

Tuesday Morning, November 1, 2011

Plasma Science and Technology Division

Room: 202 - Session PS+MN+TF-TuM

Plasma Processing for Disruptive Technologies

Moderator: M.C.M. van de Sanden, Eindhoven University of Technology

8:00am **PS+MN+TF-TuM1 Scallop Free TSV Etching Method for 3-D LSI Integration**, *Y. Morikawa, T. Murayama, T. Sakuishi, S. Toyoda, K. Suu*, ULVAC, Inc., Japan **INVITED**

Thru silicon via (TSV) etch process for deep and high-aspect ratio structure has been studied thoroughly for applications such as MEMS and CMOS devices. Recently, TSV used in 3D-LSI devices for logic devices may be a few microns in diameter and about 50 μm deep. On the other hand, TSVs used in stacking memory devices, the via diameter and depth would be several tens of microns. Therefore, development of TSV etching process is very important for realizing these applications. In this study, a large via size etching in a high-pressure process was focused by using very high frequency capacitive coupled plasma (VHF-CCP) with an ultra self-confined system. This plasma system is simple parallel plate CCP about 100Pa or more process. High-pressure process was carried out on the plasma confined, because mean free path is very short. And, ion energy distribution (IED) is also controllable by high-pressure process with VHF bias. The bimodal IED changes under high-pressure. The peak of high-energy side is reduced, and a charge exchange peak appears. It is considered that the charge exchange is important to anisotropic Si etching of large size TSV with VHF bias.

And next, the high-density and small size of TSV below 10 μm diameter is indispensable to the utilization and improvement in high performance of 3D-LSI. We have developed a new etching system for TSV application for small size and high aspect ratio via. This system is a planer type magnetic neutral loop discharge (NLD) plasma. For high rate silicon etching, it is very important to understand not only the high density of the ICP plasma generation but also the high density of fluorine atoms. In this study, a novel RF antenna 'Multi Stacked rf Antenna' has been developed for highly accurate and high rate etching process. This antenna consists of multistage spiral turn rf antennas to reduce self-inductance (L). The L of this antenna is below 1.0 μH and it is lower than the standard spiral antenna. As a result of performing the electron density measurement of the planer NLD plasma using this MS antenna, it succeeded in the high-density plasma production of $1 \times 10^{12} / \text{cm}^3$ by the process pressure of 7 Pa. Next, the Si etching process development was performed using the advanced NLD etcher. As a result, the etching rate improved 4 times more compared to the standard cylindrical NLD plasma. Finally, the diameter of 2 μm was attained by the anisotropic etching of 5 $\mu\text{m}/\text{min}$, and the aspect ratio is above 10 using the planer NLD etcher. VHF CCP and planer NLD etching processes are non-cycle etch methods, and these processes were demonstrated about smooth sidewall TSV formation.

8:40am **PS+MN+TF-TuM3 Deep Silicon Etching of 0.8 μm to Hundreds of Microns Wide Trenches with the STiGer Process**, *T. Tillocher, W. Kafrouni*, GREMI, France, *J. Ladroue*, STMicroelectronics - GREMI, France, *P. Lefaucheur*, GREMI, France, *M. Boufnichel*, STMicroelectronics, France, *P. Ranson, R. Dussart*, GREMI, France

The STiGer process is designed to achieve high aspect ratio features in silicon. Like the Bosch process, passivation steps (SiF₄/O₂ plasmas) and etching steps are cycled to get vertical structures. The etching steps can be purely isotropic (SF₆ plasmas) or anisotropic (SF₆/O₂ plasmas). It is required to cool the silicon substrate with liquid nitrogen to form a SiO_xF_y passivation layer. It desorbs and disappears when the substrate is heated back to room temperature. Thus, there is no need to clean neither the microstructures nor the chamber walls after each process run. Then, the robustness of the process is enhanced in comparison with standard cryoetching: the profiles are less sensitive to temperature or flow rate variations. But, like in Bosch etching, a scalloping is present on the sidewalls.

Submicron trenches having critical aperture of about 0.8 μm can be etched with high aspect ratios (> 40). In these cases, the average etch rate is around 1.8 $\mu\text{m}/\text{min}$. These features exhibit both undercut and a special defect, which is called "extended scalloping". This defect 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

only for high aspect ratios (typically above 10). Thus, we will also investigate the role of trench critical dimension (from 0.8 μm to 100 μm). A mechanism explaining the formation of the extended scalloping will be proposed.

We have studied the influence of both the duty cycle (etch/(etch+passivation)) and the chamber pressure on the profiles and the extended scalloping. Basically, when the duty cycle increases, etching dominates passivation, which leads to higher defects. Pressure is a way to tune the slope of the sidewalls. Actually, decreasing the chamber pressure helps to shift from positively tapered features to more vertical profiles, and even negative slopes, hence with dovetailed shape.

This will be correlated with plasma analysis by means of mass spectrometry and optical emission spectroscopy. Actually, it is relevant to investigate how changes in the plasma chemistry can modify the trench profiles.

These trends have been used to optimize two methods that can help to reduce the extended scalloping. 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 SF₆ flow from a low value to the nominal value. Consequently, the process starts with a low etch rate and a more efficient passivation, which helps to limit the extended scalloping.

9:00am **PS+MN+TF-TuM4 Evaluation of Alternative Passivation Chemistries for TSV Applications**, *E.A. Joseph*, IBM T.J. Watson Research Center, *G. Matsuura*, ZEON Chemicals L.P., *S. Engelmann*, IBM T.J. Watson Research Center, *M. Nakamura*, ZEON Chemicals L.P., *N.C.M. Fuller*, *E.M. Sikorski*, *M. Gordon*, *B.N. To*, IBM T.J. Watson Research Center, *H. Matsumoto*, *A. Itou*, Zeon Corporation

With the current advent of 3D integration for advanced interconnect and packaging applications, there has been a renewed focus on deep silicon etch technology to satisfy the need for Through Silicon Via (TSV) patterning. The most common etch method used to fabricate said devices is a time-multiplexed (BoschTM) process, based on years of maturity in the MEMS field.[i] However, issues such as scalloping, mask undercut and limited etch rates are becoming more pronounced as feature sizes scale to meet the ITRS roadmap requirements. This has prompted efforts to attempt to either develop a more conventional etch process[i][ii] or to modify the Bosch process to circumvent these issues.[iii][iv] [v] In this work, we explore a novel polymerizing feedgas chemistry for the deposition step of the Bosch process to improve mask undercut while simultaneously increasing TSV etch rate. Initial results indicate a 5x larger deposition rate as compared to C4F8 (under nominal conditions) and under optimized conditions, enables a 50% decrease in undercut along with 10% increase in TSV etch rate. Optical emission spectra also differ substantially between the two feed gases, indicating different dissociation pathways and radical densities. Further results and a detailed characterization of the deposition properties of the novel chemistry will also be discussed leading to a proposed mechanism for the profile improvements as compared to C4F8. [i] B. Wu, A. Kumar and S. Pamarthy, J. of Applied Physics 108, 051101 (2010) [ii] I. Sakai, N. Sakurai and T. Ohiwa, J. Vac. Sci. Technol. A 29(2), Mar/Apr 2011 [iii] N. Ranganathan et al, Proceedings of the Electronics Components and Technology Conference, 2005 [iv] H. Rhee et al, J. Vac. Sci. Technol. B 27(1), Jan/Feb 2009 [v] S.-B. Jo et al, J. Vac. Sci. Technol. A 23(4), Jul/Aug 2005

9:20am **PS+MN+TF-TuM5 Wafer Scale Hermetic Packaging of MEMS**, *C.S. Gudeman*, IMT **INVITED**

The explosion of MEMS in automotive and cell phone markets has been enabled by low cost wafer level packaging (WLP) technology that provides a robust and hermetic enclosure for an otherwise delicate device. The more obvious advantage of WLP is greatly improved reliability, because the device is protected from organic and particulate contaminants while in the hands of the end user. A less obvious advantage is the protection provided by WLP during the manufacturing process, which often produces the highest levels of stress that a MEMS device experiences. These processes include wafer grinding, wafer dicing, and chip solder re-flow attachment to circuit boards and other chips. Firstly in this talk, wafer level packaging technologies will be outlined, focusing on the truly hermetic methods -- alloy, glass frit, Au-Au thermo-compression, anodic, and fusion bonding. Secondly the integration of Through Silicon Vias (TSV) with WLP will be discussed. Finally the performance of these technologies will be compared from a manufacturing perspective, including yield and thermal budget.

