Monday Morning, October 18, 2010

Frontiers in Inkjet Technology Topical Conference Room: Tesuque - Session IJ+BI+MN-MoM

Frontiers in Inkjet Technology

Moderator: T. Boland, University of Texas at El Paso

8:20am IJ+BI+MN-MoM1 Drop Impact on Liquid, Solid and Porous Surfaces, A.L. Yarin, University of Illinois at Chicago INVITED The talk covers drop impacts on thin liquid layers, dry impermeable surfaces, and porous surfaces with nano-scale texture. Splashing and corona formation and propagation on liquid layers are discussed first. Then, some additional kindred, albeit non-splashing, phenomena like drop spreading and deposition, receding (recoil), jetting, fingering and rebound on liquid and dry impermeable solid surfaces are covered. A number of practical applications of drop impacts are mentioned and relevant experimental, theoretical and computational aspects are considered.

After that, a novel method of enhancement of drop and spray cooling for microelectronic, optical and radiological elements and server rooms, which require extremely high heat fluxes, is discussed. The key idea of the method is to cover the heat transfer surfaces with electrospun nonwoven polymer nanofiber mats. The experiments reveal that drop impacts on nanotextured surfaces of nanofiber mats produce spreading similar to that on the impermeable dry surfaces. However, at the end of the spreading stage the contact line is pinned and drop receding is prevented. All the mats appear to be dynamically permeable for water drops. The enhanced efficiency of drop cooling in the presence of nanofiber mats observed experimentally results from a complete elimination of drop receding and bouncing characteristic of the current spray cooling technology. Therefore, the drops evaporate completely, and the large cooling potential associated with the latent heat of water evaporation is more fully exploited. This is paradoxical: the best cooling can be provided by a "furry overcoat"! The results on drop impact on porous surfaces are also relevant for drop impacts on paper and nonwovens in the context of ink-jet-printed microelectronics.

9:00am IJ+BI+MN-MoM3 Upper and Lower Bounds for the Stability of Inkjet Printed Lines, *B. Derby*, *J. Stringer*, University of Manchester, UK

Many applications for inkjet printing require the ability to print continuous linear features. Inkjet printing achieves this through the overlap and coalescence of a series of liquid drops on a planar substrate, which then transform to a solid through phase change or solvent loss. In order to produce regular parallel sided printed lines, the intermediate fluid thread must retain morphhological stability prior to solidification. Drying fluid drops often show considerable hysteresis between the advancing and receding contact angles. This behaviour is shown to impose upper and lower bounds for the width of a line formed by the overlap of printed drops. The lower bound for line width is determined by the minimum separation distance for spreading drops to spread, overlap and coalesce. However, for liquids with zero receding contact angles (as is the case for many evaporating solvents) there is a further limit for parallel sided lines [1]. The upper bound for line width is determined by a dynamic fluid instability that occurs through competing fluid flows between the spreading and coalescence processes [2]. This dynamic instability is a function of both drop spacing and the rate of droplet deposition. By considering both the upper and lower bound limits we can construct a map in a parameter space defined by drop size, drop spacing, drop/substrate contact angle and linear printing velocity that shows the conditions under which stable linear features can be printed.

9:20am IJ+BI+MN-MoM4 Particle Deposition and Assembly of Inkjet-Printed Colloidal Drops in Line and Pattern Printing, A. Joshi, V. Chhasatia, Y. Sun, Drexel University

Precise control of process parameters during inkjet printing is essential to enable uniform, accurate and repeatable deposition of functional materials. In this work, we present a combined in-situ observation and computational study to examine particle deposition and assembly during evaporation of inkjet-printed colloidal drops. Unlike previous computational models that use tracer particles and known velocity fields inside the drop, our computational model uses a multi-phase lattice Boltzmann method (LBM) that directly simulates the flow of the liquid drop, surrounding vapor phase and the motion of the liquid-vapor interface. The motion of suspended particles within the liquid phase is directly coupled to the fluid flow and also influences the velocity field in the liquid. Evaporation is accomplished by reducing the vapor pressure above the drop and different evaporation modes including evaporation with contact line pinning and self-similar evaporation with a constant contact angle are examined. A novel visualization technique is developed wherein aqueous suspensions of fluorescent particles are jetted onto transparent surfaces and the evaporation dynamics are observed in real-time using a fluorescence microscope. The effects of drop spacing, jetting frequency, substrate wettability, particle size and volume fraction, and environmental conditions (temperature and humidity) on the final deposition morphology are presented for line and pattern printing of functional materials on substrates.

9:40am IJ+BI+MN-MoM5 Inkjet Printing of Flexible Hybrid Solar Cells based on P3HT and ZnO, G. Carryon, J.B. Baxter, Y. Sun, Drexel University

Inkjet printing of organic solar cells offers an inexpensive alternative to conventional solar cell fabrication methods. Despite the attractiveness of organic solar cells, they have demonstrated some degradation problems and have yet to achieve the efficiencies necessary to make them economically viable. In contrast to their organic counterparts, inorganic semiconductors have demonstrated advantages in their high dielectric constant which facilitates carrier generation processes, high carrier mobility, and thermal morphological stability. In recent years, improvements in device performance have been seen in the development of organic-inorganic hybrid materials (e.g., ZnO nanoparticle-polymer composites or CdSe quantum dot-polymer composites) as the photoactive layer. To date, most studies on hybrid solar cell fabrication have focused on using lab-scale spin-coating methods to deposit ZnO nanoparticle-polymer materials. In this paper, we present our results in using an industrial piezoelectric-driven printing device for inkjet printing of ZnO nanoparticles/nanorods-polymer [e.g., poly(3hexylthiophene (P3HT)] ink materials for large-scale processing of hybrid solar cells. The deposition morphology and properties of printed photoactive layer are examined as a function of the solvent properties (e.g., wettability and vapor pressure), particle size, volume fraction, and polydispersity, as well as the aspect ratio of nanorod. The effects of jetting parameters (e.g., wave form and jetting frequency) and printing patterns on film thickness and uniformity are also discussed in detail. Finally, the feasibility of printing P3HT onto aligned ZnO nanorod arrays for novel heterogonous nanostructures for reduced exciton diffusion lengths is explored.

10:00am IJ+BI+MN-MoM6 Anomalies in Applications of Inkjet Printing in Microfluidic Device Fabrication, W.E. Dieterle, C.P. McNary, California University of Pennsylvania

Utilization of inkjet-generated masks for UV photosensitive materials as a cost-effective method for the generation of microfluidic devices requires resolution of certain anomalies related to combinations of various color components. These anomalies are demonstrated for UV exposures with a high-pressure mercury vapor source and possible solutions are discussed, including implications for inkjet manufacture designs targeted to similar applications.

10:40am **IJ+BI+MN-MoM8 Fabrication of Miniature Drug Screening Platform Utilizing Low Cost Bioprinting Technology**, *J. Rodriguez*, *T. Xu*, University of Texas at el Paso

In the pharmaceutical industry, new chemicals and substances are being tested to find appropriate compounds for treating a specific disease. The demand for screening large compound collections against and increasing number of therapeutic targets has stimulated technology development in the areas of assay automation and miniaturization. Current methods for evaluating the reactions of cells use a relatively large volume in the range from microliters to milliliters; since reliability has to be met, it exists the need to have several assays to confirm the biochemical reactions, which ultimately cause the usage of large amounts of volume for each substance. Unfortunately, some of these new compounds are rather hard to obtain, which causes an expensive researched and limited material availability; therefore, increasing the time development for future cures. We have developed a new and low-cost deposition method to fabricate miniature drug screening platform that can realistically and inexpensively evaluate biochemical reactions up to 4 substances per trial in a picoliter-scale volume.

This study focues on the development of the controls for a deposition method (inkjet printing technology) which will simultaneously place therapeutic drugs and cells onto target sites to fabricate cell/drug chips for drug screening application. Using a modified HP 5360 CD printer, droplets of GFP expressing Escherichia coli have been deposited in an agar coated coverslip chip as small reliable volume of 180 picoliters per each colony dot, along with this bacteria it has been patterned different antibiotics in

such a way that we evaluated the growth of the bacteria under antibiotics presence. The viability and function of the printed cells were evaluated by the live/dead and plasmid gene transfection experiments resulting in 98% viability and maintaining DNA function. Moreover, it has been recorded as high throughput process printing 250,000 droplets/second. Due to the reduction of volume, this method will increase the effectiveness of the resources utilized for emerging drug screening processes. The results show promising usage of resources for future drug screening through new biochemicals.

11:00am **IJ+BI+MN-MoM9** Inkjet Printing of Oxygen Releasing Materials for Improved Cell Survival and Growth, *A. Arteaga*, *T. Xu*, University of Texas at el Paso

Introduction: A major barrier in tissue engineering is the impossibility of providing adequate oxygen to all cells within the engineered tissue before a full vascularization is achieved. To overcome this limitation, a variety of oxygen-producing particles have been developed for improving tissue survival. However, most of these particles are used in random mixtures with scaffolding materials, which usually leads to an uneven distribution of oxygen in bioengineered tissues. An ideal oxygen supply requires a precise spatial-temporal control of the oxygen-producing particles in scaffolds. Unfortunately, current oxygen delivering scaffold techniques are unable to perform as described and to precisely incorporate oxygen particles into the scaffolds. Cell inkjet printing is a novel tissue fabrication approach, in which a special inkjet printer can be programmed to deposit cells and/or biomaterials of various types and sizes in a very precise pattern. In this study we have applied the inkjet printing technology to allocate oxygen releasing materials to their designed positions for optimal cell viability and growth.

Methods: The controlled oxygen-releasing platform is fabricated by printing different patterns ("Black", "White", "Grey", and "Dots" to represent different densities of the oxygen particles on the substrates) of encapsulated calcium peroxide (CPO) particles that were analyzed against C2C12 mouse myoblast cell line for cell viability. CPO has been found to release its oxygen over an extended timeframe. The effects of controlled oxygen-releasing particles on cell viability were analyzed using the cell morphology study, live/dead assay, and the MTS assay.

Results: These analyses showed the concentration of the oxygen-releasing particles in "Black" was toxic to the cells based on the decreasing trend in cell viability. The "White" did not have oxygen-releasing particles, which correlates to the decrease in cell viability over time due to oxygen deprivation. Both "Grey" and "Dots" showed a similar trend in absorbency, in which the absorbency was low at 24 hours, there was an increase in absorbency at 48 hours, and then an abrupt decrease at 72 hours. Both these results suggest that the amount of oxygen released was beneficial to the cells within the first 48 hours, yet may not have been sufficient to sustain cell viability after that time span. The cells treated in the printed "Dots" showed to have the most compatible treatment for an overall increase in cell viability.

Conclusion: The amount of oxygen released can be controlled to optimize the cell by bioprinting different densities of the oxygen releasing materials onto a substrate.

11:20am **IJ+BI+MN-MoM10** Understanding Volume Ejection of Complex Fluids through Pressure Measurements, *G.E. Mårtensson*, *W. Holm*, Micronic Mydata AB, Sweden

In conventional ink jetting applications, a pressure difference is used to ensure the continuous and prompt filling of the jetting chamber between jetting actuations. The delivery of precise fluid volumes utilizing inkjet-like drop-on-demand jetting technology is primarily controlled by the piezo voltage that actuates the jetting chamber (Gerhauser et al. 1983, SID 83 Digest). The jetting of large volumes, in excess of 1 nL, of complex viscous fluids is complicated by the difficulty of filling the ejection chamber quickly after the previous droplet ejection.

A novel jetting mechanism for highly viscous complex fluids, that utilizes a viscous micropump to control the amount of fluid that is ejected by a piezo actuated mechanism, has been developed and implemented by the authors. In this paper , we report results of volume, exit velocity, and pressure measurements. The piezo voltage, V_p , and the angular speed, N, of the viscous pump has been varied. A chosen V_p translates directly to displaced volume and N to flow rate.

The ejected volume has been measured by weighing a large number of droplets and via 3D profilometry methods. It has been shown in the experimental jetting setup that the volume of a jetted deposit is only affected to a minor degree, of the order of 5% of the target volume, by the chosen piezo voltage, V_p . Thus the ejected volume is almost independent of the displaced volume within the experimental range and determined by the flow rate, which in turn is controlled by N.

The exit velocity of the jet has been measured using high speed double exposure imaging. It has been shown that the speed of the ejected droplet has a nearly linear response to V_p (at least for all but the smallest N). For a given V_p , the exit velocity increase with increasing N. Thus, it seems that a larger displaced volume results in a higher exit velocity, but the ejected amount is unaffected.

In order to probe these seemingly counterintuitive results, the pressure in the ejection chamber, as well as in the viscous micropump, was measured over a large number of ejection cycles. The volume of the resulting depositions were correlated with the chamber and pump pressures. Additional measurements were performed to correlate the speed of the resulting shot with the chamber and pump pressures. Simple models are proposed to correlate the above mentioned quantities.

11:40am IJ+BI+MN-MoM11 Determination of Effective Jet Radius from Measurements of the Perturbation Growth Rate in Thermally Stimulated Continuous Microjets, J.M. Grace, G. Farruggia, E.P. Furlani, Z.J. Gao, K.C. Ng, Eastman Kodak Company

Drop formation in continuous inkjet devices is based upon the Rayleigh-Plateau instability - a phenomenon in which surface tension drives the break-up of a column of fluid into droplets. In thermally stimulated continuous inkjet devices, heat pulses applied to the jet at the nozzle couple to the instability to stimulate drop formation. The level of stimulation depends upon the size of the effective perturbation and its growth rate along the jet. While the growth rate depends upon characteristics of the jet itself (fluid properties and jet diameter), the effective perturbation depends upon coupling between the source of stimulation and the jet, as well as the fluid properties. The coupling efficiency can be inferred from measurements of the perturbation growth rate and the jet diameter. For liquid microjets with diameters of 10 micrometers or less, direct determination of the jet diameter by optical microscopy is extremely challenging. Although the lateral dimensions of the microjets may be difficult to measure precisely, the break-up length can be determined with relatively good precision. Measurements of break-up length as a function of input power provide a means to determine the perturbation growth rate. From the experimentally determined growth rate as a function of stimulation frequency, the diameter of the microjet can be determined by fitting to a model for jet break-up. The experimentally determined growth rate and jet diameter provide a basis for comparing the effective coupling for different designs of jetting modules. Measurements of jet break-up and methods for determining the effective jet diameter will be presented and discussed.

