokyo Institute of Technology, Japan

Recently, demands for micromanipulation techniques have increased in order to realize highly functional microdevices such as MEMS. A technique to deposit a conductive microparticle onto a conductive substrate by using a single conductive probe as a manipulator has been proposed as one of the techniques. The technique can be used to increase the yield of a ball-grid-array (BGA), which is used for IC packaging, by fixing the individual soldering defect. Adhesional force between the probe and the microparticle is dominantly greater than gravitational force on the microparticle due to scaling law. Thus, repulsive force must be generated to detach the microparticle from the probe. In the technique, a solder particle with a diameter of 20–30μm, initially adhering to the probe tip, is detached and deposited onto a substrate by applying a voltage between the probe and the substrate to exert an electrostatic force on the particle. However, when a constant voltage was applied, the detached particle mostly went out of the microscopic view due to the excessive impact of the collision between the particle and the substrate. In the previous research, a voltage sequence was optimized in order to reduce the excessive impact. The success rate of the particle deposition in the previous research was 44%, and is not sufficient for industrial applications. In this study, a technique to deposit the particle on the substrate by applying a single rectangular pulse is proposed, and the mechanism of the deposition by the proposed technique is described. In the mechanism, an electric discharge between the probe and the particle when the particle reaches the substrate plays a dominant role in the particle deposition. The current of the electric discharge generates the Joule heat due to the contact resistance between the particle and the substrate. The small part of the particle which contacts the substrate is melted by the Joule heat, and the melted part absorbs the impact of the collision between the particle and the substrate. Consequently, the particle is successfully deposited onto the substrate. Moreover, the mechanism of the proposed technique is verified by experiments of particle deposition, which are observed by using a high-speed camera (645,000 frames per second), a scanning electron microscope (SEM) and an oscilloscope. The success rate of the particle deposition has improved to 93% by the proposed technique. Furthermore, the Joule heat and the volume of the melted region are evaluated as indicators of the damage to the particle caused by the electric discharge using an RC circuit model, and the applicability of the proposed technique is discussed.

Late Breaking Session
Room: 14 - Session LB+EM+GR+MN+TR-WeA

Select Topics in Surface and Interface Science
Moderator: C.R. Eddy, Jr., U.S. Naval Research Laboratory, J.M. Fitz-Gerald, University of Virginia

2:00pm **LB+EM+GR+MN+TR-WeA1 Degradation Kinetics of Hard Gold Tribofilms**, ***N. Argibay***, *M.T. Dugger, M.T. Brumbach, S.V. Prasad*, Sandia National Laboratories

Hard gold coatings are low alloy (> 98% Au) films exhibiting relatively low friction, electrical contact resistance (ECR) and chemical reactivity, making them uniquely suited for use in dynamic electrical connections. Hardness is primarily a result of grain refinement achieved through alloying. At relatively low temperature (approx < 0.5Tm) the diffusion of codeposited and underlayer species toward the free surface, dominated by grain boundary and pipe diffusion, has been identified as a principal degradation pathway. The consequent formation of metal oxides deteriorates ECR and often contributes to increased wear and friction. A clear antagonistic relationship exists between the hardening mechanism that improves tribological performance and the diffusion phenomena that reduce useful lifespan. This talk focuses on the role of diffusion and film morphology on the aging and degradation of the tribological and electrical characteristics of hard gold films.

2:20pm **LB+EM+GR+MN+TR-WeA2 Effect of Nitrogen Concentration on the Surface Properties of Plasma Nitrided Tool Steels**, ***P. Abraha***, *J. Miyamoto*, Meijo University, Japan

The nitriding of tool steel was performed in electron beam excited plasma using neutral nitrogen species and nitrogen ions. The plasma apparatus is composed of three regions: the discharge region, the acceleration region and the processing region. This set up has the advantage of controlling the energy and number of electrons involved in producing the plasma independently.

In this study, the control of the nitrogen concentration on the formation of the hard but brittle compound layer and the effect on the tribology of the tool steel surface were investigated. Electron probe micro-analyzer (EPMA) results revealed that nitrogen concentration of samples nitrided by neutral nitrogen species had deep diffusion layer before reaching the threshold value of 6% nitrogen concentration that is necessary for the formation of the compound layer. Whereas in the samples nitrided by nitrogen ions, compound layer was confirmed right from the onset of the nitriding process.