10:40am **PS+MN+TF-TuM9 Challenges in Plasma Etch for NVM: Scaling and Materials**, *M. Kiehlbauch*, Micron Technology, Inc.
INVITED

With advances in non-volatile memory, the major challenge confronting plasma etch is the introduction of new materials while simultaneously shrinking critical dimensions. This talk will address key development aspects including profile control, feature level uniformity, and plasma microdamage. Plasma microdamage is not the traditional, charge/voltage/current based impact to, for example, gate oxides. Rather, it is the changes to the atomic scale morphology in the sidewall or landing film of a plasma etch process. This results in a disruption of local stoichiometry, film defects, and other issues that impact device performance. The etch process and hardware changes to address this will be presented.

11:40am **PS+MN+TF-TuM12 Mechanisms of Selective Etching for Magnetic Materials: Ni, Co and Ta Etching by Carbon Monoxide/Methyl Alcohol Based Plasmas**, *K. Karahashi, T. Ito, S. Hamaguchi*, Osaka University, Japan

Dry etching of magnetic thin films is a crucial step in micro fabrication of magnetic random access memories (MARMs) and read/write heads for magnetic data storages. Argon (Ar) ion milling seems to be almost the only etching technique available in the current manufacturing processes. However Ar ion milling is incapable of achieving anisotropic and selective etching of magnetic films (Ni, Co etc.) over hardmasks (Ta etc.) and therefore highly selective reactive ion etching (RIE) of magnetic thin films is a highly sought-after technology. RIE processes based on CO/NH₃ or CH₃OH is a candidate for selective etching of magnetic thin films. In this study, we have examined etching processes of Ni, Co and Ta thin films by energetic CO⁺, O⁺ or OH⁺ ions, which are considered to be major etchants of CO/NH₃ or CH₃OH plasmas. We have determined the etching yields and analyzed surface reactions, using a mass-selected ion beam system. The ion beam system is designed to inject mono-energetic single-species ions into a sample surface in ultra-high vacuum conditions. The reaction chamber, where the sample is placed, is equipped with an X-ray photoelectron spectroscopy (XPS) for in-situ chemical analyses of irradiated surfaces. The ion beam energy used in this study is in the range of 150-1000 eV. The etching yields are determined from measured depth profiles of irradiated surfaces and ion fluxes. The etching yields of Ni and Co by CO⁺ ions are higher than that by O⁺ ions but lower than the yields of possible physical sputtering, which are estimated from interpolation of sputtering yield data of inert atom ions (He⁺, Ne⁺, Ar⁺, Kr⁺ etc.). From XPS analysis for O⁺ irradiated Ni and Co surfaces, oxidation is found to occur under O⁺ irradiation, which suggests that the oxide layer hinders sputtering by ion bombardment. It is found that little oxidation occurs on Ni or Co surfaces under CO⁺ ion irradiation and etching by CO⁺ ion bombardments proceeds. On a Ta surface, on the other hand, in the both cases of O⁺ and CO⁺ irradiations, oxidation occurs and its etching yield is far smaller than the yield of its possible physical sputtering. Therefore we have found that high selectivity of Ni and Co etching against hard masks (Ta, TaN) arises from the prevention of sputtering by mask oxidation. Etching characteristics by OH⁺ irradiation were also studied in a similar manner. This work was supported by the Semiconductor Technology Academic Research Center (STARC).

Tuesday Afternoon, November 1, 2011

Biofabrication and Novel Devices Focus Topic

Room: 105 - Session BN-TuA

Biofabrication Methods and Devices

Moderator: L. Gamble, University of Washington

2:00pm **BN-TuA1 Microengineered Hydrogels for Stem Cell Bioengineering and Tissue Regeneration**, A. Khademhosseini, Brigham and Women's Hospital, Harvard Medical School, MIT, and Harvard University **INVITED**

Micro- and nanoscale technologies are emerging as powerful tools for controlling the interaction between cells and their surroundings for biological studies, tissue engineering, and cell-based screening. In addition, hydrogel biomaterials have been increasingly used in various tissue engineering applications since they provide cells with a hydrated 3D microenvironment that mimics the native extracellular matrix. In our lab we have developed various approaches to merge microscale techniques with hydrogel biomaterials for directing stem cell differentiation and generating complex 3D tissues. In this talk, I will outline our work in controlling the cell-microenvironment interactions by using patterned hydrogels to direct the differentiation of stem cells. In addition, I will describe the fabrication and the use of microscale hydrogels for tissue engineering by using a 'bottom-up' and a 'top-down' approach. Top-down approaches for fabricating complex engineered tissues involve the use of miniaturization techniques to control cell-cell interactions or to recreate biomimetic microvascular networks within mesoscale hydrogels. Our group has also pioneered bottom-up approaches to generate tissues by the assembly of shape-controlled cell-laden microgels (i.e. tissue building blocks), that resemble functional tissue units. In this approach, microgels were fabricated and seeded with different cell types and induced to self assemble to generate 3D tissue structures with controlled microarchitecture and cell-cell interactions.

2:40pm **BN-TuA3 Nanoscale Architectures for Probing Cell Mechanics**, S. Wind, M. Schvartzman, M. Palma, M. Biggs, T. Fazio, R. Piqueras Jover, M. Sheetz, Columbia University

The physical properties of a cell's environment are important factors in determining cell behavior and ultimately, phenotype. Two key factors that have been associated with major changes in cell morphology and behavior are (1) spatial organization of extracellular matrix (ECM) molecules and (2) rigidity. In order to understand how cells sense these factors at the nanoscale and how these factors affect cell function, we have developed new nanofabricated surfaces in which these physical characteristics of the ECM are simulated.

The first type of surface combines nanoimprint lithography with selective biofunctionalization to precisely control the placement and geometric arrangement of integrin binding sites. The binding sites consist of sub-10 nm metallic nanodots functionalized with ECM binding ligands, designed so that each site can accommodate only a single integrin molecule. Cell spreading and motility assays were performed using 3T3 fibroblasts on arrays in which binding site spacing, density and number were independently varied. Cell spreading efficiency was markedly enhanced for clusters comprising at least 4 liganded sites spaced ≤ 60 nm apart, with little or no dependence on global density. This points to the existence of a minimal matrix adhesion unit defined in space and stoichiometry.

A second type of surface consists of elastomeric substrates with locally variable rigidity. We have found that exposure of poly(dimethylsiloxane) (PDMS) to an electron beam alters the rigidity of the elastomer, with the modulus of the exposed regions increasing with the applied electron dose. In addition to planar surfaces, pillared substrates can be patterned with no measurable change to the pillar dimensions. Immortalized mesenchymal stem cells plated on soft PDMS surfaces patterned in this manner displayed a distinct preference for the more rigid, exposed regions, forming focal adhesion nearly exclusively there. Furthermore, focal adhesion formation diminished significantly as the size of the exposed features was reduced below 1 μm , indicating that there is a length scale for cellular rigidity sensing, with the critical length in the range of a few hundred nanometers.

By adapting the tools of nanomanufacturing to cellular systems, we are able to define important parameters that can control aspects of cell function and behavior and will help identify conditions under which these functions may be altered. Potential applications range from therapeutic treatments that block metastasis to the development of new adoptive immunotherapies, as well as the development of new guidelines for the design of tissue scaffolds that can optimize healing without scarring.

3:00pm **BN-TuA4 Production of Functionalized 3D Micro Environment for Cell Culture**, J. Nowak, D. Mehn, P. Colpo, M. Zurn, T. Martin, F.J. Rossi, European Commission, JRC Institute for Health and Consumer Protection, Italy

One of the main challenges for the robust *in-vitro* studies is to obtain adaptable 3D culture systems that may mimic the tissue environment. Unfortunately the universal condition used in 2D cell culture techniques may hinder the full functionality of cells and generate misleading results.

Fabrication of firm and flexible micro-structures from organic polymers offers benefits for making smart 3D environments capable of driving cell behavior and surpassing the limitations of the 2D systems. These 3D bio-scaffolds can be employed to study various aspects of cell biology. Furthermore upon functionalization with the extra-cellular matrix proteins or signaling molecules they can be used as platforms for governing stem cell differentiation into the specialized cell types.

Here we present the straightforward approach to generate 3D bio-scaffolds that can facilitate cell growth under controlled geometrical and chemical conditions.

The technique involves UV cross-linking of the polymeric precursors to create the micro-well structures. The geometrical features of the structures are obtained by introducing a physical mask in contact with a liquid precursor, therefore restricting the region of the polymerization. We used PDMS mold as a physical mask to direct the polymerization of the PEG-DA and epoxy based polymers. However the technique can be used with various UV-sensitive polymeric materials.

The chemical and geometrical properties of the structures were characterized by XPS and microscopic techniques.