Vacuum Technology Room: Laguna - Session VT+MN-MoM

MEMS Sensors, Vacuum Gauges, Measurements and Pumps

Moderator: J. Fedchak, National Institute of Standards and Technology

8:20am VT+MN-MoM1 Practical Issues and Applications for Vacuum and Hermetic Microsystems Packaging, L. Fang, D. Chu, K. Ewsuk, Sandia National Laboratories INVITED Microsystems, packaging, involves, physically, placing, and electrically

Microsystems packaging involves physically placing and electrically interconnecting a microelectronic device in a package that protects it from and interfaces it with the outside world. When the device requires a hermetic or controlled microenvironment, it is typically sealed within a cavity in the package. Sealing involves placing and attaching a lid, typically by welding, brazing, or soldering. Materials selection (e.g., the epoxy die attach), and process control (e.g., the epoxy curing temperature and time) are critical for reproducible and reliable microsystems packaging. This paper will review some hermetic and controlled microenvironment packaging at Sandia Labs, and will discuss materials, processes, and equipment used to package environmentally sensitive microelectronics (e.g., MEMS and sensors).

9:00am VT+MN-MoM3 New NIST Comparison Method Calibration Service for Vacuum Gauges Based on MEMS Pressure Sensors, J.H. Hendricks, D.A. Olson, NIST

A new calibration service based on a secondary pressure transfer standard spanning the pressure range from 0.65 Pa to 130 kPa (5 mTorr to 975 Torr) has been developed at NIST. Until now, vacuum gauges in this range could only be calibrated using the NIST Ultrasonic Interferometer Manometers (UIMs). However, many customers desire direct traceability to NIST but cannot justify the cost of the NIST UIM calibrations. The new service follows a similar model of other calibration services where a lower cost, and less accurate service is offered to customers who do not require the lowest uncertainty possible but still desire direct NIST tracibility. The comparison method utilizes a high accuracy transfer standard package that consists of a 133 Pa (1 Torr) Capacitance Diaphragm Gauge (CDG), a 13.3 kPa (10 Torr) CDG and a 130 kPa (975 torr) MEMS type Resonance Silicon Gauge (RSG) all encased in a temperature controlled enclosure that is periodically calibrated against the NIST 160 kPa UIM and 140 Pa oil UIM primary pressure standards. Due to the superior calibration stability of the MEMS based RSG, the transfer standard package, and ultimately the comparison method vacuum gauge service, provides expanded uncertainties as low as 0.05 % from 1.33 kPa to 130 kPa (10 Torr to 975 Torr) and 0.3 % from 1.33 Pa to 1.33 kPa (0.01 Torr to 10 Torr).

9:20am VT+MN-MoM4 Pirani for Industrial Processes, B. Andreaus, R. Enderes, M. Wuest, INFICON Ltd, Liechtenstein

In modern Pirani heat transfer gauges a filament is usually kept at a constant temperature and the necessary heating power is measured as a function of pressure.

Pirani gauges operated in coating and etching applications suffer from degradation due to process contamination or corrosion. Eventually, the Pirani may fail because the filament is etched away, its resistance and/or its emissivity have changed. Standard procedure for those processes is to use a corrosion resistant filament material adapted to the process in question, e.g. Nickel. Yet the choice of suitable filament material is limited as it needs to be manufactured as very thin coils, be electrically conducting, have a high and well-defined temperature coefficient for the resistance and be chemically inert. For some of the latest manufacturing processes in sufficiently resistant against corrosion.

We will present here a different approach in that we present first data on a coated Pirani sensor. The coating allows for a much broader field of application as electrical and mechanical requirements of the filament are separated from its chemical properties. Chemical stability is solely due to the coating, all other requirements, unaffected by the coating, can be met using a standard filament material.

9:40am VT+MN-MoM5 Nitrogen Incorporated Ultrananocrystalline Diamond as a Robust Cold Cathode Material for Miniature Mass Spectrometry Application in Space Exploration, X. Wang, University of Puerto Rico; Argonne National Laboratory, S. Getty, NASA Goddard Space Flight Center, A.V. Sumant, O.H. Auciello, Argonne National Laboratory, D. Glavin, P. Mahaffy, NASA Goddard Space Flight Center

Ultrananocrystalline diamond (UNCD) thin films have been investigated for over a decade for application to electron field emission devices since they offer very low threshold voltage (1-3 V/µm) and reasonably stable field emission with time. Due to the small grain size (2-5 nm) and unique atomically abrupt grain boundary structure containing mixed sp^2/sp^3 carbon bonding, it has been postulated that field emission occurs mainly at the grain boundary due to the high field enhancement effect at the grain boundary and stable field emission has been observed independent of surface geometry or film thickness. In addition to low power consumption and potential for miniaturization, robust field emission materials are compelling for applications as long life electron sources for mass spectrometers for space exploration where electron sources are exposed to harsh environments. A miniaturized mass spectrometer under development for in situ chemical analysis on the moon and other planetary surfaces requires a robust, long-lived electron source, to generate ions from gaseous sample using electron impact ionization. To this end, we have explored the field emission properties and lifetime testing of nitrogen-incorporated ultrananocrystalline diamond (N-UNCD). The N-UNCD films were synthesized in a microwave plasma chemical vapor deposition system by introducing nitrogen in the Ar/CH4 gas chemistry. Characterization of the N-UNCD films were carried out by using visible and UV Raman spectroscopy confirming characteristic signature of a good quality N-UNCD film. We will present results revealing that UNCD films with nitrogen incorporation during growth yield stable/high field-induced electron emission in high vacuum for up to 1000 hours.

This work was done with support from the NASA Astrobiology Science and Technology Instrument Development Program, under Grant Number 07-

ASTID07-0020, and the NASA Goddard Space Flight Center Internal Research and Development Program. Use of the Center for Nanoscale Materials was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

10:40am VT+MN-MoM8 The Pumping Synergies of Integrated NEG and SIP Pumps for UHV Applications, A. Bonucci, A. Conte, L. Caruso, L. Viale, P. Manini, SAES Getters S.p.A., Italy

A variety of vacuum systems, such as particle accelerators, synchrotrons, surface science chambers or laboratory equipments, do require the achievement of very high or even extremely high vacuum conditions(UHV-XHV). To this purpose, lon pumps and Non Evaporable Getter (NEG) technologies are widely applied, since they complement each other effectively. Ion pumps remove ungetterable species like noble gases and methane, while the NEG provides a constant and large pumping speed for all the other gases, in a very compact volume.

So far ion and NEG pumps have been mostly used as separate units mounted in separate part of the vacuum system. In this paper, we investigate how overall pumping performances are influenced by the mounting geometry of the two pumps. In particular we will show that a remarkable synergic effect arises when the two pumps are integrated into one unit having optimized design, known as NEG⁺.

This configuration allows to minimize the detrimental effect given by outgassed species released by the SIP $[^{1,2}]$ This increases the real pumping speed of the SIP, generally masked in the UHV range by gas desorption from SIP internal surfaces. This effect is particularly noticeable for ungetterable gases like Argon and Methane.

The resulting pumping speed of the NEG⁺ is therefore larger than the sum of the pumping speed of the two separated pumps.

Reducing the degassing effect also increases the overall pumping efficiency at the lower pressure.

In fact, the presence of oxides and nitride compounds onto the cathode surface are known to slow down the diffusion of hydrogen and helium into the cathode[³], which is mainly an ion implantation driven process.

In the present paper we discuss some of these effects as well as the synergies arising from the NEG+ integrated design. A specific focus will be given to argon [⁴] and methane, which are important gases to consider in a variety of application including electron microscopy and electron/ion optics.

[[1]] K.M.Welch, D.J.Pate and J.Todd, "Pumping of helium and hydrogen by sputter-ion pumps. II. Hydrogen pumping", J.Vac.Sci.Technol.A 12(3), May/Jun 1994

[2]A Calcatelli et als. "Study of outgassing of sputter-ion pump materials treated with three different cleaning procedures", Vacuum vol. 47 n. 6-8, 1996

[3]M. Audi and M. De Simon, "The influence of heavier gases in pumping helium and hydrogen in a an ion pump", J. Vac. Sci. Technol. A 6 (3), May/Jun 1988

[4]D. Andrew, D.R. Sethna and G.F. Weston, "Inert-Gas pumping in a magnetron pump", proc. 4th AVS, (1968)

11:00am VT+MN-MoM9 NEG+: A Novel Route to Compact, High Performance Pumping in UHV-XHV Vacuum Systems, *P. Manini*, *A. Bonucci*, *A. Conte*, *L. Viale*, *L. Caruso*, SAES Getters S.p.A., Italy

The need to miniaturize and reduce the footprint of vacuum systems is growing in a variety of industrial and R&D applications, encompassing scanning and transmission microscopes, vacuum chambers for surface science, material preparation or portable analyzers.

This is posing serious challenges to the UHV pumping groups in term of design, space constraints and weight. Even in large vacuum systems, like synchrotrons and particle accelerators, fitting the vacuum pumps is becoming increasingly difficult, due to the presence of magnets, power stations, a variety of diagnostic tools and instrumentation, as well as service and experimental devices. Non Evaporable Getter (NEG) pumps are very compact and light, vibration-free devices, able to deliver extremely high pumping speed per unit volume with minimal power requirement. One of the main drawbacks of NEG pumps is their inability to pump noble gases and methane. In the present paper the design of a novel combination pump, called NEG⁺, is introduced. In the NEG⁺, the getter cartridge acts as the main pumping element, leaving to an ancillary and small sputter ion pump the task of removing noble gases and methane, which are not pumped by the NEG. Pumping of all the gases as well as the ability to provide a pressure reading, so helpful in a variety of applications, is therefore possible in one single and compact unit. The design of the NEG⁺ is optimized to ensure a high integration between the getter element and the ion pump. This provides large pumping speed in a very compact volume as well as interesting synergies in the pumping of gases at the UHV level. The design

and general properties of this new pump, including pumping tests and example of applications will be reported.

11:20am VT+MN-MoM10 Direct Simulation Monte Carlo Modeling of Miniature Vacuum Pumps, *B.J. Davis*, *R.W. Hill*, *P.H. Sorensen*, *R.J. Kline-Schoder*, Creare Incorporated

NASA and other organizations have pressing needs for miniaturized high vacuum systems. Recent advances in sensor technology at NASA and commercial laboratories have led to the development of highly miniaturized time-of-flight, quadrupole, and ion trap mass spectrometers. However, high vacuum systems of adequate performance continue to be too large, heavy, and power hungry for man-portable mass spectrometers or spectrometers deployed on UAVs, balloons, or interplanetary probes. Terrestrial, man-portable applications impacted by this problem include military and homeland defense systems for detecting hazardous materials as well as portable leak detectors for commercial use.

For over 10 years, Creare has been developing the technologies required to design and build miniature high vacuum pumps. We have designed and built pumps that are as small as a D-cell battery, reach an ultimate pressure of 10e-7 torr, have a flow rate in excess of 5 L/s, and spin at 200,000 RPM. As mass spectrometers are reduced in size, the vacuum system requirements can be relaxed. As a consequence, Creare is developing an extremely low-cost and rugged high vacuum system whose performance is optimized for miniature mass spectrometers. The vacuum system is based on an innovative molecular drag pump designed to match the requirements of portable analytical instruments.

To support our miniature vacuum pump design efforts, Creare has developed statistical models of molecular drag pumps (MDP) in the free molecular flow regime. In this method, individual molecular trajectories through a simplified three-dimensional representation of the pump are calculated. The initial positions and velocities of the particles as they enter the pump are randomly generated, with statistics consistent with the gas states at the inlet and outlet of the pump. The free-molecular statistical simulation can be used to determine the probability that a molecule entering the pump at the inlet (outlet) exits through the outlet (inlet). In the freemolecular regime, these probabilities are sufficient to determine the pump's capabilities for compression, flow rate, etc.

We will describe the modeling methods, the verification of the models using previously published data, and the results of special experiments performed to verify that the models can be used to support new miniature pump designs.

11:40am VT+MN-MoM11 Improvements in the Performance of Turbomolecular Pumps Beyond the Molecular Range, A. Chew, B. Brewster, I. Olsen, S. Ormrod, Edwards Ltd, UK

A new range of turbomolecular pumps, nEXT, has been developed. This incorporates a new damping mechanism and pumping stage options. A new Siegbahn drag stage in combination with a regenerative mechanism are described in their combination with pure turbomolecular stages. Consequent increased backing pressures, high compression ratios and the facilitisation of a boost port being used to back other turbos will be described.

Wednesday Morning, October 20, 2010

Nanometer-scale Science and Technology Room: La Cienega - Session NS+AS+MN-WeM

Characterization and Imaging at Nanoscale

Moderator: E.I. Altman, Yale University

8:20am NS+AS+MN-WeM2 Surface Preparation of Supported Flat Gold Nanoparticles for use as Au(111) Single Crystal Substrates, *D.H. Dahanayaka*, *L.A. Bumm*, The University of Oklahoma

Flat gold nanoparticles (FGNPs) grown in aqueous solution have large Au(111) facets that are excellent substrates for scanning probe microscopy. However adsorbed stabilizers (e.g. polyelectrolytes) must be removed or displaced before the FGNP surfaces can be used as single crystal surfaces. We have explored the effects of plasma cleaning, UV ozone, and thermal annealing on the surface roughness and the Au(111) terrace structure using STM.

This work has been supported by NSF CAREER grant No. CHE- 0239803, NSF MRSEC No. DMR-0080054, NSF No. DMR-0805233d NSF, and AFOSR No. FA9550- 06-1-0365.

8:40am NS+AS+MN-WeM3 Determination of the Adsorption Site for Alkanethiol Monolayers on Au(111), *Q. GUuo*, University of Birmingham, UK

The bonding sites for Au-adatom-octanethiolate within the $(\sqrt{3} \times \sqrt{3})$ R30° structure on Au(111) has been investigated with high-resolution scanning tunneling microscopy (STM) imaging. By establishing the relationship between the lateral positions of adsorbates on the top layer of gold and those inside an etch pit, we are able to determine the adsorption configuration with a high degree of accuracy for the illusive $(\sqrt{3} \times \sqrt{3})$ R30° molecular layer. Within any one particular domain, the Au-adatom-octanethiolate species are found to occupy either the fcc hollow or the hcp hollow site.