The results of our experiments show that in nitriding the tool steel for 6h, below the threshold value, a mirror finish surface (Ra=14nm) with a deep diffusion layer of (up to 80 micrometers) and a surface hardness of more than two times (1300 Hv) that of the untreated sample (600 Hv) were produced. Our results demonstrate that neutral species based nitriding is effective for high performance and high precision mechanical components that require high hardness and wear resistance without altering the as finished dimensional accuracy, surface roughness and appearance.

2:40pm **LB+EM+GR+MN+TR-WeA3 High Strength Carbon Fiber Composite Wafers for Microfabrication**, ***L. Pei***, *K. Zufelt, R. VanFleet, R.C. Davis, J. Lund, K. Jones, B.D. Jensen*, Brigham Young University, *J. Abbott, M. Harker, M. Zappe, S. Liddiard*, Moxtek

Carbon fiber composites are very high strength materials that could be enabling materials for micro and mesoscale applications. These materials have comparable strength to silicon but are much less brittle and can achieve four times higher strain. Several challenges must be overcome before carbon fiber composite devices can be fabricated on this scale. One challenge is the fabrication of ultra-thin wafers with low void density and low surface roughness. Another challenge is the ability to reliably machine the material into desired patterns. Here we present a method for curing carbon fiber wafers (~100 μm thick) with low surface roughness, low void density, a modulus of 50 GPa, and a yield strength of ~3.6 GPa. These wafers are suitable for laser machining into high fidelity micro and mesoscale structures. We will present laser micromachined devices made from these wafers including a series of high strength support structures for ultrathin membranes and a high-dynamic-range accelerometer.

4:00pm **LB+EM+GR+MN+TR-WeA7 Selective Graphitization using Multi-Ion Beam Lithography**, ***J. Fridmann***, Raith USA Inc., *S. Tongay*, University of California, Berkeley, *M. Lemaitre, A.F. Hebard, B. Gila*, University of Florida, *A. Nadzeyka*, Raith GmbH, Germany, *F. Ren, X. Wang*, University of Florida, *D.K. Venkatachalam, R.G. Elliman*, Australian National University, Australia, *B.R. Appleton*, University of Florida

Promising techniques for growing graphene on SiC single crystals for electronic device fabrication include heating in UHV above the graphitization temperature (T_G)[1]; or processing them in vacuum using pulsed excimer laser[2].

We report recent findings on the graphitization of SiC using a patterned Ga implantation, in which the implanted regions exhibit reduced T_G and enhanced graphitization above T_G. Here we report an approach that combines ion implantation, thermal or pulsed laser annealing (PLA), and multi-ion beam lithography (MIBL) to both pattern and synthesize graphene nanostructures on SiC single crystals at low temperatures. This approach utilizes a MIBL system developed at the University of Florida in collaboration with Raith for implantation/nanofabrication, in combination with thermal annealing in vacuum or PLA with a 25 ns pulsed ArF laser in air. To investigate the mechanisms and the effects of the implanted species, ion damage, and annealing, samples were also subjected to broad-area ion-implantations using facilities at the Australian National University.

It has recently been shown that implantation of Si, Ge, Au, or Cu followed by thermal annealing in vacuum below the T_G of SiC can selectively grow graphene only where the ions are implanted, and that graphene nanoribbons a few nanometers to microns wide can be formed using MIBL[3]. Additionally, we will show that graphene can be formed on implanted and/or unimplanted SiC by ArF PLA in air, at fluences from 0.4-1.2 J/cm^2. AES, SEM, X-sectional TEM, micro-Raman analyses and heat flow simulations are presented to verify graphene growth and explain the effects and mechanisms involved.

1. C. Berger, Z. Song, T. Li, X. Li, A. Y. Ogbazghi, R. Feng, Z. Dai, A. N. Marchenkov, E. H.Conrad, P. N. First, and W. A. de Heer, J. Phys. Chem. 108, 19912 (2004)

2. Sangwon Lee, Michael F. Toney, Wonhee Ko, Jason C. Randel, Hee Joon Jung, Ko Munakata,Jesse Lu, Theodore H. Geballe, Malcolm R. Beasley, Robert Sinclair, Hari C. Manoharan, and Alberto Salleo; ACS Nano Vol.4, No. 12, 7524-7530 (2010).

3. S. Tongay, M. Lemaitre, J. Fridmann, A. F. Hebard, B. P. Gila, and B. R. Appleton, Appl. Phys. Lett. 100, 073501 (2012).