The features of the scaffolds lead to the development of a geometrically defined neuronal network when applied as platforms in a primary-neuron culture. Cell morphology and expression of the neuronal markers were characterized by fluorescent microscopy.

Vacuum Technology Division

Room: 111 - Session VT+MN+NS+SS+AS-TuA

Surface Science for Future Electronic Materials and Accelerator Applications

Moderator: M. Wüest, INFICON Ltd, Liechtenstein

2:00pm **VT+MN+NS+SS+AS-TuA1 New UHV Low Temperature Scanning Probe Microscopy Facility for the Study of Future Electronic Materials**, J.A. Stroscio, National Institute of Standards and Technology **INVITED**

Since the beginning of the last century new frontiers in physics have emerged when advances in instrumentation achieved lower experimental operating temperatures. Notable examples include the discovery of superconductivity and the integer and fractional quantum Hall effects. New experimental techniques are continually adapted in order to meet new experimental challenges. A case in point is scanning tunneling microscopy (STM) which has seen a wealth of new measurements emerge as cryogenic STM instruments have been developed in the last two decades. In this talk I describe the design, development and performance of a scanning probe microscopy facility operating at a base temperature of 10 mK in magnetic fields up to 15 T [1]. The STM system can be connected to, or disconnected from, a network of interconnected auxiliary UHV chambers used for sample and probe tip preparation. Results from current measurements on graphene and topological insulators will be described.

[1] *A 10 mK Scanning Probe Microscopy Facility*, Y. J. Song, A. F. Otte, V. Shvarts, Z. Zhao, Y. Kuk, S. R. Blankenship, A. Band, F. M. Hess, and J. A. Stroscio, *Rev. Sci. Instrum.* **81**, 121101 (2010).

2:40pm **VT+MN+NS+SS+AS-TuA3 Contact Resistance of RF MEMS at a Randomly Rough Surface in the Presence and Absence of Adsorbed Organic Monolayers**, D. Berman, J. Krim, M.J. Walker, North Carolina State University

Understanding of current flowing through the asperities is interesting for many applications: in RFMEMS, Molecular electronics, Nanotube tunneling etc.

Previous results [2] suggest that the films are displaced from the contacts themselves, but remain present in nearby regions. The increase in resistance is associated with elimination of vacuum electrical tunneling currents in

those regions. This raises the question of the relative proportions of contact resistance (R_c) and effective tunneling resistance (R_t).

Measurements on the gold on gold contacts adhered in the closed position, where the contamination film cannot possibly be placed inside the contacts are reported, to investigate vacuum tunneling current contributions to the total current at the contact. Electrical Contact Resistance measurements are reported for RF micro-electromechanical switches with Au/Au and Au/RuO₂ contacts, situated within an ultrahigh vacuum system equipped with *in situ* oxygen plasma cleaning capabilities. Fused Au/Au switch resistance increases by 3-5% (which corresponds to 20W tunneling resistance in parallel) after adding pentane to the switch environment. Moreover, the results are repeated with a different substrate (Ruthenium rather than Au), known for higher resistance, to change the resistance values with almost the same work function. If this is tunneling, the same effective tunneling resistance is expected, because tunneling depends on the work functions of the tip and substrate, which are close for gold and ruthenium oxide. In addition, the results are investigated for two different adsorbates, pentane and dodecane. Measurements have been recorded as the function of film coverage and the same tunneling resistance impact is observed. This is consistent with elimination of vacuum tunneling when adsorbed films are present.

Theoretical analysis of two possible mechanisms of the impact of molecular uptake is performed to interpret the experimental results: a) parallel connection of contact resistance and effective tunneling resistance before molecular adsorption, followed by molecules blocking the tunneling current; b) in series connection of contact resistance and pentane layer after adsorption. The data are more consistent with model a).

This work was supported by US National Science Foundation, AFOSR MURI and DARPA. We are grateful to C. Nordquist at Sandia National Lab and J. Hammond at RF Micro Devices for providing the experimental switches.

[1] D. Berman, M. Walker, C. Nordquist, J. Krim, *in preparation for Journal of Applied Physics*

[2] M. Walker, C. Nordquist, J. Krim, *in preparation for Tribology Letters...*

3:00pm VT+MN+NS+SS+AS-TuA4 Surface Issues for Solid Niobium SRF Accelerator Cavities, M. Kelley, College of William and Mary

The world-wide physics community looks forward to a slate of accelerator projects of unprecedented magnitude and diversity. Certainly its sheer size makes the International Linear Collider the most visible to the public eye, with 16,000 solid niobium cavities performing at historically high gradient, and built (and operated) for historically low unit cost. Net performance makes superconducting radiofrequency (SRF) technology the approach of choice.

Solid niobium is the material most widely used for construction of SRF cavities because it has the highest critical transition temperature ($T_c = 9.2$ K) of the pure metals, sufficiently high critical magnetic field ($H_c > 2$ k Oe) for SRF applications, and metallurgical properties adequate for fabrication and service load. Studies of the SRF performance of niobium cavities began to be reported more than 30 years ago and continue now with the application of improved experimental techniques. Niobium metal superconductivity is a nanoscale, near-surface phenomenon because of the shallow RF penetration. Considerable evidence indicates that cavity interior surface chemistry and topography strongly impact SRF accelerator performance, motivating investigation of how they are affected by post-fabrication treatments.

Current status and prospects are discussed with respect to accelerator needs and opportunities.

4:00pm VT+MN+NS+SS+AS-TuA7 Examples of Surface Related R&D on Nb Samples and SRF Cavities for Particle Accelerators at JLab, A.T. Wu, Thomas Jefferson National Accelerator Facility

This contribution will review some examples of surface related R&D on small and flat niobium (Nb) samples and single cell Nb superconducting radio frequency (SRF) cavities done at Jefferson Lab in the past few years. Most of the surface measurements were performed via the experimental systems available in the surface science lab that was set up¹ at JLab to study the various problems on the Nb surfaces in the SRF field.

The first topic is about a new Nb surface polished technique called buffered electropolishing (BEP) that was developed at JLab². This technique can produce the smoothest surface finish ever reported in the literature³. It was also demonstrated that under a suitable condition, a Nb removal rate higher than 10 $\mu\text{m}/\text{min}$ could be realized. Efforts have been made to try to understand the polishing mechanism through experiments with a well defined experimental geometry on small flat Nb samples. A unique versatile vertical polishing system was constructed to perform BEP on Nb single cell cavities. Small flat samples, Nb dumbbells and Nb single cell cavities were also studied and treated at CEA Saclay in France and Peking University in

China and the cavities were RF tested at JLab. Experimental results will be analyzed and summarized. It is showed that BEP is a very promising candidate for the next generation surface polishing technique for Nb SRF cavities.

A second topic will deal with a new Nb surface cleaning technique employed gas cluster ion beam (GCIB)⁴. This is a result of collaboration with Epion Corporation, Fermi Lab, and Argonne Lab. Beams of Ar, O₂, N₂, and NF₃ clusters with accelerating voltages up to 35 kV were employed in this technique to bombard on Nb surfaces. The treated surfaces of Nb flat samples were examined by several surface experimental systems such as SEM, EDX, AFM, SIMS, and 3-D profilometer. The experiments revealed that GCIB technique could not only modify surface morphology of Nb, but also change the surface oxide layer structure of Nb and reduce the number of field emission sites on the surface dramatically. Computer simulation via atomistic molecular dynamics and a phenomenological surface dynamics was employed to help understand the experimental results. A system was set-up at Epion Corporation to do treatments on Nb single cell cavities and then RF-tested at JLab. The experimental results will be summarized and the perspective of this technique for real applications is discussed.

Finally, I will show two typical examples of surface studies of Nb using a high resolution transmission electron microscope⁵ and a home-made scanning field emission microscope⁶ respectively.