9:00am NS+AS+MN-WeM4 Atomic Co Wires: Room and Low Temperature STM/STS Measurements, N. Zaki, Columbia University, D. Acharya, Brookhaven National Laboratory, D.V. Potapenko, Columbia University, P. Johnson, P. Sutter, Brookhaven National Laboratory, R.M. Osgood, Columbia University

We recently reported [1] on a new surface phase of the Co-vicinal-Cu(111) system which exhibits self-assembled uniform Co quantum wires that are stable at 300K. STM images show that the wires form along the leading edge of the step rise, differentiating it from previously theoretically predicted atomic-wire phases as well as experimentally observed step-island formation. Our observations allow us to comment on the formation kinetics of the atomic-wire phase and on the fit of our data to a recently developed lattice-gas model. Low-temperature STS measurements, taken on selfassembled Co chains, reveal a resonance at the Fermi energy. While it has been shown that single Co atoms and Co-Cu_n clusters [2] exhibit a Kondo effect, a Co chain at a Cu step may exhibit a different many-body effect that is the cause for our Fermi-energy resonance observation. Furthermore, we have observed different charge-density modulation that is dependent on tip bias. Since these charge-modulations are observed for tip-bias relatively far away from the Fermi level, we suspect that these modulations are not ground state charge-density-waves (CDW), but rather excited states of this 1-D system.

[1] N. Zaki et al, Phys. Rev. B 80, 155419 (2009)

[2] N. Néel et al, Phys. Rev. Lett. 101, 266803 (2008)

9:20am NS+AS+MN-WeM5 Atomic-Resolution Spin Mapping by Exploiting Magnetic Exchange Forces, R. Wiesendanger*, University of Hamburg, Germany INVITED

While Spin-Polarized Scanning Tunnelling Microscopy (SP-STM) [1] is nowadays well established for revealing atomic spin configurations at surfaces, its application is limited to electrically conducting samples such as magnetic metals or semiconductors. In order to map atomic spin structures at surfaces of insulators and to open up the exciting possibility of studying spin ordering effects with atomic resolution while going through a metalinsulator transition, we have developed Magnetic Exchange Force Microscopy (MExFM) [2]. This technique is based on the detection of short-range spin-dependent exchange and correlation forces at very small tip-sample separations (a few Angstroms), in contrast to Magnetic Force Microscopy (MFM) where the magnetic dipole forces are probed with a ferromagnetic probe tip at a typical tip-to-surface distance of 10-20 nm [3]. MExFM has allowed a first direct real-space observation of spin structures at surfaces of antiferromagnetic bulk insulators [2] as well as ultrathin films [4]. Moreover, it provides a powerful new tool to investigate different types of spin-spin interactions based on direct-, super-, or RKKY-type exchange down to the atomic level. By combining MExFM with high-precision measurements of damping forces [5] localized or confined spin excitations in magnetic systems of reduced dimensions become experimentally accessible [1].

[1] R. Wiesendanger, Rev. Mod. Phys. 81, 1495 (2009).

[2] U. Kaiser, A. Schwarz, and R. Wiesendanger, Nature 446, 522 (2007)

Y. Martin and K. Wickramsinghe, Appl. Phys. Lett. 50, 1455 (1987); J.
J. Saenz, N. Garcia, P. Grütter, E. Meyer, H. Heinzelmann, R.
Wiesendanger, L. Rosenthaler, H. R. Hidber, and H.-J. Güntherodt, J. Appl.
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10:40am NS+AS+MN-WeM9 Chemical Imaging and Interaction Quantification on the Surface Oxide Layer of Cu(100) Using High-Resolution Atomic Force Microscopy, *M.Z. Baykara*, *T.C. Schwendemann, H. Mönig*, Yale University, *M. Todorovic, R. Pérez*, Universidad Autónoma de Madrid, Spain, *E.I. Altman, U.D. Schwarz*, Yale University

Chemistry is governed by the interactions between atoms and molecules. On surfaces, chemical forces extending into the vacuum direct the behavior of many scientifically and technically important phenomena including surface catalysis. Therefore, it would be useful to map and quantify the interactions between a catalytically active surface and a probe with atomic resolution in order to study the role and effectiveness of various surface defects such as vacancies, impurities, steps, kinks, and domain boundaries as active sites. An ability to discriminate between different chemical species on the sample surface would offer further insight. In this talk, we will show with the example of an oxygen-reconstructed copper (100) surface that much of this information can be derived from combining the new method of three-dimensional atomic force microscopy (3D-AFM) [1], a variant of noncontact atomic force microscopy, with scanning tunneling microscopy. The surface oxide layer of Cu(100) features domain boundaries and a distinct structure of the Cu and O sublattices that is ideally suited for such model investigations. While different tips show different chemical contrasts, 3D data sets enable site-specific quantification of force interactions and tunneling currents. In order to clarify the different contrast modes data, DFT total-energy calculations and Non-equilibrium Green's Function (NEGF) methods for electronic transport have been used to determine the interaction and the tunneling current [2-4] for a large set of tip models. These calculations provide insight into (1) the fundamentals of contrast formation in this experimental technique and (2) into the correlation between tip-sample forces and local chemical reactivity, factors that are essential for the further development and application of this novel approach to characterizing catalytic activity.

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11:00am NS+AS+MN-WeM10 Comparison of Resonant-Frequency Techniques for AFM Nanomechanical Mapping, D.C. Hurley, J.P. Killgore, A.B. Kos, NIST, A. Gannepalli, R. Proksch, Asylum Research Contact-resonance force microscopy (CR-FM) is an emerging AFM technique for quantitative imaging of near-surface nanoscale mechanical properties. In CR-FM, the resonant frequency f of the cantilever is measured when the tip is in contact with the sample. Mechanical-property values are obtained from the frequency data with the use of models for the vibrating cantilever and the tip-sample contact. Contact-resonance measurements were originally made at a fixed sample position with off-the-shelf

^{*} NSTD Recognition Award

electronics. However, to achieve sufficient speed for CR-FM imaging, it has been necessary to develop new instrumentation approaches. Here we describe work to directly compare three methods for CR-FM imaging: the SPRITE (Scanned Probe Resonance Image Tracking Electronics) approach developed at NIST, the DART (Dual AC Resonance Tracking) approach developed by Asylum Research, and the BE (Band Excitation) method originally developed at Oak Ridge National Laboratory and implemented by Asylum Research. Each method enables contact-resonance frequency mapping but achieves it through different practical implementations. First, we will discuss the concepts on which each method is based. Next, results of comparison experiments will be presented in which images were acquired with each method in succession on the same AFM instrument. A variety of specimens were imaged to probe the strengths and limitations of each method. For example, we found that DART could operate at higher scan speeds, while SPRITE and BE were better able to track very asymmetric peaks that presumably originate from nonlinear tip-sample interactions. Both DART and BE provide values of the resonance quality factor Q in addition to the resonant frequency f, while currently SPRITE measures f only. Despite these differences, the methods yielded similar results in many cases. The availability of a broader array of frequency mapping tools will ultimately facilitate the widespread application of CR-FM to nanoscale materials science.

11:20am NS+AS+MN-WeM11 Single Molecule Structural Transitions of Water Polymer Chains in a Nanoscale Confined Space Studied by COIFM, *B.I. Kim*, Boise State University

Interfacial water structures have been investigated in an ambient environment between two silica surfaces using a newly developed cantilever based optical interfacial force microscope (COIFM). As the gap distance decreases between the tip and the substrate, a remarkable oscillatory behavior is oberved in normal and friction forces. Our further analysis suggests that water molecules confined between tip and substrate in an ambient environment form a bundle of water chains through hydrogen bonding. Each chain length is analyzed by a model called "freely jointed chain" (FJC) model in which the individual segments can rotate freely. The bundle of water chains experiences multiple layering transitions (without single layer transition) from l = 36 diameters to l=12 diameters as the tip approaches the surface. The analysis shows that the number of links (1) decreases by 5,4,3,3,3,3,4,5 in water diameters. The result indicates that, as the gap between the tip and the substrate decreases, the interfacial water molecules favor multilayer transitions to stepwise, single layer transitions. The multilayer transitions consist of a sequential reduction of total chain length by integer number of water diameters. The loss of one water diameter in overall chain length represents a highly cooperative transition, whereas the loss of two additional water diameter along the sigmoidal shape is reminiscent of a well-known two state transition. As a model to describe these transitions, a kink is introduced in the chain by rotating one water molecule 90 degrees while maintaining the number of hydrogen bonds in the chain. The other remaining molecules still follow the freely jointed chain model.

Plasma Science and Technology Room: Galisteo - Session PS+MN-WeM

Plasma Processing for 3D Integration, TSV, and MEMS Moderator: M. Darnon, CNRS-LTM, France

8:00am **PS+MN-WeM1 High Etch Rate of TSV using by Ultra Self-Confined VHF-CCP**, *Y. Morikawa*, *M. Yoshii*, *N. Mizutani*, *K. Suu*, ULVAC, Inc., Japan

Thru silicon via (TSV) etch process for deep and high-aspect ratio structure has been studied thoroughly for applications such as MEMS devices. Recently, TSV used in 3D-LSI devices, the via diameter and depth would be several tens of microns, and, the package for CMOS image sensors using TSV may have via diameters and depths up to 100 microns. A diameter of above 50um account for 50 % of TSVs. Therefore, development of high etch rate about 50um via is very important for realizing these applications. In this study, a large via size of 50 um etching in a low-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. And the cathode has a structure designed to minimize the stray capacitance (Cs) and impedance (L) to get a low-pressure process of about 100Pa or more. Low-pressure process was carried out on the plasma confined, because mean free pass is very short. And, ion energy distribution (IED) is also controllable by low-presser process with VHF bias. The bimodal IED changes under low-pressure. The peak of highenergy side is reduced, and a charge exchange peak appears. It is considered that the charge exchange is important to anisotropic Si etching with VHF bias. Finally, an etch rate of more than 60 μ m/min was realized. It was found that the Si etch rate depended on fluorine radical density and ion energy distribution, so, the high rate was obtained by creating a high fluorine radical density condition by using a high pressure condition of 100Pa using a VHF-CCP reactor with an ultra confined system and SF6 gas chemistry.

8:20am **PS+MN-WeM2 Very Uniform and High Rate TSV Etching Process in Advanced NLD Plasma**, *Y. Morikawa*, *T. Murayama*, *K. Suu*, ULVAC, Inc., Japan

The h igh-density of thru silicon via (TSV) is indispensable to the utilization and improvement in performance of 3D-LSI. Advanced high aspect ratio TSV etching technologies are required for high-density TSV formation. We have developed a new etching system for TSV application. This s ystem is a planer type magnetic neutral loop discharge (NLD) plasma, which is named as advanced NLD. For high rate silicone etching, it is very important to understand not only the high density of the plasma generation but also the high density of fluorine atoms. In this study, a novel RF antenna ' M ulti Stacked rf A ntenna' has also been developed for the purpose of high rate etching. This antenna consists of multistage spiral turn rf antennas to reduce self- inductance (L), and is increased from turn of spiral to extend the inductive coupling discharge region. T he L feature of this antenna is 0. 95 uH and it is a low L antenna compared to the standard spiral antenna (1.7uH). As a result of performing the electron density measurement of the NLD plasma using this MS antenna, it succeeded in the high-density plasma production of 1×10^{12} / cm³ by the process pressure of 7 Pa. Next, the Si etching process development was performed using the a dvanced NLD etcher. Si etching characteristics employing advanced NLD plasma were studied with respect to distance from an antenna. As a result, the etching rate improved 4 times more compared to the standard NLD. Finally, the diameter of 1.5 um was attained by the anisotropic etching of 8. 5 um/min, and the aspect ratio is 5.3 using the a dvanced NLD etcher.

8:40am **PS+MN-WeM3 Deep Reactive Ion Etch Process Optimization for Control of Sidewall Profile and Morphology as a Function of Aspect Ratio**, *R.J. Shul*, *R.L. Jarecki*, *T.M. Bauer*, Sandia National Laboratories, *M. Wiwi*, LMATA Government Services

Deep reactive ion etching (DRIE) has become an enabling technology for the fabrication of many integrated microsystems, including accelerometers and gyroscopes, micro-fluidic devices, sensors, electrostatically actuated devices, and devices requiring back side optical access. The ability to etch deep Si structures with anisotropic sidewalls hundreds of microns deep has established a new set of devices in the MEMS area. Significant improvements in equipment and understanding of the process conditions have improved device yield and performance, and process reliability. Even with these improvements, several process issues are not well understood and often limit applications for the process. For example, sidewall morphology is often dominated by scalloping created by the iterative deposition-depassivation-etch cycle. Scalloping may make it difficult for deposition of materials on the sidewalls post DRIE or create non-optimal flow conditions for micro-fluidic devices. In addition, profile control of deep structures as a function of aspect ratio has not been optimized. For example, we have observed that creating positively tapered trench sidewalls often results in a trench bottom that exhibits a characteristic micromasked, grassy appearance. Conversely, eliminating the grass often results in a profile that undercuts the etch mask. Depending on the application, these phenomenon prevent the use of DRIE for device fabrication or cause the process to be optimized for specific structures thus preventing yield of other structures. In this presentation, we report on our efforts to vary DRIE process conditions to optimize sidewall profile and sidewall morphology as a function of aspect ratio. Structures considered in this study range from 10 microns to 700 microns in width, with etch depths to several hundred microns. We observe that passivation time, as well as ion energy, and ion flux in both the depassivation and etch cycles, have significant effect on the sidewall profile as a function of aspect ratio. We have also included morphing experiments in this study, where morphing is changing DRIE process parameters as a function of total process time. To optimize sidewall profile and morphology, the magnitude of the process changes during the morphing process is not necessarily linear with time. Results of these experiments will also be reported.