4:20pm **LB+EM+GR+MN+TR-WeA8 Unrippling and Imaging of Extra-Large Free-Standing Graphene with Atomic Precision**, ***W.W. Pai***, *R. Breitweiser, Y.C. Hu, Y.C. Chao*, National Taiwan University, Taiwan, Republic of China, *Y.R. Tzeng*, Institute of Nuclear Energy Research of Taiwan, Republic of China, *L.J. Li*, Academia Sinica, Taiwan, Republic of China, *K.C. Lin*, Catholic Fu Jen University, Taiwan, Republic of China

Nanoscale ripple is believed to be a common feature most manifested in free-standing graphene and is expected to play an important role in altering the coupling of graphene's electronic and geometric structures. Direct characterization of free-standing graphene ripple is challenging from atom-resolved transmission electron microscopy (TEM) due to its limited depth resolution. Recent scanning tunneling microscopy (STM) of free-standing graphene uses small suspended area (1 or 5 microns) samples and can introduce uncontrolled tension that alters the intrinsic graphene structure. Here we report an STM study of suspended extra-large (~4000 micron^2) Cu CVD graphene that was prepared with a resist-free transfer and characterize its electromechanical response in details. In our study, a series of controlled "Z-V" spectroscopy were carefully conducted. In Z-V spectroscopy, the tip displacement vs. sample bias in close-loop condition is recorded. This gives hints on the nature of interaction forces and the mechanical response of graphene. In contrast to a solid surface, the graphene membrane is very compliant and Z-V curves are characterized by a fast-rise regime and a plateau regime that follows. Graphene deformation up to 100 nm with simply a small ~1 V bias ramp was observed. We discovered that our graphene is in best analogy with a curved rubber band that maintains quasi-static in shape until it is either pulled or pushed to tensile stress regimes. The graphene can be manipulated by the STM tip through electrostatic and van der Waals forces, with the latter being significant when it is repulsive. In its transit to tensile-stressed state, the graphene exhibits a series of sudden speed jump; we interpret these events as unrippling of graphene ripples and render support with molecular dynamics (MD) simulation. Atom-resolved graphene images provide direct evidence of nanoscale structure ripples in its intrinsic state and the smoothing out of such ripples in the tensile regimes. Surprisingly, on rippled monolayer graphene,

coexistence of triangular and hexagonal graphene lattices without tip condition change were observed. Our study provides a foundation to understand and control the electromechanical response of graphene (or other flexural atomic crystals) in its pristine two-dimensional form when subjected to a local proximal probe, therefore paves way to further investigate its structure-property correlation with atomic precision.

4:40pm **LB+EM+GR+MN+TR-WeA9 Ultrafast Charge Transfer at Monolayer Graphene Surfaces with Varied Substrate Coupling**, ***S. Lizzit***, ELETTRA Sincrotrone Trieste, Italy, *R. Larciprete,* CNR , Institute of Complex Systems, Italy, *P. Lacovig,* ELETTRA Sincrotrone Trieste, Italy, *K. Kostov,* Bulgarian Academy of Sciences, Bulgaria, *D. Menzel*, Technische Universität München and Fritz Haber Institute, Germany

The importance and scientific appeal of graphene monolayers (Gr) are out of question, and investigations of its electronic properties abound. Most of these center on the most spectacular region, that around the Dirac cone, which is also the most relevant region for possible devices. But regions outside of this region are also important, since their correct representation requires basic understanding, and since they may relate to applications in photonics, photochemistry, and contact formation. Also, static investigations are more frequent than those of dynamics.

We present here the first investigation of electron dynamics at energies above the Fermi (and Dirac) energy but below the vacuum level [1]. To this purpose we used the core hole clock (CHC) method with adsorbed argon and measured the transfer rate of a localized electron (the 4s electron on core-excited Ar) to the surface of Gr monolayers with variable substrate coupling: strong but graded coupling for Gr on Ru(0001) ("valleys" and "hills"), and decoupled Gr ML on SiO_2. We obtained the latter system by using the recently developed transfer-free approach [2] based on the synthesis of SiO_2 layers directly below Gr epitaxially grown on Ru(0001), through a stepwise reaction between intercalated silicon and oxygen. This method provides the optimal system to study the electronic properties of Gr using spectroscopic approaches, such as the CHC method.