4:20pm VT+MN+NS+SS+AS-TuA8 Early Stages of Nb Growth on Cu for SRF Accelerator Applications, C. Clavero, The College of William and Mary, N.P. Guisinger, Argonne National Laboratory, R.A. Lukaszew, The College of William and Mary

Among the large range of possible applications for superconducting Nb thin films, coatings for superconducting radio-frequency (SRF) cavities in linear accelerators have greatly aroused the interest of researchers in the last years[1]. Superconducting thin films and multilayer coatings are expected to increase further the maximum field gradients that SRF cavities can withstand, pushing them above 100 MeV/m [2]. In this regard, Nb coated Cu cavities have been proposed as a prototypical system for this purpose since they combine the better thermal stability of Cu due to its much higher thermal conductivity and the superconducting properties of Nb thin films [3]. Nevertheless, it is well known that structural dislocations and localized surface resistive defects on the thin films have a dramatically negative influence on their superconducting properties and resonator quality. Indeed, the quality of the films is strongly conditioned by the growth mode below the single atomic layer coverage at the very early stages of growth, and thus special attention needs to be devoted to this range. Here we present a complete study on the early stages of growth of Nb on Cu(111). Different growth and annealing temperatures ranging from room temperature (RT) to 600 °C were used in order to investigate the characteristic growth mode of Nb in the sub-monoatomic coverage range. Scanning tunneling microscopy (STM) and scanning tunneling spectroscopy (STS) were used to investigate morphology and chemical composition of the surfaces with atomic resolution. Growth of sub-monolayer coverages at RT leads to amorphous Nb islands with 1 and 2 AL heights. Annealing at 350 °C gives rise to crystallization of the islands pseudomorphically with the substrate, *i.e.* Nb(111). Further annealing at 600 °C promotes interdiffusion of Nb atoms into the Cu substrate and alloying of the islands. Growth of higher coverages above 1 AL at 350 °C reveals preferential Volmer-Weber growth mode.

1. H. Padamsee, Annual Review of Nuclear and Particle Science , 635 (1993).
2. A. Gurevich, Applied Physics Letters (1), 012511 (2006).
3. C. Benvenuti, S. Calatroni, I. E. Campisi, P. Darriulat, M. A. Peck, R. Russo and A. M. Valente, Physica C: Superconductivity (3-4), 153-188 (1999).

4:40pm VT+MN+NS+SS+AS-TuA9 Epitaxial Niobium Thin Films for Accelerator Cavities, W.M. Roach, D. Beringer, C. Clavero, College of William and Mary, C. Reece, Thomas Jefferson National Accelerator Facility, R.A. Lukaszew, College of William and Mary

The currently proven superconducting radio frequency (SRF) technology used in linear accelerators is based on bulk niobium cavities. Since this has a high cost and these cavities are approaching the maximum field gradients that they can withstand [1], development of a suitable, reliable, cost effective alternative to bulk niobium SRF cavities is needed. Attempts have been made to replace bulk niobium cavities with niobium-coated copper cavities since the thermal conductivity of a suitable base material such as copper is better than bulk niobium [2]. Coating niobium on SRF cavities is a promising but also challenging path, since there are several difficulties associated with various thin film deposition techniques and a lack of systematic studies pertinent to niobium thin film nucleation and growth leading to surfaces of greatest benefit.

Our systematic studies show that the transport properties, in particular the residual resistance ratio (RRR), are improved when niobium is epitaxially grown on crystalline ceramic substrates such as MgO and Al₂O₃, compared to niobium grown on (001) copper templates. Since grain boundaries are typically one of the main obstacles to superconducting transport, we show how the increased number of crystallographic domains that can occur during epitaxial niobium growth onto copper surfaces leading to higher density of grain boundaries can explain our results. We will discuss a route to improved transport properties while maintaining thermal efficiency by using alternative seed-layers grown on copper templates that can limit increased grain boundary density. We will show our correlated studies of microstructure and surface morphology (RHEED and AFM) and the resulting transport/magnetic properties (four point probe and SQUID magnetometry) illustrating possible mechanisms to improve SRF cavity performance of such niobium films.

This work is funded by HDTRA1-10-1-0072 from the Defense Threat Reduction Agency as well as a subcontract from Thomas Jefferson National Accelerator Facility under contract DE-AC05-06OR23177 from the Department of Energy as supplemented by ARRA funds.

References:

- [1] P. Kneisel *et al.*, Proceedings of 2005 Particle Accelerator Conference, Knoxville, TN, TPPT076 (2005).
- [2] S. Calatroni, *Physica C* **441**, 95 (2006).

5:00pm VT+MN+NS+SS+AS-TuA10 Development via Energetic Condensation of Niobium Thin Films Tailored for Superconducting RF Applications, A.-M. Valente-Feliciano, Jefferson Lab

For the past three decades, bulk niobium has been the material of choice for SRF cavities applications. In the recent years, RF cavities performances have approached the theoretical limit for bulk niobium. For further improvement of RF cavity performance for future accelerator projects, an interesting alternative has been recently proposed by Alex Gurevich with the Superconductor-Insulator-Superconductor multilayer approach, using the benefit of the higher critical field H_{c2} of higher-T_c superconductors without being limited with their lower H_{c1}.

JLab is pursuing this approach with the development of multilayer structures based on NbTiN via magnetron sputtering and High Power Impulse Magnetron Sputtering (HiPIMS). Insulators such as, AlN, Al₂O₃ and MgO are being investigated as candidates for the insulator layers.

This paper presents the characteristics of NbTiN and insulator layers produced and results on NbTiN-based multilayer structures on bulk Nb and thick Nb films.

5:20pm VT+MN+NS+SS+AS-TuA11 Evaluation of Secondary Electron Emission Yield Suppression Coatings at CEsrTA, Y. Li, X. Liu, J. Calvey, J. Conway, J.A. Crittenden, M.A. Palmer, J.P. Sikora, Cornell University, S.De. Santis, Lawrence Berkeley National Laboratory

The performance of particle accelerators may be significantly limited due to buildup of electron cloud (EC) in the vacuum chambers. The EC buildup intensity is strongly affected by secondary electron emission from interior surfaces of the chambers. Application of coatings with reduced secondary electron yield (SEY) onto vacuum chamber interior surfaces is one of the most economical EC suppression techniques. As a part of the International Linear Collider (ILC) R&D program, the Cornell Electron Storage Ring (CESR) has been successfully reconfigured as a Test Accelerator (CesrTA) to study EC buildup and suppression techniques. During the CesrTA program, various passive SEY-reduction coatings (TiN, amorphous-carbon and diamond-like carbon thin films) have been applied to diagnostic vacuum chambers in CESR in order to evaluate the efficacy of the EC suppression and the vacuum performance of these coatings in an accelerator environment. These chambers are equipped with both vacuum instrumentation (ion gauges and residual gas analyzers), as well as EC diagnostics (retarding field analyzers and RF-shielded pickups). In this paper, we present the results of studies of the vacuum conditioning and EC mitigation performance of these coatings.

5:40pm VT+MN+NS+SS+AS-TuA12 Electron Cloud Mitigation for the Large Hadron Collider (LHC), V. Baglin, G. Bregliozzi, P. Chiggiato, P. Costa Pinto, J.M. Jimenez, G. Lanza, M. Taborelli, C. Yin Vallgren, CERN, Switzerland

One of the main issues for the vacuum system of the Large Hadron Collider (LHC) is the build-up of electron clouds generated by electron multipacting

in presence of beams. The occurrence of spatially distributed negative charges can lead to beam instabilities and emittance blow-up, pressure rises with a consequent background growth in the experimental areas, and increased thermal load in the cryogenic sections. The development of electron clouds depends on beam intensity and structure, magnetic field, and, in particular, the secondary electron emission of the beam pipe walls. With respect to this latter point, electron clouds can be eradicated whenever the maximum secondary electron yield becomes lower than a critical threshold. In the LHC the problem has already been tackled at the design phase by introducing TiZrV non-evaporable getter thin film coatings as the baseline for most of the room temperature sectors of the ring. After activation by in situ heating, this material provides maximum secondary electron yield lower than 1.1. In addition, during operation, dedicated scrubbing runs are carried out by generating intentionally electron clouds and electron impingement onto the non-coated vacuum chambers, in a way to reduce their secondary electron yield. Recently magnetron sputtered carbon coatings have been also studied because they can reach exceptionally low secondary electron emission without any heating; their application in the LHC injectors and future LHC components is under investigation.

The effect of electron clouds in the pressure variations during the first months of LHC operation will be presented, together with the effects ascribed to the mitigation techniques.

Wednesday Morning, November 2, 2011

Graphene and Related Materials Focus Topic

Room: 208 - Session GR+MN-WeM

Graphene: Mechanical and Thermal Properties, Graphene MEMS and NEMS

Moderator: J. Rabe, Humboldt University Berlin, Germany

8:00am **GR+MN-WeM1 Graphene Atomic Membranes: From Patchwork Quilts to Atomic Drums, P.L. McEuen**, Cornell University
INVITED

Graphene is the world's first atomic membrane, a robust, one-atom thick freestanding layer of sp²-bonded carbon. The physical properties of these membranes straddle the border between soft and hard condensed matter. They are strong but highly flexible, with bending stiffness comparable to a lipid bilayer but stretching stiffness similar to diamond. Meter-scale polycrystalline graphene films can now be produced cheaply and easily, opening the door to applications in both science and technology. In this talk we will present new results on the structural and physical properties of this remarkable 2D material, including the first STEM images of graphene grain boundaries and the patchwork-quilt-like structure of graphene grains. We also discuss experiments on atomic drums made with graphene membranes that can be actuated and probed either electrically or optically. Unlike for traditional MEMS and NEMS, we find that stiction and entropy are key parameters in determining the drum's vibration frequency.