Sandia National Laboratories is a multi program laboratory operated by Sandia Corporation, a Lockheed Martin Company for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

9:00am **PS+MN-WeM4 XeF₂ Vapor Phase Silicon Etch used in the Fabrication of Movable SOI Structures**, *J. Stevens*, *R.J. Shul*, Sandia National Laboratories, *M. Wiwi*, *C.L. Ford*, LMATA Government Services, *T. Plut*, *T.M. Bauer*, Sandia National Laboratories

Vapor phase XeF₂ has been used in the fabrication of various types of devices including MEMS, resonators, RF switches, and micro-fluidics, and for wafer level packaging. In this presentation we demonstrate the use of XeF₂ Si etch in conjunction with deep reactive ion etch (DRIE) to release single crystal Si structures on Silicon On Insulator (SOI) wafers. XeF₂ vapor phase etching is conducive to the release of movable SOI structures due to the isotropy of the etch, the high etch selectivity to silicon dioxide (SiO₂) and fluorocarbon (FC) polymer etch masks, and the ability to undercut large structures at high rates. Also, since XeF₂ etching is a vapor phase process, stiction problems often associated with wet chemical release processes are avoided. Monolithic single crystal Si features were fabricated by etching continuous trenches in the device layer of an SOI wafer using a DRIE process optimized to stop on the buried SiO₂. The buried SiO₂ was then etched to handle Si using an anisotropic plasma etch process. The sidewalls of the device Si features were then protected with a conformal passivation layer of either FC polymer or SiO2. FC polymer was deposited from C₄F₈ gas precursor in an inductively coupled plasma reactor, and SiO₂ was deposited by plasma enhanced chemical vapor deposition (PECVD). A relatively high ion energy, directional reactive ion etch (RIE) plasma was used to remove the passivation film on surfaces normal to the direction of the ions while leaving the sidewall passivation intact. After the bottom of the trench was cleared to the underlying Si handle wafer, XeF2 was used to isotropically etch the handle Si, thus undercutting and releasing the features patterned in the device Si layer. The released device Si structures were not etched by the XeF₂ due to protection from the top SiO₂ mask, sidewall passivation, and the buried SiO₂ layer. Optimization of the XeF₂ process and the sidewall passivation layers will be discussed. The advantages of releasing SOI devices with XeF2 include avoiding stiction, maintaining the integrity of the buried SiO₂, and simplifying the fabrication flow for thermally actuated devices. Sandia National Laboratories is a multi program laboratory operated by Sandia Corporation, a Lockheed Martin Company for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

9:20am PS+MN-WeM5 SF₆/O₂/HBr Plasma Processes for the Etching of High Aspect Ratio through Silicon Via, S. Avertin, STMicroelectronics, France, E. Pargon, T. Chevolleau, Ltm - Umr 5129 Cnrs, France, F. Leverd, P. Gouraud, C. Verove, STMicroelectronics, France, O. Joubert, Ltm - Umr 5129 Cnrs, France

Today, the integration density and the chip dynamic power consumption are limiting and restricting phenomena. More than 50% of this consumption is due to long horizontal interconnects, and this rate is projected to increase. One solution to resolve these problems is 3D-Integration which provides smaller wire-length distribution by minimizing the connection length thanks to the fabrication of vertical vias through the silicon substrate or/and the chip. The ITRS roadmap requirement is to etch vias with 2-5 µm in diameter and high aspect ratio (>5). For deep silicon etching, the Bosch etch process which consists in alternating isotropic etching and deposition steps leads to the formation of the so-called scalloping phenomenon on the sidewalls (>100nm). In this paper, we propose to characterize and develop conventional plasma etching processes as an alternative to the Bosh process. The etching development is carried out in ICP reactor accepting 300mm wafers (DPSII from AMATTM) using SF₆/O₂/HBr plasma chemistries. The scientific objectives are to study the etching mechanism and passivation layer formation in order to get high etch rate (>3µm.min⁻¹), straight profiles and a controlled undercut (<50nm). The etching profiles and etch rates have been analysed using Scanning Electron Microscopy while etch and passivation mechanisms have been studied by quasi-in-situ X-ray Photoelectron Spectroscopy (XPS) and plasma diagnostics (Mass Spectroscopy, ion flux probe..). Preliminary results indicate that the etch mechanisms are strongly driven by the ratio of neutral over ion fluxes and that the etch process is very sensitive to microscopic effects such as the local loading of fluorine and oxygen radicals which is directly correlated to the local pattern density. Through a better understanding of the etch mechanisms, high aspect ratio silicon via with anisotropic profiles and minimized undercut have been obtained.

9:40am PS+MN-WeM6 Study on the High Aspect Ratio Si Etch for D2x Devices, Y. Gwangyong, P. Jongchul, Samsung Electronics, Republic of Korea

As the design rule of the semiconductor devices decreases, the device fabrication technology is facing many difficulties. One of issues is the STI trench etching profile in case of the aspect ratio(A/R) over 20, and the traditional etching technology is not working properly any more. One among those problems is intra-cell loading which is due to the insufficient exhaustion of by-product from the narrow space (less than 30nm). The other

is the bowing profile which results in the bad STI filling to generate the severe electrical short fails of a DRAM device. In this study, we researched and developed the innovative STI trench etching technologies to improve those problems. The one is the bias-pulsed plasma etching that repeats periodically plasma ON and OFF, which gave the dramatic decrease of the intra-cell loading. In addition that gave the side p assivation effect to result in straight side-slope without bowing. These two effects are due to the byproduct exhaustion and the radical attaching during the plasma OFF time. A nd the high temperature etching process and the multi-step Oxygen flashing process also improved the intra-cell loading significantly. We got to know that these new Si etching technologies are successfully applied to the future high A/R(> 15:1) STI process for D2x DRAM devices.

10:40am PS+MN-WeM9 Key Challenges in Extremely High-Aspect-Ratio Dielectrics Etching at 3x nm DRAM and Beyond, S.K. Lee, J.-H. Sun, S.O. Lee, J.-S. Bang, S.-I. Lee, C.-M. Lim, S.-Y. Kim, D.-G. Lim, S.-K. Park, J.-G. Jung, HYNIX Semiconductor Inc., Republic of Korea

One of key issues in fabricating the dynamic random access memories (DRAM) is to control the vertical profile effectively during the etching of a SiO2 high aspect ratio contact holes (HARC). In order to ensure acceptable Cs (>25fF/Cell) for DRAM at half pitch (HP) 3x nm and beyond generation, it is required of fabricating cell capacitors having very highly aspect ratio above 50:1. Thus, the HARC etching technology to get smaller bowing width as well as larger opening area becomes the most difficult challenges among numerous DRAM fabrication steps. This is because of trade-off between both bowing and opening requirement during HARC etching. Although the mechanism of bowing and not-opening has reported in several studies at above 70nm technology nodes, still has not yet been reported at hp 3x nm and beyond. In this presentation, especially, we will focused on the HARC etching issues at Nitride Fence supported Capacitor (NFC) scheme which is used to prevent leaning. Capping is arose by several factors, which reduce the etch rates and cause the contact opening failure, then eventually affects on the electrical characteristics. The types of capping studied in this work can be divided into three categories as the etching proceeded, such as polymer pinch-off, excess polymer capping originating from polymer rich chemistry at top region, and non-steady polymer deposition and removal at etch front. In this study, we investigated that capping issues become more serious when 2MHz range power is added to increase contact opening margin. To avoid these types of different failures aforementioned, it is necessary to understand the plasma etching behavior at hp 3x nm and beyond compared to previous technology nodes. In addition, beyond typical bowing position, an additional bowing position at NFC is also key concern issues within oxide layer between hard mask (HM) and Nitride from the wafer top surface. This is caused by the ions scattered from the mask side-wall slope on the contact-hole. It can be reduced effectively by adjusting HM material, thickness, and etching conditions. Especially, HARC etching parameters also play an important role to suppress the bowing and capping. We will report here how contact hole's opening and bowing are enhanced, and how they can be controlled by adjusting etching conditions also. It is suggested that optimizing the etching condition with a suitable concept in this work would be the most effective solution during the HARC etching process. Consequently, Key approaches on HARC etch processes for fabricating of a contact hole in SiO2 with aspect ratios of 50:1 and beyond were evaluated in this work in detail.

11:00am **PS+MN-WeM10 Microstructures Etching on Silicon with the STiGer Process**, *T. Tillocher*, GREMI, France, *J. Ladroue*, GREMI -STMicroelectronics, France, *F. Moro, G. Gommé*, *P. Lefaucheux*, GREMI, France, *M. Boufnichel*, STMicroelectronics, France, *P. Ranson, R. Dussart*, GREMI, France

The STiGer cryoetching process can be alternatively used to the Bosch process or the cryogenic process to etch high aspect ratio structures. It has been developed thanks to our knowledge of the passivation mechanisms in cryoetching. In the standard cryogenic process, patterned Si substrates cooled down to very low temperatures are exposed to continuous SF₆/O₂ plasmas. A SiO_xF_y-type passivation layer is formed on the sidewalls and prevents etching. This film has the property to desorb under ion bombardment or when the substrate is heated. We also showed that SiF₄/O₂ plasmas can be used to create or reinforce a passivation layer in cryogenic etching.

The STiGer process consists of cycling passivation steps (SiF₄/O₂ plasmas) and etching steps to get vertical structures. The etching steps can be either isotropic (SF₆ plasmas) or anisotropic (SF₆/O₂ plasmas). Like the cryogenic process, it is required to cool the Si substrate with liquid nitrogen.

The STiGer process combines advantages of both Bosch process and cryogenic process. Due to the cyclic passivation steps, the SiO_xF_y film is stronger than in "standard" cryoetching. In addition, the passivation layer desorbs when the substrate is heated back to room temperature. Thus, unlike the Bosch process, there is no need to clean the microstructures and the chamber walls after each process run. Moreover, the robustness is enhanced

in comparison with "standard" cryoetching : the profiles are less sensitive to temperature variations.

But, like the standard cryogenic process, a cooling is required and like in Bosch etching, a scalloping is present on the sidewalls. However, it is possible to minimize this effect by tuning the etching and the deposition steps.

We will present our most recent performances with the STiGer process. Our objectives are to etch sub-micron trenches and holes that will be further used for the realization of integrated capacitors and Through Silicon Vias (TSV). But obviously, the STiGer process can be utilized for silicon micromachining in general.

Finally, we will see how such a process can amplify Columnar MicroStructures (CMS).

Thursday Morning, October 21, 2010

MEMS and NEMS

Room: Santo Domingo - Session MN-ThM

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

Moderator: R.B. Ilic, Cornell University

8:00am MN-ThM1 Mechanical Devices Incorporating Ultra-Thin Membranes, H.G. Craighead, Cornell University INVITED

8:40am MN-ThM3 Functionalized CMOS Nanomechanical Resonators for Trace Explosives Detection, J.W. Baldwin, Naval Research Laboratory, J.S. Burgess, National Research Council, M. Zalalutdinov, F.A. Bulat, Global Strategies Group, B.H. Houston, Naval Research Laboratory

We used functionalized CMOS integrated Nanoelectromechanical (NEM) resonators to selectively detect specific families of analyte. Unfunctionalized resonators have femtogram sensitivity in air, but have little selectivity. To address this problem, our resonators have been functionalized with adsorbate groups (e.g. perfluoroalkane and hexafluoroisocarbonol). These groups were bound to the surface using a spatially selective process using UV hydrosilation reactions. This process allows for the functionalization of the resonator without functionalized resonators showed enhanced selectivity. These functionalized resonators showed enhanced selectivity towards target molecules. For example, the hexafluoroisocarbonol group showed selectivity towards nitrobenzene but not towards cyclohexane or water. The operation of these devices in air, functionalization methods, and limits of detection will be discussed. This work was supported by the Office of Naval Research.

9:00am MN-ThM4 Performance of Nanomechanical Mass Sensors Containing Nanofluidic Channels, R.A. Barton, R.B. Ilic, S.S. Verbridge, B.R. Cipriany, J.M. Parpia, H.G. Craighead, Cornell University

9:20am MN-ThM5 Fabrication of Integrated Nanomagnets Overhanging Batch-Fabricated Attonewton-Sensitivity Cantilevers, J.G. Longenecker, S.A. Hickman, E.W. Moore, S.G. Lee, S.J. Wright, Cornell University, L.E. Harrell, United States Military Academy, J.A. Marohn, Cornell University

Mechanical detection of magnetic resonance opens up exciting possibilities for characterizing soft materials and biomolecules with elemental specificity at nanometer-scale, and potentially atomic-scale, resolution. Achieving atomic-scale resolution requires using cantilevers with a low minimum detectable force at small tip-sample separations and fabricating magnetic tips with only a few nanometers of damage at the leading edge. We address these challenges by 1) fabricating cantilevers with overhanging magnetic tips, 2) protecting the nanomagnet leading edge by atomic layer deposited (ALD) alumina, and 3) characterizing the extent and chemical mechanism of damage by nanometer-resolution electron energy loss spectroscopy (EELS).

After determining by EELS analysis that the nickel magnet leading edge incurred substantial damage during processing, we introduced tantalum and ALD alumina interdiffusion barriers into our forty-two step fabrication process. We demonstrate that these modifications have significantly reduced the damage layer thicknesses. The nanomagnet grain structure, point-by-point relative atomic concentrations at the leading edge, and magnetization are determined by high-resolution transmission electron microscopy (TEM), EELS, and frequency-shift cantilever magnetometry, respectively. We will also detail ongoing work to reduce the number of processing steps after magnet deposition, which could greatly improve magnet yield and quality. Our findings suggest that fabricating a cantilever suitable for single proton detection, while a materials processing challenge, should be possible.

9:40am MN-ThM6 Fabrication and Characterization Ultra-Sensitive, Nickel-Tipped Silicon Cantilevers for Magnetic Resonance Force Microscopy, S.A. Hickman, E.W. Moore, S.G. Lee, S.J. Wright, Cornell University, L.E. Harrell, United States Military Academy, J.A. Marohn, Cornell University

Magnetic Resonance Force Microscopy is a technique which combines the elemental discrimination and three-dimensional imaging of magnetic resonance with the spatial resolution of scanned probe microscopy. Key to this technique is attonewton-sensitivity mechanical oscillators with magnetic particles at the tip.

The aim of this work was to create silicon cantilevers with nickel magnets which extended past the tip of the cantilever body - a design conceived to minimize surface-induced force noise.

The fabrication challenges of this design, including alignment across multiple lithographic modes and silicide prevention, will be covered. As well, characterization data of successfully produced devices will be presented.

10:40am MN-ThM9 Parametric Excitation of Large Amplitude Outof-Plane Vibrations of Micro Beams By Fringing Electrostatic Fields, *S.L. Krylov*, Tel Aviv University, Israel, *N. Molinazzi*, Medica Group, Italy, *T. Shmilovich, U. Pomerantz, S. Lulinsky*, Tel Aviv University, Israel

We report on an approach for efficient parametric excitation of large amplitude flexural out-of-plane vibrations of cantilever and double-clamped micro beams and present results of theoretical and experimental study of the suggested principle. An actuating electrode is located symmetrically at the two sides of the beam and is fabricated from the same layer of the wafer. The beam is free to deflect in the out-of-plane direction, whereas its stiffness in the lateral in-plane direction is significantly higher. The distributed electrostatic force, which is zero in the initial configuration, is engendered by the asymmetry of the fringing fields in the deformed state and acts in the direction opposite to the deflection.