We find strong variations of CT time between ~3 fs (Gr ML strongly coupled to substrate on Ru(0001) “valleys”) and ~ 16 fs (decoupled Gr on SiO_2). A ratio of 1.7 is found between the “hills” and “valleys” of the corrugated Gr/Ru. The very fast CT on Gr/Ru valleys is interpreted as due to hybridized Ru orbitals "reaching through" the Gr layer which change with the relative Gr/Ru alignment and distance. On the decoupled Gr layers the intrinsic coupling to the Gr empty π^* states determines the CT time. The intermediate CT time for the Gr hills on Ru shows that these regions are far from the "decoupled" condition. The results contribute new information on the still controversial states of Gr/Ru, and shed light on the empty density of states above Gr surfaces and the coupling to them in an energy range possibly important for photonic applications of Gr, such as solar energy conversion.

[1] S. Lizzit, R. Larciprete, P. Lacovig, K.L.Kostov, D. Menzel, *in preparation*

[2] S. Lizzit *et al*. Nanoletters (2012) DOI: 10.1021/nl301614j

5:00pm **LB+EM+GR+MN+TR-WeA10 Fano Interference Effects in Hydrogen Intercalated Graphene**, ***A. Boosalis***, *T. Hofmann,* University of Nebraska-Lincoln, *R. Elmquist, M. Real,* National Institute of Standards and Technology (NIST), *M. Schubert*, University of Nebraska-Lincoln

Graphene has been the focus of much recent research due to its unique electronic and optical properties, with potential for high performance electronics, tunable ultra-fast lasers, and transparent electrodes. Further development of graphene for commercial use requires effective large-area epitaxial production that maintains the desirable properties of exfoliated graphene. One such method of epitaxial graphene growth is thermal sublimation of Si from SiC. Sublimation of Si from the Si-face (0001) is the most controllable but produces a ($6\sqrt{3} \times 6\sqrt{3}$)R30° surface reconstructed layer prior to graphene formation. This layer can be altered by subsequent hydrogen intercalation, resulting in quasi-free-standing (QFS) epitaxial graphene.

In order to determine the effect of hydrogen intercalation on the optical properties of graphene we performed spectroscopic ellipsometry experiments in a spectral range of 3 to 9 eV before and after hydrogen intercalation of buffer layer only carbon growth on 6H SiC (0001). Spectroscopic ellipsometry is a widely used technique for determining the optical properties of thin films, and can provide sensitivity to film quality, morphology, and strain. In the case of graphene sensitivity is obtained through the critical-point (CP) located at 5.1 eV and modified by a Fano interference. Analysis of absorption near the CP is achieved through a parameterized model dielectric function (MDF) which is varied until a best-match between model and experimental data is obtained.

Best-match model results show drastic changes in the imaginary part of the MDF between previous measurements of buffer layer only growth on SiC, and buffer layer growth after hydrogen intercalation. Buffer layer only growth exhibits a far greater absorption throughout the spectrum, with an exciton produced maximum energy point that is shifted toward the infrared from the CP energy. After hydrogen intercalation, the QFS graphene layer exhibits a lowered absorption with a maximum closer to that of the CP energy; displaying an MDF closer to that of theoretical predictions for graphene.

In conclusion, hydrogen intercalation of buffer layer carbon growth on SiC (0001) has been shown to produce QFS graphene with optical properties closest to that of theoretical predictions for graphene, further proving its effectiveness as a tool for large-area epitaxial graphene production. In addition, buffer layer carbon growth shows optical properties sufficiently different from that of graphene to allow spectroscopic ellipsometry to become a viable in-situ monitor for commercial production of hydrogen intercalated graphene on SiC.

5:20pm **LB+EM+GR+MN+TR-WeA11 *In Situ* Dry-Cleaning of Ge(100) Surface using H_2O_2**, ***K. Kiantaj***, *T. Kaufman Osborn, T.J. Kent, A.C. Kummel*, University of California San Diego