8:40am **GR+MN-WeM3 Nanomechanics of Graphene: Non-Linear Response, Fracture, and Crack Propagation, R. Perriot, Y. Lin**, University of South Florida, X. Gu, Aalto University School of Science and Technology, Finland, V.V. Zhakhovsky, I.I. Oleynik, University of South Florida

Recent nanoindentation experiments on graphene have revealed its exceptional strength, making it an excellent candidate for the design of nano devices such as MEMS and pressure sensors. Therefore, it is critical to understand the mechanical properties of graphene, and its response to a wide range of loading pressures beyond the elastic regime. In this work we performed molecular dynamics (MD) simulations of the nanoindentation of graphene membranes by a spherical indenter. The indentation curves (load vs. indentation depth) obtained from simulations revealed two regimes of response: linear for smaller, and non-linear for larger indentation depths respectively. The MD results are in good agreement with the theory of elastic plates and recent experiments. Using the kinetic theory of fracture, we were able to determine the breaking strength of graphene and its dependence on the indenter radius. MD simulations also provided an atomic-scale description of the breaking process, which occurs through crack formation and propagation in graphene.

9:00am **GR+MN-WeM4 The Effect of the Environment on Electrical and Mechanical Properties of Graphene, K. Bolotin**, Vanderbilt University

Every atom of graphene, a monolayer of graphite, belongs to the surface. Therefore, the environment of graphene -- the substrate onto which graphene is deposited and the coating on top of graphene -- intimately affects the properties of graphene. In this talk, we demonstrate that both mechanical and electrical properties of graphene can be tuned by varying the environment of graphene.

To study the mechanical properties of graphene, we developed a novel technique that is based on measuring the temperature-dependent deflection of a "bimetallic" cantilever composed of graphene and silicon nitride or gold layers. We demonstrate that the built-in strain, the substrate adhesion force and even the thermal expansion coefficient of graphene depend on the substrate under it.

To study the electrical properties of graphene in various environments, we developed a technique to fabricate electrically contacted multiterminal *suspended* graphene devices that are submerged into liquids. We explore the dependence of electron mobility of graphene on dielectric constant and ionic concentration of liquids surrounding graphene. We find that ions in liquids can cause strong scattering in graphene and that very large values of mobility (>40,000 cm²/Vs) can be achieved in ion-free liquids.

9:20am **GR+MN-WeM5 Nanoscale Friction and Adhesion Behavior of Graphene: The Effect of Sliding History, X.-Z. Liu, Q. Li, B. Zhang, R.W. Carpick**, University of Pennsylvania

As a prominent example of a two-dimensional (2-D) material, graphene has drawn much attention because of its extraordinary physical properties.

However, in contrast to its electronic and thermal properties, the mechanical and tribological properties of graphene remained poorly understood. These properties are interesting scientifically because of the extremely high strength and low defect density of the bonds, and the intrinsically wrinkled structure of graphene. These properties are important for integrating graphene with devices. We studied the friction and adhesion between nanoscale single-asperity tips and exfoliated graphene sheets using atomic force microscopy (AFM). We have previously reported that friction on few-layer graphene (and other 2-D materials) depends on the number of layers[1], whereby the friction is higher for fewer layers. The layer-dependence is associated with the presence of a "strengthening" effect, where the static friction force builds up as scanning proceeds, most evident for the thinnest layers. This suggested that the increase was due to the build-up of a puckered area in front of the tip due to the high compliance of the graphene and adhesion with the tip. However, adhesion between the tip and graphene, measured by regular AFM force-displacement spectroscopy, does not change appreciably when the layer number changes. This result agrees with trends obtained from finite element method (FEM) simulations. However, we observed that both friction and adhesion exhibit a contact history dependence. For friction on single layer graphene, the strengthening is not present initially, but rather, it gradually builds up as the tip is rastered over the surface. This suggests that the puckered structure requires repeated scanning before it geometrically develops to a point where friction is enhanced. In addition, we find that adhesion is enhanced if it is measured without breaking the tip-graphene contact after sliding the AFM tip over the same area for a sufficient distance. This sliding-history dependence was not observed on bulk graphite or SiO₂ substrates, and thus appears to be yet another unique feature of the tribological behavior of atomic sheets. These two observations strongly suggest that the geometric structure of the sheet and the contact area it makes with the tip is significantly affected by the sliding history.

[1] Lee, C., Q. Li, W. Kalb, X. Liu, H. Berger, R. Carpick, and J. Hone, *Frictional Characteristics of Atomically Thin Sheets*. Science, 2010. **328** (5974): p. 76.

10:40am **GR+MN-WeM9 Molecular Dynamics Simulations of Melting of Graphene, B. Steele, V.V. Zhakhovsky, R. Perriot, I.I. Oleynik**, University of South Florida

It has long been predicted by Peierls, Landau, and Mermin that infinite two-dimensional solids are unstable and should melt at any finite temperature. The stability of graphene, a two-dimensional layer of carbon atoms, is thus explained by the presence of an additional degree of freedom that allows it to buckle in the third direction normal to the 2-D perfect plane. We performed large-scale molecular dynamics (MD) simulations of graphene melting in order to provide a fundamental insight into the stability of graphene, as well as to investigate the nature of the defects naturally created by thermal excitations at high temperatures. We will discuss the types of defects appearing before melting, the atomic-scale mechanisms of melting, and the nature of the resulting carbon melt. In addition, the graphene melting in 3D space *versus* 2D-constrained melting is also discussed. The importance of a proper description of chemical bonding in graphene at high temperatures is illustrated using different interatomic potentials: the reactive bond order (REBO) potential and the newly developed screened environment dependent SED-REBO. Comparisons with other recent simulations of graphene melting (including those using the LCBOPII potential for carbon) are also presented.

11:00am **GR+MN-WeM10 Inhomogeneous Strain in Monolayer Epitaxial Graphene, D.A. Schmidt**, Ruhr-University Bochum, Germany, T. Ohta, L.B. Biedermann, T.E. Beechem, S.W. Howell, G.L. Kellogg, Sandia National Laboratories

We report a large in-plane compressive strain (up to 0.5%) and its inhomogeneous variation at micrometer length scale in single layer graphene films on silicon-carbide (SiC) (0001). The strain, due to the difference in lattice constants and thermal expansion coefficients of graphene and SiC substrate, is probed using Raman scattering. We show that both the growth mechanism and the relaxation along the mismatched symmetry of the graphene and underlying substrate can affect the exact amount of local strain. The large compressive strain implies that monolayer graphene is tightly grafted to the underlying interface layer and SiC substrate; otherwise it would delaminate to relieve the strain. The magnitudes of the structural strain and its local variation are significant and

need to be taken into account for electronics applications based on the graphene-SiC(0001) system.

The Raman microscope was supported under BMBF grants 05KS7PC2. D. A. S acknowledges support within the BMBF funded projects 05KS7PC2 and 05K10PCA. The work was also supported in part by the LDRD program at SNL and the US DOE Office of Basic Energy Sciences' Division of Materials Science and Engineering (Contract No. DE-AC04-94AL85000). Part of this work was performed at CINT (Contract No. DE-AC04-94AL85000). SNL is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Thursday Afternoon, November 3, 2011

MEMS and NEMS Group

Room: 105 - Session MN-ThA

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

Moderator: A.V. Sumant, Center for Nanoscale Materials, Argonne National Laboratory

2:00pm MN-ThA1 Heterogeneous Microsystem Integration with Self-Assembly, *K. Bohringer*, University of Washington **INVITED**

Self-assembly is the spontaneous and reversible organization of components into ordered structures, representing an alternative to the conventional manufacture of systems made of components from milli to nano scales. First commercial applications of self-assembly have appeared in recent years, for example in the fabrication of radio frequency identification (RFID) tags.

However, the full impact of this new approach towards hetero system integration will only be realized once self-assembly can be programmed on demand. This presentation gives an overview of several projects that aim at programmable self-assembly. A key concept is the ³programmable surface² an interface whose properties can be controlled with high spatial and temporal resolution. Several crucial topics are discussed: real time control of interfacial properties; optimization of binding site designs; and algorithms for the modeling and control of self-assembly. Promising novel manufacturing methods are emerging that combine the precision and reproducibility of semiconductor fabrication with the scalability and parallelism of stochastic self-assembly and with the specificity and programmability of biochemical processes.