The force can be effectively viewed as a reaction of an elastic foundation, which increases the stiffness of the system. The time-varying voltage applied to the electrode results in the modulation of this electrostatic stiffness and consequently in the parametric excitation of the structure. The device is distinguished by a simple single-layer architecture and may exhibit large vibrational amplitudes, which are not limited by the pull-in instability common in close-gap actuators. In contrast to previously reported devices excited by the fringing fields, the force considered here is of distributed character. The reduced order model was built using the Galerkin decomposition with undamped linear modes as base functions and the resulting system of nonlinear differential equations was solved numerically. The electrostatic forces were approximated by means of fitting the results of three-dimensional numerical solution for the electric fields. The devices fabricated from a silicon on insulator (SOI) substrate using deep reactive ion etching (DRIE) based process combined with the critically timed etching were operated in ambient air conditions and the responses were registered by means of Laser Doppler Vibrometry. The experimental resonant curves were consistent with those predicted by the model. Theoretical and preliminary experimental results illustrated the feasibility of the suggested approach.

11:00am MN-ThM10 The Effect of Temperature on Etch Rate and Surface Roughness for Si Etched with Vapor Phase XeF₂, Z.C. Leseman, J. Butner, University of New Mexico

In this work we present results from a pulsed etching system with XeF2 for an expanded temperature range while at the same time determining the roughness of the substrate left behind. The experimental apparatus used for the work presented in this paper is capable of temperature ranges from approximately 100 K to 800 K. Data was taken at a constant etching pressure (1 Torr) so the effect of temperature on etch rate and roughness could be studied. Etch rates were determined by varying the duration of the pulse and surface roughness was characterized using an AFM.

11:20am MN-ThM11 Fabrication of High Density Single Crystal Silicon Nanowires for Ensemble Measurements, D.A. Czaplewski, L.E. Ocola, M.V. Holt, Argonne National Laboratory

Silicon nanowires have shown promise in applications such as photovoltaic cells, lithium storage for batteries, transducers, sensors, and many more. Single crystal silicon nanowires (SCSN) have been used to study materials and electrical properties of Si as the nanowires have been scaled down towards several nanometers. These experiments have been designed to test the classical predictions of materials behavior as the assumption of continuum mechanics starts to break down. Due to the small sizes of SCSNs, measurements of materials properties have been inconsistent due to the variation in dimensions and fabrication methods from wire to wire. Typically, SCSNs are fabricated at very low densities, which make measurements of ensembles very difficult. Here we present two top-down processes to create SCSNs at relative densities approaching 50% of a continuous film with dimensions as small as 30 nm. Both fabrication processes start with e-beam lithography. In the first fabrication process, a positive e-beam resist is patterned and developed. The resist is exposed to a second dose of electrons to increase the etch selectivity during reactive ion etching. The silicon structures are reactive ion etched in a CHF3-O2 plasma chemistry to define the structures. In the second approach, a negative ebeam resist, hydrogen silsesquioxane (HSQ), is patterned and developed on a thin thermally-cured HSQ layer. After development, the pattern is transferred into the silicon via a reactive ion etch using a Chlorine chemistry followed by a HBr-O₂ chemistry. After the wires are defined, they are subsequently released in a dilute HF-DI water mixture and then dried using a super critical CO₂ drying technique. The released structures are being used for studies of coupled mechanical oscillators and to study materials and electrical properties in ensembles of 1-D wires.

11:40am MN-ThM12 Analysis of a Dip-Solder Process for Self Assembly, M.R. Rao, J. Lusth, S.L. Burkett, University of Alabama

Dip-soldering is a crucial step in forming certain self-assembled metal structures. However, this particular use of dip-soldering is not well described in the literature. The goal of this work is to characterize the thickness and roughness of solder layers deposited by dipping metallic films into solder melt over a range of temperatures. Control of the solder thickness and roughness will improve the yield of structures whose selfassembly is driven by surface area minimization during solder reflow. Film thickness and overall film roughness for four solder alloys, each with different melting points, were measured on unpatterned and patterned copper films. Additionally, two variations in flux treatment were investigated: flux maintained at room temperature and flux preheated to 98 °C. Findings include the determination of critical temperatures, particular to each alloy, above which the roughness and thickness of the deposited solder dramatically decreases. Preheating the flux improves the nature of the deposition below these critical points. Above the critical points, thickness and roughness of the solder vary little and preheating the flux does not provide significant improvements. This study provides insight into designing a process flow that optimizes the folding characteristics of selfassembled metal polyhedra by controlling the volume and quality of the solder layer.

Thursday Afternoon, October 21, 2010

MEMS and NEMS

Room: Santo Domingo - Session MN-ThA

Integration, Packaging and Reliability of MEMS and NEMS

Moderator: M. Metzler, Cornell University

2:00pm MN-ThA1 Electrothermal Tuning of Bistability in a Large Displacement Micro Actuator, Y. Gerson, Tel Aviv University, Israel, R.B. Ilic, Cornell University, S.L. Krylov, Tel Aviv University, Israel

We report on an approach allowing efficient tuning of the bistability properties in large displacement micro actuators. The devices fabricated from highly doped silicon on insulator (SOI) wafers using Deep Reactive Ion Etching (DRIE) based process incorporate elastic suspension realized as a pair of initially curved beams and are operated electrostatically by a comb-drive transducer. The tuning principle is based on the control of the initial elevation and consequently of stability characteristics of the suspension by passing a current through the beams and electrothermal heating of the beams's material. Experimental results, which are in good agreement with the Finite Elements model predictions, demonstrate the feasibility of the suggested approach and show that the application of a tuning current significantly increases the device deflection and allows efficient control of the critical snap-through and snap-back voltages.

2:20pm MN-ThA2 Measured and Predicted Temperature Profiles along MEMS Bridges at Pressures from 0.05 to 625 Torr, L.M. Phinney, J.R. Serrano, E.S. Piekos, J.R. Torczynski, M.A. Gallis, A.D. Gorby, Sandia National Laboratories

We will present experimental and computational investigations of the thermal performance of microelectromechanical systems (MEMS) as a function of the surrounding gas pressure. Lowering the pressure in MEMS packages reduces gas damping, providing increased sensitivity for certain MEMS sensors; however, such packaging also dramatically affects their thermal performance since energy transfer to the environment is substantially reduced. High-spatial-resolution Raman thermometry was used to measure the temperature profiles on electrically heated, polycrystalline silicon bridges that are nominally 10 microns wide, 2.25 microns thick, 12 microns above the substrate, and either 200 or 400 microns long in nitrogen atmospheres with pressures ranging from 0.05 to 625 Torr. Finite element modeling of the thermal behavior of the MEMS bridges is performed and compared to the experimental results. Noncontinuum gas effects are incorporated into the continuum finite element model by imposing temperature discontinuities at gas-solid interfaces that are determined from noncontinuum simulations. The experimental and simulation results indicate that at pressures below 0.5 Torr the gas-phase heat transfer is negligible compared to heat conduction through the thermal actuator legs. As the pressure increases above 0.5 Torr, the gas-phase heat transfer becomes more significant. At ambient pressures, gas-phase heat transfer drastically impacts the thermal performance. The measured and simulated temperature profiles are in qualitative agreement in the present study. Quantitative agreement between experimental and simulated temperature profiles requires accurate knowledge of temperaturedependent thermophysical properties, the device geometry, and the thermal accommodation coefficient.

Sandia National Laboratories is a multi-program laboratory 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.

2:40pm MN-ThA3 Micro- and Nanoswitches: Materials, Design, Packaging and Reliability, *M.P. De Boer*, Sandia National Laboratories, *D.A. Czaplewski*, Argonne National Laboratory, *M.S. Baker*, Sandia National Laboratories INVITED

Ohmic micro- and nanoswitches are of interest in numerous potential applications including phased-array radars, cell-phone circuitry, circuit breakers and power savings in advanced CMOS circuits. However, many challenges remain with respect to materials, design, packaging and reliability. An important issue is maintaining a low contact resistance as switch cycling approaches high counts. In this talk, a prototype ohmic microswitch will be presented, and its electrical performance when coated by Pt and Ru films will be compared. A whole-wafer singulation, post-processing and metallization process will be demonstrated. Then, a nanoswitch design will be introduced and initial results including processing and test will be discussed. Continued progress in micro- and nanoswitch technology will lead to insertion in multiple applications.

Sandia ia a multiprogram laboratory operated by Sandia Corporation, a Lockheed-Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

3:40pm MN-ThA6 Bulk Focused Ion Beam Fabrication of Nanoelectromechanical Systems, W.K. Hiebert, D. Vick, V. Sauer, National Institute for Nanotechnology (NRC Canada), A.E. Fraser, M.R. Freeman, University of Alberta, Canada

Although the focused ion beam (FIB) has previously been used for fabrication of nanostructures and devices, including MEMS and NEMS, FIB nanomilling out of bulk material has rarely been discussed. In this talk, we will present our methods and results for using FIB to fabricate NEMS devices out of bulk materials. Ion impingement from multiple directions allows sculpting with considerable 3-dimensional control of device shape, including tapering and notching. The tailoring of large gaps between device layer and bulk allows large amplitude NEMS motion, access to a nonlinear readout regime, and a novel calibration method for optical interferometric displacement detection. Finite element modeling of device frequencies agrees with interferometric measurements, including for the effect of a localized notch. The measurements are sensitive enough to determine the thermomechanical noise floor of a bulk FIBed NEMS device with displacement sensitivity of 166 fm / $Hz^{\frac{1}{2}}$, limited only by a combination of optical shot noise and detector dark current. We envision that bulk FIB fabrication will be useful for NEMS prototyping, milling of tough-tomachine materials, and generalized nanostructure fabrication with 3dimensional shape control.

4:00pm MN-ThA7 NanoFIBrication of UHAR Vias Using a Material Shaping Technique for Phononic Crystals, *D.F. Goettler*, *Z.C. Leseman*, University of New Mexico

In this paper we present both experimental and theoretical results showing the effective use of material shaping to fabricate ultra-high-aspect-ratio (UHAR) vias with a focused ion beam (FIB). With this technique, one can create vias with aspect ratios of 50:1 and higher. This is achieved by placing a 'lower sputter rate' material on top of a 'higher sputter rate' material. We model the FIB as a Gaussian beam with an angular dependent sputter rate. With our model we predict a high sputter rate ratio (high/low) can achieve vias with aspect ratios near 50:1. Experimental results support this prediction. By placing a thin layer of pyrolized carbon on top of silicon, we fabricated UHAR vias. For completeness, we also reversed the sputter rate ratio by placing a 'higher sputter rate' material on top of a 'lower sputter rate' material. Once again, experimental results support the model's predictions. Vias with radii 15 nm have been NanoFIBricated. Using these techniques we have created phononic crystals operating in the GHz regime.

4:20pm MN-ThA8 Highly Robust Hydrogen Selective MEMS Nanogap Sensor Utilizing the Schottky Barrier at Electrode/Sensing Material, A. Kumar, P. Zhang, H.J. Cho, S. Seal, University of Central Florida

The growing need to explore hydrogen as a near future fuel demands a robust hydrogen sensor which offers high sensitivity, selectivity and response time in order to avoid the danger associated with storage, transportation and use of this highly combustible gas. The sensor platform with Au interdigitated electrodes (IDE) having 8 fingers in each electrode and a gap of 100 nm was fabricated using E-beam lithography on a silica substrate. The Au IDE was dip coated with sol-gel preparation of nanocrystalline 6.5 mol % Indium oxide (In₂O₃)- doped tin oxide (SnO₂) to yield an excellent thin film room temperature hydrogen sensor. The variation in the I/V response of the sensor with atmosphere suggested that the Schottky barrier height could be modulated to sense hydrogen and utilizing this a large sensitivity (~2000) and fast response time (~27 seconds) was observed at a low applied voltage of 0.4 V in 0.09 vol% hydrogen gas atmosphere. The sensing characteristics were severely affected in presence of moisture (>40%). Various polymeric coatings on the sensor were compared in an effort to make the hydrogen sensor robust even in high moisture environment. It was observed that the fluoropolymer coatings improved the sensor behavior in varying moisture environment without deteriorating other characteristics such as sensitivity, response time and recovery of a sensor.

4:40pm MN-ThA9 Development of Lab-on-a-chip on Mn Induced Nano-arrayed Structures in Sol-gel Derived TiO2 Platforms for Biosensing Applications, *R.R. Pandey*, Centre for Cellular and Molecular Biology, India, *K.K. Saini*, National Physical Laboratory, India, *M. Dhayal*, Centre for Cellular and Molecular Biology, India

Development of low cost point-of-care diagnostic system for effective health care is important for rapid screening of routing biochemical tests. To address this, we have used sol-gel derived process in which Mn induced nano-arrayed structured were developed in TiO2 as a platforms for lab-ona-chip. Mn doped nanopore TiO2 platforms were prepared by using wet chemistry deposition with the help of metal alkaloxide precursor and assembled with micro-fluidics networks to screen multiple interactions. To demonstrate usefulness of this, enzymatic biocatalyst has been used to determine the specificity of multiple interactions on these platforms. These platforms has been characterized by XRD, XPS, FTIR, SEM, cyclic voltametry to determine structure, surface chemistry and electron transfer characteristics for biosensor applications. Mono-enzyme ChOX, urease and GOX were prepared by immobilizing the separate enzymes onto nanopore TiO2 surfaces modified with Mn doping. The electrochemical detection sensitivity for detection of low concentrations of cholesterol, urea and glucose has been enhanced.

Thursday Afternoon Poster Sessions

MEMS and NEMS Room: Southwest Exhibit Hall - Session MN-ThP

MEMS and NEMS Poster Session

MN-ThP1 A Novel Micro-droplet Proteomic Identification Chip for Protein Digestion and MALDI-TOF MS, *T.T. Huang*, Instrument Technology Research Center, Taiwan, Republic of China

Proteomic identification at the point of care would be valuable for a wide variety of applications and importance, including clinical diagnostics, food safety, and environmental monitoring. Traditional proteomic identification makes use of protein digestion of interesting sample and could be detected using matrix assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF MS). Protein digesting reaction in vitro often wastes much time (usually overnight) and reagent volume, and then the reaction products should be transferred to the MS sample plate and air-dried. The analysis process is not effective for convenience and wastes time and analysis sample.

For more effective protein identification, we construct a novel microdroplet chip system with auto-positioning and enriching the sample for rapid analysis. Presently, MALDI-TOF MS has been widely used in proteomic research, and it is the important issue to identify the micro sample. Utilizing the novel micro-droplet chip, protein sample would be cocrystallized with MS matrix and concentrated on the detection area. Through drying the sample and matrix mixture, protein digestion would be reacting at the same time. The sample would be digested to smaller dried peptides on chips and then detected with <u>matrix assisted laser</u> <u>desorption/ionization time-of-flight mass spectrometry (MALDI-TOF MS)</u>. Comparing to the traditional methods, the proteomic identification chip offers four advantages: 1. shortening reaction and analysis time (total ~2.5 hours); 2. auto-positioning and enriching the sample concentration; 3. lower reaction volumes (1~5 μ L); 4. entire process sequentially on the chip.