Since Ge has higher hole and electron mobility compared to silicon, it is a good candidate for development of a new channel material in CMOS semiconductor devices. One of the obstacles in using Ge as a channel material is the high interface trap density between Ge and Ge native oxide. Air exposed Ge surfaces have a high density of defects and contaminants, but, in order to make optimal semiconductor devices, nearly perfect bonding between each unit cell and the gate oxide layer is required. Although there are many methods available for cleaning the Ge surface, the effectiveness of each of these methods highly depends on the cleanliness of the processing chambers. After cleaning, the Ge surface is typically functionalized with OH groups via water (H2O) or hydrogen peroxide (HOOH) during atomic layer deposition of the gate oxide. This OH functionalized surface ideally provides a high density of reactive sites for precursor nucleation. We have studied the effect of a very small amount of hydrocarbon in the processing chambers, and its effect on both the clean Ge surface and the OH functionalized surface since this may increase the density of interface traps and limit Equivalent Oxide Thickness (EOT) scaling. In-situ cleaned Ge surfaces as well as HOOH dosed surfaces have been studied after exposure to hydrocarbon contaminants with x- ray electron spectroscopy (XPS) and scanning tunneling microscopy (STM). An Argon ion source sputtering system was employed for in-situ cleaning of the Ge surface. After exposure to trace hydrocarbon contaminants, two different nanoscale features were observed by STM on the Ge and HOOH/Ge surfaces. One type of contamination denoted as carbon "nanoclusters" which are typically 0.3-0.5nm in height and 2-4nm in diameter. A distinctly different feature is observed on the Ge-OH terminated surface denoted as carbon "nanoflakes". In contrast to nanoclusters, nanoflakes were only observed on the Ge surfaces dosed with low concentration hydrogen peroxide. In the next step, a high concentration hydrogen peroxide source in combination with an ozone source was employed to study the removal of the contaminants from the Ge surface. Several dosing conditions and sample temperatures were studied and optimized. As the result, an atomically clean Ge surface were achieved by employing an all-dry in-situ process. The all-dry cleaning procedure does not involve any ion-milling or wet- cleaning procedures as both of these methods involve surface etching and result in surface roughness which is not desirable for semiconductor devices.

5:40pm **LB+EM+GR+MN+TR-WeA12 Hf-based High-k Dielectrics for Ge MOS Stacks**, *S. Fadida*, ***M. Eizenberg***, Technion Israel Institue of Technology, Israel, *L. Nyns, D. Lin, S. Van Elshocht, M. Caymax*, IMEC, Belgium

Ge has drawn much attention recently, being a leading candidate to serve as the channel material of future metal oxide field effect transistors (MOSFETs) due to its high carrier mobility with respect to Si. The interest in Ge is mostly because of its high hole mobility. Most of Ge related researches were focused so far on the challenge of Ge surface passivation. In this research we have moved on to the next challenge - finding a suitable high-k dielectric for a Ge-MOS stack. The high-k dielectric has to be chemically and thermally stable on top of the chosen passivation layer, have sufficiently high energy barriers with respect to Ge energy band edges, and have a large dielectric constant in order to obtain the required low effective oxide thickness (EOT). We have studied the chemical, structural and electrical properties of various Hf-based high-k dielectrics: HfO_2, $Hf_xZr_{1-x}O_2$, $Hf_xAl_{1-x}O_2$ and $Hf_xGd_{1-x}O_2$. All high-k dielectrics (4 nm thick) were deposited by atomic layer deposition (ALD) on top of a constant passivation stack composed of a thin GeO_2 layer (0.7 nm thick) followed by a thin (2 nm) ALD Al_2O_3 layer. The Al_2O_3 layer, which has high band offsets to Ge and GeO_2, was added since HfO_2, as many of the leading candidates for high-k dielectrics, are unstable on top of Ge or GeO_2. A thorough and systematic electrical and chemical characterization of this complex gate

stack was carried out. The interesting results show that this challenge of seeking for a superior high-k is not detached from the passivation challenge. Surprisingly, we have found that although the passivation stack was kept constant for all systems studied, the apparent D_{it} (density of interface states) changes when the top high-k material is modified. Another interesting phenomenon is revealed when different methods of D_{it} characterization are compared - each method points out a different high-k as the one with the lowest D_{it}. These observations imply that the C-V characteristics do not reflect only the role of Ge interface traps, but also of traps throughout the whole stack, at least to a distance of 2.7 nm (the total thickness of the passivation stack) from the Ge surface . These results emphasize even more the great challenges in integrating Ge as a new channel material. We have also analyzed the band alignment for all high-k dielectrics using XPS with respect to the underlying layers. All high-k dielectrics have similar band gaps at the range of 5.2-5.9 eV. The conductance and valence band offsets with respect to Ge are all larger than 1 eV, which make them all suitable for Ge-MOSFETs in terms of band alignment.

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