2:40pm MN-ThA3 A Study of Solder Bridging for the Purpose of Assembling Three Dimensional Structures, *M.R. Rao, J.C. Luths, S.L. Burkett*, The University of Alabama

Recently, dip soldering has been used as a mechanism for driving the assembly of three-dimensional (3D), microscale structures. Solder is deposited on adjacent metallic faces of planar polyhedral patterns, bridging the small gaps between individual faces. When all but one face of a polyhedral pattern are freed from the substrate and solder is reheated to a

liquid state (reflow), the free faces of the pattern fold upwards, out of the plane, to form the desired polyhedron. The wetting of solder with regards to coverage of metallic faces has been described previously, but the lateral bridging between the metal faces remains relatively unexplored. The goal of this work is to characterize the parameters influencing the bridging and folding process for two different ways of dip-soldering: face and edge soldering. Face soldering refers to the complete wetting of metal faces while edge soldering refers to selectively applying solder on the edges of a face that come in contact with other faces when folded. Our work explores bridging yield for various gap sizes and face thicknesses for eight different polyhedral patterns. Experiments show that the thickness and gap size strongly influence successful bridging. Experiments also show that improved control over the bridging process increases the yield of folded structures. In particular, gap size is positively correlated to face thickness for successful folding. Moreover, face soldering results in higher yields than edge soldering for all patterns.

3:00pm MN-ThA4 Fabricating Arrays of Graphene Nanomechanical Resonators with High, Size-Dependent Quality Factors, *R.A. Barton, A.M. van der Zande, R.B. Ilic, C.S. Ruiz-Vargas, J.S. Alden, W.S. Whitney, J. Park, P.L. McEuen, J.M. Parpia, H.G. Craighead*, Cornell University

Graphene's unparalleled strength, stiffness, and low mass per unit area make it an ideal material for nanoelectromechanical systems (NEMS), but graphene resonators have been challenging to fabricate in large numbers and have exhibited poor quality factor. Here, we present simple methods of fabricating large arrays of graphene resonators from CVD-grown graphene and discuss their properties. We focus on circular graphene resonators with diameter of up to 30 microns, for which we observe highly reproducible resonance frequencies and mode shapes, as well as a striking improvement in the membrane quality factor with increasing size. The largest graphene resonators display quality factors as high as 2400 ± 300 , about an order of magnitude greater than previously observed quality factors for monolayer graphene. These measurements shed light on the mechanisms behind dissipation in monolayer graphene resonators and demonstrate that the quality factor of graphene resonators relative to their thickness is high compared to nanomechanical resonators demonstrated to date. We conclude by providing an outlook for graphene NEMS and their applications.

3:40pm MN-ThA6 Modal Dependence of Dissipation in Ultra Thin Silicon Nitride Drum Resonators, *V.P. Adiga, R.B. Ilic, R.A. Barton*, Cornell University, *I. Wilson-Rae*, Technische Universität München, Germany, *H.G. Craighead, J.M. Parpia*, Cornell University

We have fabricated up to 1 mm diameter high tensile stress (1.2 GPa) circular SiN membranes. Stoichiometric amorphous high tensile stress SiN is a useful material for nanomechanical devices and resonators made from it have shown extremely high Q ($> 1,000,000$) at room temperature.¹ We used both optical and electron beam lithography to define circular structures and measured their resonant frequency and Q using optical interferometric detection methods. The measured mechanical Q shows a strong modal dependence, indicating the influence of clamping losses. Azimuthal harmonics of circular resonators with diameter $s > 200$ nm show an exponential drop in dissipation within an individual modal family ($n = 1, 2, 3, \dots, m$) apparently due to the destructive interference between the waves radiated by adjacent sections of periphery.² However, still higher order modes of large resonators and modes of smaller resonators are strongly influenced by a characteristic fQ limit of 2×10^{13} possibly indicating the presence of intrinsic dissipation in the high frequency limit. These findings pave the way for identifying optimum high Q modes of stressed oscillators for applications in mass sensing and fundamental research in optomechanics.

1) D. Southworth et al, PRL, 2009

2) I. Wilson-Rae et al PRL, 2011

4:00pm MN-ThA7 Stress-based Flammable Gas Sensing with Nanocoated Resonant Microbridge at Critically-Buckled State, *D.J. Joe, Y. Linzon, V.P. Adiga, R.A. Barton, M. Kim, B. Ilic*, Cornell University, *S. Krylov*, Tel Aviv University, Israel, *J.M. Parpia, H.G. Craighead*, Cornell University

In this work we demonstrate robust flammable gas sensing using stress-based resonant microelectromechanical systems (MEMS) bridges at ambient pressure and temperature. In contrast to previously reported works, which were based on either measuring a static deflection or changes in frequency due to added mass, we report a method of tracking shifts in resonant frequency of microbridges in real time due to alteration of stress from swelling of a reactive polymer coating near the Euler buckling configuration. Experimental results clearly demonstrate that the suggested approach is efficient for selectively sensing trace vapor. We show projected vapor content sensitivity as low as ~ 13.4 ppm for ethanol vapor in low concentration regime, and demonstrate actualized proven sensitivity of less than 1 part per thousand, with a few seconds response time for the functionalized microbridge.

4:20pm MN-ThA8 Rapid Serial Prototyping of Magnet-Tipped Attonewton-Sensitivity Cantilevers, *J.G. Longenecker, E.W. Moore, J.A. Marohn*, Cornell University

There is a critical need for a technique capable of non-invasive high resolution imaging of single copies of delicate biomolecules and as-fabricated semiconductor and spintronics devices. Magnetic resonance force microscopy (MRFM) is a non-invasive, three-dimensional imaging technique that employs attonewton-sensitivity cantilevers to mechanically detect electron spin resonance [1] and nuclear magnetic resonance [2]. The recent demonstration of 4 nm resolution imaging of a virus using MRFM establishes that the technique can achieve single-particle imaging with resolution competitive with cryo-electron microscopy [2]. The sample-on-cantilever geometry used in the experiment of Ref. 2, however, requires small, robust samples and is inapplicable to as-fabricated devices. We propose to image semiconductor devices by instead affixing to the cantilever the submicron magnetic particle required to achieve high spin sensitivity and spatial resolution. To minimize surface dissipation and achieve high signal to noise, the magnet must overhang the leading edge of the cantilever [3]. We recently demonstrated an approach to fabricating cantilevers with such integrated overhanging nanomagnets that achieves high yield [4]. Moreover, the novel tip fabrication method enabled the prototyping of new tip designs in less than sixteen hours of processing time [4], compared to the more than two weeks of processing time required for the best previous method [3].

Here we report harnessing this rapid prototyping technique to fabricate and characterize nickel and cobalt-iron-boron (CoFeB) nanorods. All nanomagnets are defined using electron beam lithography. The nickel nanorods are evaporated followed by liftoff, whereas the CoFeB nanorods are deposited by conformal sputtering and patterned by ion milling. The magnetic properties of the nanomagnets are determined using frequency-

shift cantilever magnetometry and superconducting quantum interference device measurements. The elemental composition – paying particular attention to the extent of surface damage – is determined by scanning transmission electron spectroscopy and electron energy loss spectroscopy. We will detail work to develop a protocol for improved encasement of nanorods overhanging the cantilever leading edge to protect against damage, as well as our progress in implementing the nanomagnet-tipped cantilevers in MRFM experiments to rapidly detect single electron spins.

- [1] EW Moore *et al.*, *Proc. Natl. Acad. Sci.* **106**(52), 22251 (2009).
- [2] C Degen *et al.*, *Proc. Natl. Acad. Sci.* **106**(5), 1313 (2009).
- [3] SA Hickman *et al.*, *ACS Nano* **4**(12), 7141 (2010).
- [4] JG Longenecker *et al.*, *J. Vac. Sci. Technol. B*, in press.

4:40pm **MN-ThA9 Microfabrication of On-Chip Electrodeposited CoNiP Micromagnets and Integration into MEMS Sensors**, *D. Schreiber, O. Berkh, S. Krylov, Y. Shacham-Diamand*, Tel Aviv University, Israel

Motion sensing of microelectromechanical systems (MEMS) devices is often a problem due to limited available chip footprint. On-chip thin film hard magnetic materials, when used as elements of integrated induced current displacement sensors, can help significantly simplify the designs and reduce device footprint due to the relatively high field density attainable. Device element spacing can also be increased thereby reducing fabrication tolerance requirements and improving robustness.

Electrochemical deposition is an attractive method for batch processing of magnetic films in patterned structures. Electroplating is a relatively simple process with a wide variability and control of film thickness and good scalability and compatibility with most of the MEMS microfabrication processes. Additionally, electrochemical deposition allows for the controlling of magnetic film anisotropy a key factor for the design of devices that operate in-plane or out-of-plane. Interest has been shown in CoNiP thin films for use in a number of MEMS applications however, the issues of integration were not addressed.