MN-ThP2 Test Instrument for the Mechanical Strength of Micro-Nano Materials, A. Kasahara, H. Suzuki, M. Goto, H. Araki, M. Tosa, National Institute for Materials Science, Japan

There is considerable research at present on the performance and properties of nanosheets, nanofibers and other functional nanomaterials such as fullerenes and nanotubes. This is particularly true of carbon nanotube, made from carbon atoms, where many research projects throughout the world are looking at measurement techniques for evaluating electrical and electronic characteristics with a view to developing electronic device applications such as high-intensity field-emitted electron sources and ultra-fast transistors. 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. 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 nanoscale equivalent of mechanical strength testers for ordinary materials. This is due to the inherent difficulties associated with the manipulation and transportation of materials at the micro-nano scale level. The most difficult part of the handling of small materials is to fix material samples.

We have improved it to the device with an easy fixation of the sample from the last time. Here, we will discuss our recent results on mechanical strength measurement of micro-nano wires in diameter several nm through several thousand nm and in length several mm by means of prepared micronano tensile strength tester device.

MN-ThP3 A Micro-droplet PCR Device with Low Volume Reaction and Rapid Amplification, *T.T. Huang, Y.C. Ou*, Instrument Technology Research Center, Taiwan, Republic of China

Nucleic-acid amplification and analyses techniques have become the most significant tools for many important applications. Besides the frequent molecular diagnosis of diseases and assessments of therapies in clinics and hospitals, they are also broadly applied in environment surveillances, food processing industry and agricultural researches. The amplification of the amount of nucleic-acid analyte in the test sample is essential for sufficient detection. Polymerase chain reaction (PCR) is the major process to amplify interesting nucleic-acid. However, it always spends too much analyte volume and 3~4 hours for carrying out the reaction. In the study, we construct a novel micro-droplet PCR device and PCR chips utilizing MEMS techniques.

The temperature variation of the micro-droplet PCR device achieves 1 °C per second. Moreover, we develop the micro-droplet PCR chips fabricated by silicon wafer with two concentric circles structure. Then, the chip is coated with a layer of PPFC (plasma polymerization fluorocarbon) film on its surface. Utilizing the MEMS process, the solution of nucleic-acid reactants is concentrated in the inner circle and 5µL the mineral oil in the outer circle is covered on the reaction solution. Comparing to the traditional PCR method, the micro-droplet PCR device and chips have the advantages of extremely low-volume reaction solution (only $1\sim$ 2µL) and rapid amplification time (~30 minutes). The novel micro-droplet PCR device is quite promising to efficiently DNA amplify.

MN-ThP4 Boundary Slip and Nanobubble Study in Micro/Nanofluidics with Atomic Force Microscope, B. Bhushan, The Ohio State University

The boundary condition at the liquid-solid interface in micro/nano scale is an important issue in micro/nanofluidics systems. Recent studies have shown that the fluid velocity near solid surfaces is not equal to the velocity of the solid surface on hydrophobic surfaces, which is called boundary slip. The degree of boundary slip is evaluated by a slip length. Theoretical and experimental studies suggest that at the solid-liquid interface, the presence of nanobubbles is responsible for the breakdown of the no-slip condition. Nanobubbles are long lasting on hydrophobic surfaces, and movement and coalescence of nanobubbles are observed with higher scan loads during imaging with tapping mode AFM.

In this study, both contact and dynamic AFM methods have been applied to study the boundary slip on hydrophilic, hydrophobic, and superhydrophobic surfaces. An AFM based technique is developed to study boundary slip. Nanobubble movement and coalescence, as well as tip-bubble interaction, are studied in detail. The physical interaction between nanobubbles and the surfaces supporting them is investigated.

MN-ThP5 Rapid Point-Of-Care (POC) Diagnostics by Droplet-Based Detection Instrument, C.S. Yu, Y.C. Hu, H.-S. Huang, J.-S. Kao, ITRC, Taiwan, Republic of China

This paper proposes a novel point-of-care (POC) instrumentation, provides fast, simple, low cost detection and easy used. This instrument including three main parts, micro fluid chip module, electronic module and optics detection module. We use the standard the LIGA-Like process to complete the micro fluid chip. Metal master which using the electroplating technology, the massive manufactures uses the Hot-Embossing process. We characterize a gradient of surface tension force to manipulation droplet and a droplet-based reagent can be transported, precisely positioning, and mixed on the detection zone without any power source. A variety of applications are also possible such as food testing, drinking water testing, alcohol testing, anti-oxidation measurement and cosmetic analysis.

MN-ThP6 Absorption and Emission of Plasmonic Antenna Arrays, K.E. O'Brien, M.R. Davidson, P.H. Holloway, University of Florida

New and more portable means of generating narrow band radiation is 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 and 3-dimensional arrays of theses resonant structures fabricated on Ag thin films using electron-beam lithography and lift-off. Patterns vary from arrays of structures of identical lengths and widths, to those with alternating and increasing lengths and widths. 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 arrays has been measured. The effect of different antenna structures on the in absorption and emission will be discussed.

MN-ThP7 Biomimetic Application of Localized Hydrophobicity for Increased Drag Reduction Performance on Shark Skin-Inspired Riblet Surfaces, B. Dean, B. Bhushan, The Ohio State University

The skin of fast swimming sharks exhibits riblet structures aligned in the direction of flow which are known to reduce skin friction drag in the turbulent flow regime. Fish secrete mucus through their skin which greatly reduces drag during swimming. Small amounts of mucus have been seen on the scales and riblets of fast swimming sharks, which affect the near-skin flow properties in some way. The drag reduction effect of this trace amount of mucus on the surface of the shark skin is approximated by the slip length present in water flowing over a hydrophobic surface. A biomimetic study is done in which the drag reduction benefit of localized and non-localized hydrophobic surface applications onto drag reducing riblet structures are

studied. The combined effect of turbulent drag reduction by localized or non-localized hydrophobicity and by riblet mechanisms is presented.

MN-ThP8 Development of High-Density Cylindrical Ion Trap Array for Mass Spectrometer, *T. Wu*, *A. Chaudhary, F. Amerom, T. Short, J. Wang*, University of South Florida

This paper presents the development of high-density cylindrical ion trap array for mass spectrometer (CIT-MS). The previous research has mainly focused on the adjusting the ring electrode radius r_0 , cylinder length z_0 , and endplate hole electrode r_H (Figure 1) and developing a fabrication process involving a back-to-back bonding of two half CIT structures. However, recently it has been noticed that a key factor that limits the performance of the CIT is the high capacitance between endplate electrodes and ring electrode due to the small gap; besides, the back-to-back bonding of two half structures could bring a maximum 5 micron misalignment. Based on these concerns, a new geometry of CIT array for mass spectrometer has been designed; also a new fabrication process has been developed accordingly.

In the newly developed generation of CIT-MS, the geometry design of CIT has been focusing on increasing the gap between the ring electrode and endplate electrode. Several simulations have been done on this subject. Other important improvements include better ring-to-endplate aperture alignment using dedicated alignment marks during flip-chip bonding, hexagonal orientation of traps leading to smaller pitch between each trap to increase density (thereby more trapping volume per unit area of wafer), larger vacuum gaps for operation at higher voltages for increased mass range and selective metallization using lithography techniques to reduce the overlap area of ring and endplate electrodes for lower capacitance.

A new fabrication process has been designed to achieve the improvements mentioned above. A suspended endplate electrode structure, using KOH/DRIE etching techniques, was used to increase the gap and reduce the overlapping area at the same time. To avoid surface charging of the dielectric surface that is exposed to ions, Atomic Layer Deposition (ALD) was investigated to deposit highly resistive (ZnO) layer on the dielectric surface to dissipate charge, while adding minimum to the capacitance. Flip-chip bonding was used to bond the ring-electrode and endplate electrodes and minimize the misalignment between the two substrates. With these considerations, both the esolution specificity and sensitivity are expected to improve. The design of optical mask for this process is underway and we report preliminary progress based on the latest findings.

MN-ThP9 Development of Optimum Ti/TiN Dark Reference Structure to Improve Dark Leakage Characteristics in CMOS Image Sensor, *S.*-

Y. Kim, Korea Polytechnic College IV, Republic of Korea, N.-H. Kim, Chonnam National University, Republic of Korea, K.-G. Oh, Chosun University, Republic of Korea

Dark leakage is one the most effective factors influencing the characteristics of CMOS image sensor (CIS), which makes the unrelated signals instead of the when the low intensity of illumination lighted up to image sensors. To solve this problem, there are so many efforts into changing the designs and fabrication processes. One of these efforts is the usage of dark reference to improve the dark leakage characteristics. In this study, the CIS including dark reference was fabricated. The dark reference located in the edge of the valid pixel makes no sigmal from light through perfectly blocking out light, which is used to by using the metal thin films. For easy fabrication and excellent stability, Ti/TiN structure was employed in this experiment. The dark reference by Ti/TiN structure showed the lower leakage characteristics the under-lying photodiode than that of the valid pixel in the general H₂ annealing time (30 min). The dark leakage characteristics were improved by increasing the H₂ annealing time; however, the increased annealing time lead the fabrication ability and yield to be lower. Therefore, the optimized thickness (150 nm) of Ti/TiN structure were obtained by theoritical estimation for the under 0.01% transmittance. In experiment, the leakage characteristics were improved in the conventional H₂ annealing time by decreasing the thickness of Ti in Ti/TiN structure. Consequently, the dark leakage characteristics and SNR of CIS were improved by optimization of Ti/TiN thickness with the short fabrication/annealing time.

MN-ThP10 Improvement of Optical Properties in 3D CMOS Image Sensor (CIS) by Using Insertion Structure of Metal Slot, S.-Y. Kim, Korea Polytechnic College IV, Republic of Korea, G.-M. Han, N.-H. Kim, Chonnam National University, Republic of Korea

High integrated CMOS image sensor (CIS) has contimuously decreased the area of photodiode in CIS structure. This is the origitnated reason to decline the optical characteristics of CIS including optical generation collection efficiency (CE), and crosstalk. Although the conventional structure used the PD structure with shallow trench isolation (STI) for pixel-to-pixel isolation, this structure brought out the high leakage characteristics. To improve this leakage problem, the conventional process employed the counter-doping

method of impurities; however, this method could not solve the crosstalk problem by the obliquely incoming light. In this study, both the counterdoping of impurities and insertion of metal slot into the center of counterdoping were proposed to form the pixel-to-pixel isolation. This structure carried out the role of wave-guide with the excellent light reflection characteristics of metal as well as the conventional ground of contact. The crosstalk issue was successfully improved with the enhancement of the optical generation characteristics of 3D CIS by using this novel structure instead of the conventional STI pixel-to-pixel isolation method.

MN-ThP11 Modeling of Diffusion, Nucleation and Growth in the Chemical Vapor Infiltration of Vertically-Aligned Carbon Nanotube Forests for MEMS, A.M. Konneker, D.D. Allred, R.C. Davis, Brigham Young University

We present preliminary results using computer simulation of chemical vapor deposition into vertically aligned carbon nanotube (VACNT) forests. The model is based upon deposition processes used in the carbon nanotube templated microfabrication (CNT-M) process. It utilizes VACNT arrays as a framework into which matter is infiltrated via a chemical vapor deposition (CVD)-type process to create solid microstructures and microelectromechanical systems (MEMS). These can have large aspect ratios (200:1) and startling heights (to date up to a mm). Long nozzles with narrow ID and other structures have been made that would be difficult by subtractive techniques. One of the biggest advantages of CNT-M is the promise of being able to prepare MEMS from any desired solid material that can be deposited by CVD, though to date this has only been amorphous and polycrystalline Si, SiO2, SiC, silicon nitride and amorphous carbon.

This work was inspired by the desire to understand how the gas diffusion, reaction and nucleation and growth models used to describe, mostly 2-D, thin-film CVD can be applied to understanding growth on the individual carbon nanotubes and nanotube bundles that make up a VACNT structure. This scaffolding is 3-dimensional, but "rarified"- that is > 99% empty and geometrically complex, and is vital for use of the CNT-M process. How it changes from mostly emptiness to a filled structure was a puzzle. Without optimized growth parameters, deposition is limited to the exterior of the VACNT forests and the structural integrity of the MEMS is poor. In addition, the adhesion of the MEMS devices to the substrate is often inadequate, which leads to low yields and frequent device failure.

Our model explores how the deposition rate, VACNT geometry, and VACNT forest density affects the filling of the forest. We base the parameters in our model on data from the TEM analysis of MEMS devices fabricated using the CNT-M process with CVD-deposited polysilicon, amorphous carbon, and silicon nitride.

Friday Morning, October 22, 2010

Biomaterial Interfaces Room: Taos - Session BI+MN-FrM

Sensors & Fluidics for Biomedical Applications

Moderator: S.L. McArthur, Swinburne University of Technology, Australia

8:20am **BI+MN-FrM1** Release of Biomolecues from a Photovoltaic Device for Targeted Drug Delivery, *S.L. Ambure*, University of Texas at el Paso, *D. Terreros*, Texas Tech University Health Sciences Center, *T. Xu*, University of Texas at el Paso

Introduction

An important goal of targeted drug delivery is to minimize the exposure of normal tissues to the drugs while maintaining their therapeutic concentration in diseased parts of the body. However, current methodologies are not yet ideal for such goal; therefore, new strategies for targeted drug delivery are needed. A photovoltaic device (PD) is a system that converts lights into electricity as well as induces charge transfer by photovoltaic effect. Motivated by such unique property, we have hypothesized that a PD can serve as a new drug delivery system to carry chemotherapeutic drugs and release them upon external photo stimulation, such as near Near-Infrared (NIR) light or Laser source. Taking advantage of repulsion between a photovoltaic device and a substance is proposed to serve as a new drug delivery method. In this study, we have investigated if the if the charged molecules can be effectively released from the PV device upon photon stimulation.

Methodology

As proof of principle, we have first experimented coating of commercially available photovoltaic devices with positively charged poly-l-lysine and negatively charged bovine serum albumin (BSA) tested the release of the molecules upon photo stimulation. These molecules were physically absorbed onto the surface of the PDs before exposed to an IR LED illuminator, which was used as an external light source. Moreover, the pure glass slide was used as a control of the device with non-photovoltaic effect for drug delivery.

Results and Discussion

During the series of experiments, we have found that, the PD has a capability to release the charged drugs by photovoltaic effect. Compared to no light stimulation, the positively charged poly-l-lysine and the negatively charged BSA when exposed to IR illuminator for 3 hours were released about 2.0 folds and 2.1 folds respectively. Moreover, in the control group (pure glass slide with no photovoltaic effect), there was no significant release of both poly-l-lysine and BSA when exposed to the IR illustrator.

Conclusion

These data showed that the new PD can effectively carry either positively or negative charged molecules on its surface and release them upon external photo stimulation, which suggests the PD has potential to be used as a new drug delivery system to carry cancer chemotherapeutic drugs. Further experiments are planned on the micro-fabricated photovoltaic devices (Size $300\mu m$ to $500 \mu m$). With this project a new approach for targeted drug delivery with micro- photovoltaic devices will be developed.

(*Proprietary: an IP application based on this study is pending)

8:40am **BI+MN-FrM2** Enzymatic Activity Enhancement on Nanostructured **TiO₂** Platforms by Ru doping for Biosensor Applications, *R.R. Pandey*, Centre for Cellular and Molecular Biology, India, *K.K. Saini*, National Physical Laboratory, India, *M. Dhayal*, Centre for Cellular and Molecular Biology, India

Devices based on nanomaterials platforms are emerging as a powerful tool for ultrasensitive sensors for the direct detection of biological and chemical species. In this work, we will report the preparation and the full characterization of Ru doped TiO2 nanostructured platforms those have been used for electrochemical detection of enzymatic activity for biosensor applications. Ru doped TiO2 platforms were prepared by sol-gel deposition onto conducting substrates and as test model cholesterol oxidase, urease and gucose oxidase were loaded onto the nanostructured platforms. Cholesterol /Urease/Glucose oxidase immobilized onto Ru doped TiO2-based nanostructured surfaces exhibited a pair of well-defined and quasireversible voltammetric peaks in CV measurements. The electron exchange between the enzyme and the electrodes was greatly enhanced in the Ru doped TiO2 nanostructured environment. The electrocatalytic activity of cholesterol, uresae and glucose oxidase embedded on Ru doped TiO2 electrodes had

enhanced significantly toward detection of low concentrations of cholesterol, urea and glucose.

9:00am BI+MN-FrM3 A Microfluidic Single Cell Isolation Device for Ensemble Measurements of Viral Hemorrhagic Fever Pathogenesis in Macrophages, M.W. Moorman, J.B. Ricken, R.F. Renzi, R.P. Manginell, C.S. Branda, O.A. Negrete, C.D. James, B.D. Carson, Sandia National Laboratories INVITED

Arenaviruses are a particular class of viruses that cause lethal hemorrhagic fever in humans, and a fundamental problem in understanding their pathogenicity is that many effects of viral infection are not mediated directly by the virus itself (primary immune response) but by the response of the immune system (secondary immune response). Thus, population level experiments on cells make it difficult to elucidate the timing of signaling events during pathogenesis in order to lay the groundwork for improved antiviral therapeutics, vaccines, and biological countermeasures. Our objective here is to deconvolute the pathogenic response by isolating and infecting individual macrophage host cells followed by real-time measurements of response-critical cytokines. We have developed a microfluidic cell isolation platform that can trap up to 150 individual host cells in fluidically isolated microchambers. The chip design eliminates chamber-to-chamber fluidic communication, thus signaling molecules that are secreted from infected cells are prevented from interacting with uninfected cells. This configuration allows us to differentiate between primary and secondary immune response when compared to bulk cell population level studies. The chip is made with a three level reactive-ion etch process in silicon that produces trapping features that place cells adjacent to an anodically-bonded coverslip to permit high-resolution confocal imaging. The microfluidic device is operated with a custom pressure controller system that permits computer-controlled delivery and routing of up to 10 different reagents. Currently, we are using experimental and computational techniques to identify the mechanisms by which arenaviruses provoke lethal cytokine production in host cells. This is accomplished with a fluorescent reporter fusion construct that we developed to measure the cytoplasm-to-nucleus translocation dynamics of a transcription factor. This construct allows us to assess the early (< 1 hour) response of host cells to viral infection and when combined with a second reporter construct for real-time monitoring of cytokine induction, we are also able to monitor a late (>1 hour) immune response event. Initial studies using a viral mimic challenge showed oscillation of the transcription factor in and out of the nucleus over the first several hours of pathogen exposure, and a rapid 2X increase in cytokine induction over the first five hours postinfection. Future work will use a live Pichinde virus to examine transcription factor (NFkappaB) translocation and cytokine (TNFalpha, IFNbeta) induction dynamics.

9:40am **BI+MN-FrM5** Elastomeric Microparticles that Exhibit Negative Acoustic Contrast in Bioassays, K.W. Cushing, M.E. Piyasena, B.A. Lopez, N. Carroll, T. Woods, D.N. Petsev, S. Graves, University of New Mexico, G.P. Lopez, Duke University

The development of more sensitive and rapid medical assays is imperative to decreasing time-to-diagnosis in diseased state individuals, and thus to improving patient outcomes. Enhanced detection limits afford the ability to detect the presence of low concentrations of biomarkers that may be present during the early-onset stages of a disease. Reduction in sample preparation requirements can further decrease assay time and the expertise level of the user. Our research aims to develop a sensitive and rapid bioassay platform using elastomeric capture microparticles (ECµPs) coupled to an acoustic sample preparation chamber and a flow cytometer. ECµPs possess unique physical and mechanical properties enabling the separation of ligand-bound ECµPs from biological particles (e.g., red blood cells) within a collected fluid sample (e.g., whole blood) by placing them under acoustic pressure. ECµPs have acoustic properties (negative contrast) that allow their positioning separately from many biological particles, which typically exhibit positive contrast, (e.g., cells) within an acoustic pressure field. Hence, an in-line acoustic sample preparation instrument can be used to separate unwanted biological particles (along with interfering soluble molecules) from ligand-bound ECµPs within biological fluids, The acoustic field will also concentrate the ECµPs, and so combined with removal of unwanted biological particles, much higher analysis rates of ECµPs may be possible. Since the acoustic sample preparation system operates continuously and can be mounted to feed separated ECµPs directly into a flow cytometer, this approach may decrease sample preparation time. Our studies show that simple emulsion polymerization methods using commercially available silicone precursors can be used to easily form elastomeric microparticles that exhibit negative contrast.

10:00am **BI+MN-FrM6 Optimization of Biosensors using Selective Chemistry**, *O. Seitz*, *P.G. Fernandes*, University of Texas at Dallas, *H.C. Wen*, Texas Instruments, Inc., *H.J. Stiegler*, *R.A. Chapman*, *E.M. Vogel*, *Y.J. Chabal*, University of Texas at Dallas

There is currently a strong need to develop sensitive and reliable biosensors, based on electronic detection, such as field-effect transistors (FET). Most of the focus has been on improving sensitivity by decreasing the FET channel size, using nanowires instead of similar devices on planar silicon. Issues of silicon functionalization, important for device reliability have been mostly ignored.

In this work, we present a robust approach to functionalize the channel region of a SOI wafer, thus achieving better reliability and sensitivity to very low analyte concentrations. The process leads to attachment of active SAM on oxide-free (H-terminated) silicon through formation of a Si-C bond on the channel. Combining IR absorption (IRAS) and X-ray photoelectron (XPS) spectroscopies, photoluminescence, atomic force microscopy (AFM) and electrical measurements, we find that this configuration results in a stable device where the active SAM is more strongly attached to the Si than silane molecules do on oxides. This functionalization is achieved by immersion in carboxylic acid (COOH)terminated alkene molecules to functionalize the H-terminated channel. After processing, XPS and IRAS confirm that the channel remains oxidefree, that the packing of the SAM on the channel is dense. Photoluminescence measurements confirm the high quality of the interface on the channel where non-radiative recombination (interface states) is not detected. The AFM pictures confirm that active molecules attach to the channel (imaged by attachment of nanoparticles). Electrical measurements, on these improved devices, indicate excellent response for both pH and protein sensing with sensitivity at least as good as the one of similar structure with a uniform SAM functionalization (i.e. using oxidized Si channels).

10:20am **BI+MN-FrM7** Nanopatterned Pores on a Gel-supported Membrane For On-chip Sample Preparation in Surface Plasmon Resonance Sensing. *G.R. Marchesini*, Joint Research Centre, Italy, *S. Rebe Raz*, Wageningen University, the Netherlands, *M.G.E.G. Bremer*, RIKILT – Institute of Food Safety, *P. Colpo, G. Giudetti*, Joint Research Centre, Italy, *W. Norde*, Wageningen University, the Netherlands, *F. Rossi*, Joint Research Centre, Italy

We present a novel approach to tackle the most common drawback of using Surface Plasmon Resonance for analyte screening in complex biological matrices - the nonspecific binding to the sensor chip surface.

By using a perforated membrane supported by a polymeric gel structure that exceeds the evanescent wave penetration depth, we created a filter above the sensing region that prevents the diffusion of large particles or aggregates that bind non specifically to the polymeric gel and interfere with SPR sensing, thus increasing assay's sensitivity, reducing sample preparation steps and shortening the analysis time in total. A 10 nm thick non-fouling membrane with nanopatterned macropores was fabricated by means of colloidal lithography and plasma enhanced chemical vapor deposition of polyethylene oxide-like films. Such a membrane was supported by carboxymethyldextran, a polymeric gel matrix commonly used in surface plasmon resonance imaging, contact angle, atomic force microscopy and scanning electron microscopy.

The performance of this surface in full fat milk and porcine serum was studied using an antibiotic detection immunoassay as a model system. Structurally, the 91.7 \pm 14.7 nm diameter pores presented an hexagonal crystal lattice and a clearance of about 5 % of the total surface. Functionally, the nanopatterned macropores showed significant improvements in the quality of the obtained measurements in comparison to bare carboxymethyldextran, displaying 100 fold increase in the limit of detection of the enrofloxacin bioassay when performed in porcine serum.

10:40am **BI+MN-FrM8** Stability and Selectivity of Biorecognition **Proteins Immobilized on Diamond Surfaces**, *R.J. Hamers, C.J. Stavis*, University of Wisconsin-Madison, *A. Radadia, R. Brashir*, University of Illinois at Urbana-Champaign, *J.A. Carlisle*, Advanced Diamond Technologies, *W.P. King*, University of Illinois at Urbana-Champaign, *H. Zeng*, Advanced Diamond Technologies

The use proteins, such as antibodies, for the detection of target biological species in water supplies or with *in situ* medical diagnostics will require immobilization of these proteins on surfaces that resist non-specific adsorption and maintain the protein's activity over time. Ultrananocrystalline diamond (UNCD) thin films are a promising material that may address several major challenges for the next generation of biosensors including detection of cellular mass loading, stability throughout multiple uses and regenerations of the sensor surface, and use at elevated temperatures.

We are currently investigating the chemical functionalization of diamond thin films with antibodies for selective recognition and detection of biological cells, using E. coli as a model system. Infrared spectroscopy and X-ray photoelectron spectroscopy measurements have been performed to characterize the covalent attachment of antibodies to the surface and to quantitatively characterize the antibody surface attachment via the N(1s) and S(2p) levels. To determine the factors controlling selectivity and stability, we have performed time-dependent cell capture studies and have correlated the time-dependent changes in cell capture efficiency with corresponding measurements of the surface composition. These measurements are used to establish whether long-term stability and selectivity for biomolecular recognition is limited by loss of the ligands directly linked to the diamond substrate, by removal of the biological layer, or by alteration of the antibody structure. Infrared measurements of the Amide I band is particularly useful in characterizing changes in the antibody secondary structures. These studies provide important fundamental insights into the chemical factors that control biological interactions at surfaces and provide guidance on efforts to make ultra-stable biological sensing platforms.

11:00am BI+MN-FrM9 Saturated Ionic Conductance at Low Electrolyte Concentration Through Solid-State Nanopores, P. Waggoner, H. Peng, S. Harrer, B. Luan, S.M. Rossnagel, IBM T.J. Watson Research Center

It has been observed that ion currents pass through biased nanopores in electrolyte solutions with bulk conduction properties until the concentration of ions decreases beyond a certain level. At this critical point, the conductance of the pores saturates at a constant value despite further decreasing the concentration of ions, an effect that has been attributed to charge shielding effects of surface charges within the pore. Below the critical concentration, the Debye length associated with screening the surface charges becomes larger and larger compared to the size of the pore and effectively cuts off bulk conduction. However, the conductance of the pore is not affected by the continuing growth of the space-charge region with the Debye length. In the following we present a model describing the nanopore-electrolyte system with an analytical solution that explains the experimentally observed behavior for five different salt solutions at low concentrations, including KCl, KCl in 50% glycerol, Tris-EDTA buffer, phosphate buffered saline, and CaCl₂. Conduction through the nanopore at low ion concentrations is analogous to hole extraction in reverse biased diodes and is related only to the diffusion of the minority carrier into the space charge region where it is then accelerated through the nanopore. These results also have important implications for solid-state nanopores being applied for DNA detection and sequencing technologies.

11:20am **BI+MN-FrM10** Fabrication of *in situ* Oligonucleotide Arrays by Inkjet Printing and their use in Gene Assembly, *I. Saaem, J. Tian*, Duke University

In our studies, we utilized an inkjet based in situ oligonucleotide synthesis platform that uses salvaged printheads from commercial printers. The platform utilizes standard four-step phosphoramidite chemistry with some modifications in order to synthesize oligonucleotides on functionalized substrates. A sensitive pressurization system is used to ensure print quality and an on-board vision system enables substrate registration and synthesis monitoring. Using this platform we synthesized oligonucleotide on prepatterned functionalized plastic slides. Such patterned substrates help in proper droplet formation and fluid mixing on the surface while mitigating satellite and irregular drops, which can lead to cumulative synthesis errors. Functional integrity of synthesized oligonucleotides was confirmed by hybridization with complementary strands. Being able to hot emboss microfluidic structures directly onto plastic slides in combination with the ability to generate arbitrary sequences provides diagnostic capabilities as well as the means to harvest pools of cheap oligonucleotides on demand. Importantly, our combination of technologies has allowed formation of genes and large DNA constructs by amplifying oligonucleotides off of the synthesized arrays and assembling them in the on-chip chambers.