The integration of CoNiP magnetic films into MEMS sensors was studied. Through-mask electrodeposition of 1-2 μm thick magnetic films from concentrated ammonium chloride electrolyte was carried out at current densities of 30-150 mA/cm² using both direct current and pulse plating modes. The effects of current density, seed layer, passivation layer, pattern size and geometry on magnetic properties and feature-scale thickness distribution were investigated. Geometries included various arrays of micron scale stripes and dots, and large 1-4 mm² square areas. Feature scale profiles and magnetic properties of the films are influenced by current density as well as by feature size and geometry. Magnetic properties of CoNiP films after post-electrodeposition processing remain in the range suitable for sensor operation and are therefore shown to be suitable for integration in MEMS sensor.

Micropatterned CoNiP magnetic thin films have been integrated into silicon-on-insulator (SOI) MEMS devices. The patterned micromagnets – large square areas, stripes and dots – were characterized for feature-scale thickness distribution in relation to pattern geometry and current density, the effects on magnetic properties due to post-electrodeposition processing and their compatibility with standard MEMS process chemicals. Thickness distribution is strongly correlated with pattern geometry and current density. Magnetic properties remain in a range suitable for integration into MEMS devices following post-electrodeposition fabrication processes such as lithography, sputtering and etching.

5:00pm **MN-ThA10 Towards an Integrated Nano-optomechanical Platform for Molecular Sensing and Magnetometry**, *W.K. Hiebert, Z. Diao, J.N. Westwood, V.T.K. Sauer, M.R. Freeman*, National Institute for Nanotechnology (NRC Canada) and University of Alberta, Canada

Nano-electromechanical systems (NEMS) have exquisite potential in fields ranging from quantum measurement to ultrasensitive mass sensing. Signal transduction has remained an important challenge for NEMS where applications demand fast, parallel, sensitive, and low-noise drive and detection of motion in ever smaller and faster devices. The burgeoning field of nano-optomechanical systems (NOMS) has offered a promising solution to this challenge in the form of unprecedented displacement sensitivity with almost unlimited bandwidth. Nanophotonic circuits provide strong local concentration of optical forces and optical phase changes interacting with embedded NEMS devices. The combination is fully integratable with modern opto-electronic and semiconductor technology paving the way to large-scale-integrated lab-on-a-chip NEMS sensing arrays.

We will present our preliminary efforts in building an integrated NOMS platform for molecular sensing and for magnetometry applications. The results include a novel measurement geometry that allows accessing nanophotonic NOMS chips in vacuum via free-space focusing onto grating couplers. The external-to-vacuum optics arrangement gives independent control over the position and input/output angles of both the input and output laser beams. This geometry allows us to directly compare photonic readout of NEMS motions with conventional free-space Fabry-Perot interferometry. Finally, we will update our progress in 3D integration of NEMS and photonics.

Friday Morning, November 4, 2011

MEMS and NEMS Group
Room: 105 - Session MN-FrM

Characterization of Materials and Structures at the Micro- and Nano-scale

Moderator: M. Metzler, Cornell University

8:20am **MN-FrM1 Nanomechanics: Controlling Near-Field Interactions between Mechanical Systems**, *D. Lopez*, Argonne National Laboratory **INVITED**

Metallic and dielectric objects are surrounded by fluctuating electromagnetic fields due to thermal and quantum fluctuations of the charge and current density at the surface of the bodies. Immediately outside the objects, this electromagnetic field exists partly in the form of propagating electromagnetic waves and partly in the form of evanescent waves that decay exponentially with distance away from the body's surface. These fluctuating electromagnetic modes are responsible for a great variety of near-field phenomena such as the Van der Waals force, the Casimir force, near-field heat transfer, and non-contact friction forces. As devices evolve from micro- to nanoscale structures, these forces become relatively stronger, and their effect cannot be disregarded any further. For example, researchers working to develop NEMS devices need to consider the effects caused by Van der Waals and Casimir forces which can lead to compromise in the range of motion or in the voltages required for actuation. To improve our understanding of these near-field interactions and to develop mechanisms to control them is extremely important for a diversity of seemingly different fields, such as nanomechanics, quantum computing with trapped ions, measurements of gravitational forces at the nanometer scale, and detection of single spins for magnetic resonance force microscopy.

In this presentation I will describe the fundamentals of near-field forces, I will review recent scientific advances regarding manipulation of these interactions in the field of nanomechanics, and I will illustrate novel applications that could be enabled once we are capable of control these forces.

9:00am **MN-FrM3 Pull-in Experiments on Electrostatically Actuated Microfabricated Meso Scale Beams**, *Y. Gerson, I. Sokolov*, Tel Aviv University, Israel, *T. Nachmias*, RAFAEL LTD, Israel, *S. Lulinsky, S. Krylov*, Tel Aviv University, Israel

Meso scale (hundreds of micrometers to several millimeters) MEMS sensors and actuators are beneficial in applications where large displacements, manufacturability, and ease of integration with existing mechanical and packaging environments are required.

In this work we report on the results of characterization and modeling of electrostatically actuated meso scale beams. The beams with clamped ends were 5000 μm long, 150 μm thick and 10, 12 and 15 μm wide and were operated by a parallel plate electrode located at the distance of 20 μm from the beam. The goal of the work was twofold. First, we demonstrate the feasibility of electrostatic actuation of the meso scale devices and ability to achieve relatively large displacement. Second, an electrostatically actuated double clamped micro beam is viewed as a kind of benchmark problem and was intensively studied. However, the number of reported experimental results, which can serve for validation of models, is limited. We anticipate that our experimental results, obtained using larger meso scale structures and therefore relatively more accurate, could provide a reliable experimental reference for a double clamped beam actuated by a parallel-plate electrode.

The devices were fabricated by deep reactive ion etching (DRIE)-based process from highly doped Si using a silicon on insulator (SOI) wafer with [111] surface orientation and 150 μm thick device layer. The experimental approach based on the use of SOI wafers allows to fabricate devices with low residual stress and excellent mechanical properties of Si. The devices were operated in ambient air conditions. Linearly increasing (ramp) voltages were applied quasistatically to the actuation electrode and easily visualized in-plane (parallel to the wafer surface) motion of the devices was registered using an optical microscope and a CCD camera. The response was video recorded, the movie was split into separate frames and the voltage-displacement dependence was built using customized edge detection image processing procedure implemented in Matlab. The critical pull-in voltage varied between 70 V in (nominally) 10 mm wide beam and up to 125 V in 15 mm wide beams. In addition, pull-in behavior of the beams was modeled using several approaches, starting from simplified

reduced order models based on the Galerkin decomposition with linear eigenmodes as base functions and up to fully coupled nonlinear large deflection three-dimensional simulations. The actual dimensions of each beam, carefully measured using scanning electron microscope (SEM) were used in calculations. Excellent agreement between the results provided by the model and the experimental data was observed.

9:20am **MN-FrM4 Absorption and Emission of Plasmonic Antenna Arrays**, *K.E. O'Brien, P.H. Holloway, M.R. Davidson*, University of Florida

New and more portable means of generating narrow band radiation are of interest, especially in the terahertz (THz) range. One potential method for generating radiation involves photo-mixing over nano/micro scale plasmonic structures. The plasmonic structures can serve as antennas for absorbing incoming photons and conversely emit radiation of a lower frequency. Designs include 2-dimensional arrays of these resonant structures fabricated on Ag thin films using electron-beam lithography and lift-off. Patterns vary from arrays of linear structures, "bowties," and interlocking structures. We have shown emission of visible radiation from similar structures when excited by space charge from electrons. The absorption and emission of light by the structures has been measured for micron-scale and nano-scale antenna arrays and has exhibited a polarization dependent behavior. The effect of different antenna structures on the absorption and emission will be discussed.

10:00am **MN-FrM6 Fabrication and Characterization of Structural and Electrical Properties of Ultrananocrystalline Diamond Nanowires**, *X. Wang*, University of Puerto Rico, *A.V. Sumant, V. Joshi, L.E. Ocola, B. Kabius, D. Lopez*, Argonne National Laboratory

Due to extraordinary mechanical, optical and electrical properties as predicated by theory, there has been tremendous amount of interest in making diamond nanowires (DNWs) and diamond nano-rods (DNRs). Synthesizing or fabricating these nanostructures is proving to be very challenging. To date, only a few attempts have been reported, either by etching single crystal diamond using focus ion beam (FIB) to produce diamond NRs or by coating Si nanowires with nanocrystalline diamond to produce diamond NWs. We report a top-down method based on e-beam lithography and reactive ion etching of ultrananocrystalline diamond (UNCD) to produce UNCD nanowires (UNCDNWs) with nanowire diameters as small as 30 nm. Since they are produced by lithographic approach (top-down), they can be fabricated at well-defined position with nanometer-scale precision. Compare to other fabrication techniques like FIB, our UNCDNWs maintain intrinsic diamond structure and properties without degradation after fabrication process, which has been confirmed by Raman spectroscopy (ultraviolet and visible), transmission electron microscope (TEM) and electron energy loss spectroscopy (EELS). Preliminary electrical measurement of UNCDNWs will be discussed. The ability to fabricate UNCDNWs provides an opportunity to study the fundamental mechanism of transport processes in UNCDNWs, which will enable new ideas and possibilities for the fabrication of new functional nanoelectronic devices.

10:20am **MN-FrM7 Investigation of Heat Transfer Enhancement in Nanofluids with Molecular Dynamics Simulations – Role of Particle Charge and Fluid Polarity**, *J.D. Schall*, Oakland University, *A.S. Comfort*, U.S. Army RDECOM-TARDEC

Thermal loads are increasing in military vehicles because of the greater use of microelectronics,

higher power density engines, and restricted air flow from up-armor kits. Conventional methods

to increase heat dissipation, such as increasing heat exchanger size produce an undesired

increase in vehicle weight and packaging issues. One approach to mitigate these issues is the

development of heat transfer fluids with improved thermal transport properties. Nanofluids are

suspension of nanometer sized particles in solvent, and represent a potential method to increase

the effective fluid thermal conductivity and heat transfer coefficient of coolants without creating

the adverse effects found in larger particle suspensions, such as settling, clogging, and abrasion.

Since their introduction by U.S. Choi in 1995, a great deal of uncertainty about the mechanisms

of enhanced thermal conductivity of nanofluids continues to employ researchers and limits

the development of optimized nanofluids in heat transfer applications. In this paper, molecular

dynamics simulations are used to investigate heat conduction between model particle surfaces

separated by a liquid layer. In particular, effects of base fluid charge, polarity, and nanoparticle

surface charge on the solid-liquid interface liquid structure, thermal (i.e. Kapitza) resistance, and

thermal conductivity are investigated. Results are compared with previous simulations from the

literature which used simple monoatomic models interacting through Lennard-Jones potentials.

10:40am **MN-FrM8 Novel CMOS MEMS Double Parallel Plate Capacitive Tactile Sensors For Blood Flow Monitoring, C.J. Hsieh, J.C. Liou, C.T. Sun, Y.C. Lin, W.-C. Tian, National Taiwan University**

This research focuses on the developments and characterizations of non-invasive tactile blood flow sensors using CMOS MEMS technologies. The capacitive sensing structure consists of two parallel plate capacitors which can be connected in different configurations in case with different measuring ranges. Sensor detection scope is set to be from 0 to 150 mmHg according to the estimated maximum human vessel pressure. The sensor is fabricated in commercial 2 polysilicon and 4 metal CMOS technology followed by the self-developed post processes. The dimension of each sensor is 400 μ m in length with the membrane thickness of 1.45 μ m.

An anisotropic inter metal dielectric layer etch step was utilized on CMOS chips to open wet metal etching holes. After this dielectric layer etch, a metal wet etching process was applied to release sensing structures. In order to protect the metal bonding pads in post CMOS MEMS processes, an Au layer was deposited on the pad areas. Based on the experiment results, the lateral metal sacrificial layer etching rate is 1.85 μ m per minute and the lateral etching rate underneath gold layer is 2.9 μ m per minute. We have successfully demonstrated the post CMOS MEMS processes for our sensors.

Initial finite element method analysis results showed that the sensitivities of two different designs are 6.7 and 2.2 fF per mmHg with a dynamic range of 75 and 200 mmHg. The sensor behavior measurement data will be presented.

11:00am **MN-FrM9 A Highly Sensitive Nanomachined TiO₂ Gas Sensor for Micro Gas Chromatography, C.H. Chou, C.H. Chen, W.-C. Tian, National Taiwan University, T.H. Chan, C.-J. Lu, National Taiwan Normal University**

The purpose of this study is to develop a sensitive gas sensor with engineered TiO₂ nanostructures using semiconductor nanotechnologies for micro gas chromatography. Many TiO₂ nanowires for gas sensing nowadays were fabricated by chemical synthesis methods, and the nanowire arrays are in irregular formats and the amount of sensing material may be varied chip to chip. The behavior of sensing repeatability of these TiO₂ nanowires using conventional methods is hard to control.

With the combination of E-beam lithography and the TiO₂ thin film deposition, the TiO₂ sensing nanowire arrays with well-controlled structures (100-300 nm wide with 1 μ m period), were placed in between the Au interdigitated electrodes. A microheater were fabricated by deposition of the 3/50 nm thick Cr/Au films on the backside of the sensor. The great linear heating with increasing input power and uniform heating (329.3 $^{\circ}$ C in average, STDV of 9.3 $^{\circ}$ C, power of ~0.8 W) were obtained through an IR camera.

The performance of nanowire detector (100 nm wide, 183.5 M Ω) is compared to the microwire detector (20 μ m wide, 24.6 M Ω) at various ethanol and benzene concentrations or at various operation temperatures. The measured resistance to the initial resistance ratio of the nanowire detector changed from 1 to 0.35 at 284 $^{\circ}$ C at 6.5% ethanol concentration. The effects of the rapid thermal annealing and an O₂ plasma treatment to improve the sensor performances is investigated and will be presented.

11:20am **MN-FrM10 Ultra-high Aspect Ratio High-speed Silicon Nanowire and Three-dimensional Formation Using a Hydrogen-assisted Deep Reactive Ion Etching, Z. Sanaee, S. Azimi, M. Poudineh, S. Mohajerzadeh, A. Sandoughsaz, University of Tehran, Iran**

We report the formation of ultra-high aspect ratio and three dimensional features on silicon substrates using a novel low-density capacitive-coupled plasma reactive ion etching (13.56MHz). The etching process is based on using three gases of hydrogen/oxygen and SF₆ in two sub-sequences called

as passivation and etching sub-cycles. All three gases are used in the passivation step and SF₆ in the etching step. Unlike Bosch process no polymer is used for passivation. By controlling the passivation sub-cycle, one is able to allow desired under-etching followed by "recovery" of the formerly under-etched features to make unique three-dimensional structures directly on silicon substrates [1].

Cleaned silicon samples are placed in an e-beam evaporation unit to deposit a 40nm chromium layer as the mask for the subsequent processing steps. The masking layer is patterned using precision projection lithography to achieve desired features between 100nm and 20 μ m. For ultra-high aspect ratio and scallop-free etching while keeping the etch-rate of 1 μ m/min, it is necessary to include trace values of H₂/O₂ during the etching step. Typical flows for H₂/O₂ and SF₆ are 200/200 and 5 sccm in the passivation step while the etching is mainly practiced with SF₆ (35 sccm). The plasma power is set at 250 W for the passivation and 130 W for the etching sub-cycle. By controlling these important parameters, we have realized three-dimensional features where the vertical structures have serpentine surfaces with desired recessions of 10 μ m. Moreover, we have been able to realize arrays of nano-metric 3-D features using Si/SiO₂ structures with a diameter of 2-3 μ m and features of the order of 100nm.

We realized 9-10 μ m high and 90nm wide nano-wires where the mask undercut is 30nm and the surface of the wires is almost free of "scallop". Scallop is side-effect of time-multiplexed processes where the periodic track of the etching step is seen on side-walls. To avoid this, while obtaining high-rates we have included H₂/O₂ gases during the etching sub-cycle. Normally H₂/O₂ gases act as the passivation layer, however the trace value of these gases does not affect the etching. Instead a slight passivation is formed on side-walls while the etching proceeds, prohibiting further lateral-etching of walls. We have studied the passivation layer using XPS and Ellipsometry. Thickness of the passivation layer is 2-3nm and it is mainly SiOF bonds (XPS). Field-emission SEM has been used to compare the results. Using this process we obtained high etch-rates of 0.8-1.1 μ m/min for features around 100nm. The height of the nano-wires is around 10 μ m, with an aspect ratio of 100 and more. This process uses low-density plasma with rapid steps and apart from MEMS/NEMS applications it can be used for "solar-cells" where nano-wires can significantly affect the efficiency and cost.

[1]. S. Azimi, A. Sandoughsaz, B. Amirsolaimani, J. Naghsh-Nilchi, S. Mohajerzadeh, "Three-dimensional etching of silicon substrates using a modified deep reactive ion etching technique", J. Micromech. Microeng. No. 21, 074005, (2011).

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