MEMS and NEMS Room: Santo Domingo - Session MN-FrM

Characterization for MEMS and NEMS

Moderator: A.V. Sumant, Argonne National Laboratory

8:20am MN-FrM1 Wafer-scale Processing of Diamond Thin Films for MEMS and NEMS, J.A. Carlisle, Advanced Diamond Technologies INVITED

The key to enabling wafer-scale processing of thin film diamond are the specifications that the technology can meet on commercially relevant wafer sizes. Due to advances in reaction design and deposition chemistry, a variety of diamond materials can now be deposited onto wafers up to 300 mm in size, with excellent thickness and property uniformity, enabling high-yield production of microdevices. In this presentation advances made over the past several years to enable thin, smooth diamond films for MEMS will be reviewed with emphasis on materials and microfabrication strategies needed to integrate diamond with other materials for MEMS devices. UNCD® (ultrananocrystalline diamond) spans a family of thin smooth diamond materials. UNCD wafers serve to demonstrate the availability of the technology and to allow designers to integrate diamond into new process flows. NaDiaProbes®, which are all-diamond AFM probes used for metrology and nano-manufacturing, are the first example of a commercially available diamond MEMS device that leverages wafer-scale processing of UNCD wafers using established microfabrication techniques. How ADT looks at wafer-scale production of UNCD-based MEMS devices going forward as well as our technology roadmap for UNCD-enabled products under development will be presented. Particular emphasis will be placed on the development of UNCD coatings and MEMS devices for RF devices, nanomanufacturing, and chem/bio-sensing related applications.

9:00am MN-FrM3 CMOS Integrated Ultrananocrystalline Diamond Capacitive RF-MEMS Switches, S. Balachandran, A.V. Sumant, O.H. Auciello, Argonne National Laboratory, S. O'Brien, C.L. Goldsmith, Memtronics Corporation, J.A. Carlisle, Advanced Diamond Technologies, C. Gudeman, S. Sampath, Innovative Micro Technology

RF-MEMS DC contact and capacitive switches are being developed, involving a broad range of designs in series and shunt configurations. The work done until now has facilitated significant maturing of the technology to overcome technical challenges such as reliability, packaging, and high power operation. In particular, the reliability of RF-MEMS capacitive switches has been limited mainly by the electrical charging of the oxide or nitride dielectric layers used until now, which exhibit discharging times in the hundred of seconds range, resulting in failure of the switches. Therefore, it is critical to develop dielectric layers with fast charging/discharging performance. In this respect, the novel ultrananocrystalline diamond (UNCD) films developed and patented at Argonne National Laboratory exhibit a unique fast charging (50-100 µsec)/discharging (≤ 100 µsec) behavior, which provides the reliability required by RF-MEMS switches. The charges are transported through a large network of grain boundaries, which occupy a large percentage of the total area of the films, characterized by a nanostructure formed by 2-5 nm grains with ~ 0.5 nm wide grain boundaries.

This paper focuses on a description of materials, materials integration strategies, device architecture and performance of prototype monolithically integrated RF-MEMS mm-wave shunt capacitive switches/CMOS devices in coplanar waveguides, using UNCD as the dielectric layer. The RF-UNCD MEMS switches are based on a MEMtronics, Inc. switch design, fabricated on sapphire wafers with high-voltage CMOS devices, provided by Peregrine Semiconductor, using standard lithography and surface micromachining techniques. Small signal measurements were performed in the frequency range of 1-20 GHz. Measurements of the UNCD dielectric layer charging and discharging were performed using both MEMS and metal-insulator-metal (MIM) capacitor device configurations, using standard I-V and C-V techniques. The charging and discharging time constants for RF-MEMS switches with UNCD dielectric were 5-6 orders of magnitude faster ($\leq 100 \ \mu sec$) than the discharging times (100s of seconds) exhibited by conventional oxide or nitride dielectric materials. Although static power consumption is an issue with UNCD-based capacitors, they can be used in applications, which demand little to no degradation in performance and allow microwatts of power consumption.

This work was mainly supported by DARPA, under contract MIPR 06-W238. Use of the Center for Nanoscale Materials was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

9:20am MN-FrM4 Mechanical Stiffness and Dissipation in Ultrananocrystalline Diamond Resonators, V.P. Adiga, University of Pennsylvania, S. Suresh, A. Datta, Innovative Micro Technology, J.A. Carlisle, Advanced Diamond Technologies, A.V. Sumant, Argonne National Laboratory, R.W. Carpick, University of Pennsylvania

Nanocrystalline materials exhibit unique mechanical properties depending on the nature of the bond, co-ordination number of atoms at grain boundaries. Tetragonal sp³-bonded diamond has the highest known atomic density. The nature of the bond and its high density enable diamond to have superior physical properties such as the highest Young's modulus, melting temperature and acoustic velocity of all materials. Recently, conformal thin diamond films have been grown at CMOS-compatible temperatures in the form known as ultrananocrystalline diamond (UNCD). We have measured the Young's modulus (E), Poisson's ratio and the quality factors (Q) for microfabricated overhanging ledges and fixed-free beams composed of UNCD films grown at lower temperatures¹. The overhanging ledges exhibited periodic undulations due to residual stress. This was used to determine a biaxial modulus of 838 ± 2 GPa. Resonant excitation and ring down measurements of the cantilevers were conducted under ultra high vacuum (UHV) conditions on a customized atomic force microscope to determine E and Q. At room temperature we found $E = 790 \pm 30$ GPa, which is ~20 % lower than the theoretically predicted value of polycrystalline diamond, an effect attributable to the high density of grain boundaries in UNCD. From these measurements, Poisson's ratio for UNCD is estimated for the first time to be 0.057±0.038. We also measured the temperature dependence of E and Q in these cantilever beams from 60 K to 450 K. Above ~ 150 K, temperature dependence of modulus is slightly higher than that of single crystal diamond averaged over all directions despite the presence of large fraction of disordered carbon at grain boundaries and below 150 K changes in modulus are extremely small. This is the first such measurement for UNCD and strongly suggests that the nanostructure plays a significant role in modifying the thermo-mechanical response of the material. We have measured a very low temperature coefficient of frequency at room temperature which has important technological applications including resonant mass sensors and filters. The room temperature Q varied from 5000 to 16000 and showed a moderate increase as the cantilevers were cooled below room temperature and it saturates resembling the plateaus observed in many disordered systems. The results suggest that defects in the grain boundaries significantly contribute to the observed dissipation.

"Mechanical stiffness and dissipation in ultrananocrystalline diamond resonators" Adiga *et al*, V 79, pp 245403.1-8, *Physical Review B*, 2009

9:40am MN-FrM5 Theoretical and Experimental Investigation of Optically Driven Nanoelectromechanical Oscillators, *R.B. Ilic*, Cornell University, *S.L. Krylov*, Tel Aviv University, Israel, *H.G. Craighead*, Cornell University

Actuation of biologically functional micro and nanomechanical structures using optical excitation is an emerging arena of research that couples the felds of optics, fluidics, electronics and mechanics with potential for generating novel chemical and biological sensors. In our work, we fabricated nanomechanical structures from 200nm and 250nm thick silicon nitride and single crystal silicon layers with varying lengths and widths ranging from 4µm to 12µm and 200nm to 1µm, respectively. Using a modulated laser beam, focused onto the device layer in close proximity to the clamped end of a cantilever beam, we concentrate and guide the impinging thermal energy along the device layer. Cantilever beams coupled to chains of thermally isolated links were used to experimentally investigate energy transport mechanisms in nanostructures. The nature of the excitation was studied through steady-periodic axisymmetric thermal analysis by considering a multilayered structure heated using a modulated laser source. Results were verified by finite element analysis, which was additionally implemented for the solution of steady-periodic and transient thermal, as well as steady thermoelastic problems. These theoretical investigations, coupled with our experimental results, reveal that the complex dynamics underpinning optical excitation mechanisms consist of two disparate spatial regimes. When the excitation source is focused in close proximity to the structure the response is primarily thermal. We show that as the source is placed farther from the clamped end of the structure, the thermal response progressively fades out indicating the possibility of mechanical wave propagation. Understanding the excitation mechanisms may be useful for applications including compact integration of nanophotonic elements with functionalized nanomechanical sensors for ultra-sensitive biochemical analysis.

10:00am MN-FrM6 Nanotribological Studies of Adaptive Optics Sliding Components in Microprojectors, B. Bhushan, H. Lee, The Ohio State University, S. Chaparala, V. Bhatia, Corning Incorporated

As portable devices are increasingly developed and used, the size of their displays are getting smaller, therefore becoming harder to view. Integrated

micro-projectors are an alternative way to see a big image on any surface chosen by users. Microprojectors require red, blue, and green lasers to perform. However, unfortunately the green laser is not commercially available. In order to generate the green laser, frequency doubling technique is used, doubling a semiconductor laser of a wavelength 1060nm to 530nm green laser. The best alignment of the lenses in frequency doubling technique effects on energy efficiency and performance of the devices. To align the lenses, there are reciprocating and stick-slip motions between the components in the devices. Therefore, nanotribological studies of adaptive optics sliding components in microprojectors are needed. In this study, a methodology to measure lubricant thickness and distribution is developed. Lubricant bonding techniques are identified to bond the lubricant to the surfaces with thermal, UV or plasma treatments. Friction, adhesion and wear mechanisms of lubricant on the sliding components are studied in various environments, such as different humidity and temperature.

10:20am MN-FrM7 Nonlinear Fracture Mechanics Model for Mode I & Mode II Stiction Failure, A. Mousavi, Z.C. Leseman, University of New Mexico

The highly nonlinear nature of the crack propagation is studied using a completely nonlinear beam theory model. A model is described that is highly accurate for various deflections of microcantilevers and agrees with the experimental data with nanometer accuracy. The micro cantilever beams used are fabricated by SUMMiT V technology and the dimensions of the beams are 500 x 20 x 2 microns. A nonlinear method is also proposed to calculate the strain energy release rate of stiction failed micro cantilever beams. It is shown that at higher deflections the previous methods fail to accurately determine the stored strain energy and the strain energy release rate. The proposed method is used to accurately determine the beam's profile and strain energy release rate at higher deflections, and it is in good agreement with the other methods at small deflections.

The method developed here is completely applicable to both Mode I and mixed mode I & II crack development problems. It is shown that when it comes to micro cantilevers the so called Mode I crack propagation turns out to be a mixture of Mode I and Mode II. Longitudinal stresses developed inside the cantilevers increases rapidly as the beam gets nonlinear and starts to play a more important role in the crack length and strain energy release rate. The strain energy release rate can be formulated both using the elastic energy stored in the beam and also using the definition of stress intensity. In this paper, the two common methods for calculation strain energy release rate are discussed and stress intensity factors determined. The obtained stress intensity factors once again verify the fact that there is some Mode II component present during the crack propagation. These results also show that the strain energy release rate is not a constant value and increases as the crack develops. A highly linear relationship is observed between stress intensity components which are in good agreement with the analytical models of the phase angle. It is observed that the phase angle is almost a constant value for all crack heights.

10:40am MN-FrM8 Novel NEMS Gas Detectors for Micro Gas Chromatograph, C.-H. Chen, C.-J. Hsieh, H.-K. Lin, T.-S. Lee, P.-H. Chen, C.-H. Chou, National Taiwan University, Republic of China, C.-J. Lu, National Taiwan Normal University, Republic of China, W.-C. Tian, National Taiwan University, Republic of China

This research reports the developments and characterizations of two types of NEMS gas detectors, based on nano-scaled titanium oxide (TiO2) and monolayer protected gold nanoclusters (MPCs) as a sensing film of chemiresistors. For sensing films based on chemically synthesized MPCs, the diversified selectivity (>10 compound detection based on various MPC shell structures), the fast response (<10 seconds) and the low power consumption (25 µW) are its main advantages. However, the irreversible adsorption due to the strong affinity between MPCs and vapor molecules could be problematic. On the other hand, nano-structured TiO₂ film, which is operated at elevated temperature, is inherently immune from irreversible condensation. The TiO_2 sensing film is fabricated through E-beam lithography in the present study. Various shapes of nano-structures are precisely aligned and placed in between microelectrodes to mimic nanocrystalline structures for the enhancement of the detector sensitivity and robustness. In addition, these NEMS gas detectors are conditioned with integrated heaters at a controlled temperature to minimize any interfering gas adsorption in between analysis. Various performance evaluations including the characterizations of two types of detectors at different temperatures, test chemicals, detection limits, and the compatibility with a gas chromatography system, are investigated and to be presented.

11:00am MN-FrM9 On the Impact of Relative Humidity and Environment Gases on Dielectric Charging Process in Capacitive RF MEMS Switches Based on Kelvin Probe Force Microscopy, U. Zaghloul, B. Bhushan, The Ohio State University, P. Pons, LAAS-CNRS, France, G.J. Papaioannou, Universite de Toulouse, France, F. Coocetti, R. Plana, LAAS-CNRS, France

Dielectric charging is among the major reliability issues that have prevented the commercialization of RF-MEMS Capacitive switches in spite of the extensive study performed on the topic. Moreover, a little work has been performed to study the effect of the relative humidity (RH) and environment gases on the dielectric charging process.

In this work we present the effect of RH and the environment gases on the charging/discharging processes in PECVD silicon nitride films based on Kelvin Probe Force Microscopy (KPFM) methodology. The measurements have been performed in ambient air and under N2 flow, both under different RH levels (from 6% to 40% RH). In addition, the influence of the dielectric film thickness, SiN deposition conditions and the substrate nature on the charging process have been investigated under different environment conditions. This has been done through depositing SiN films with different thicknesses ranges from 100nm to 400nm over bare silicon substrates and over evaporated Au layers and using both Low Frequency(LF) and High Frequency(HF) PECVD deposition modes.

For both measurements performed in ambient air and under N2 flow, the surface potential decay with time follows the stretched exponential law. The decay time constant decreases strongly as the RH increases (1.230E+03 sec and 650 sec for 6%RH and 40%RH respectively, for the HF 200nm thick SiN film deposited over evaporated Au). The measured decay time constant is found to be shorter in case of N2 than in ambient air measurements. The surface potential distribution is represented by the Full Width at Half Maximum (FWHM) for charges which have been injected in a single point with the AFM tip. The FWHM becomes smaller as RH decreases. Charge injection duration is controlled to range from 10 ms to 100 sec. The FWHM is found to be always larger in air comparing to FWHM measured in N2 flow for different charge injection durations. Moreover, FWHM increases almost linearly with increasing the charge injection time for different RH measurements. Charge lateral diffusion has been observed at larger RH only and is attributed to the more hydrophobic SiN material at smaller RH levels which prevents water condensation at the surface and thereby inhibits lateral charge migration due to the electrical conductivity of a possible water film. The FWHM is found to be smaller in thinner SiN films than in thicker ones and the relaxation time is found to be larger in the thinner SiN films, independently of the substrate nature. Finally, the decay time constant is found to be smaller in case of dielectric films deposited over Au layers comparing to films deposited over bare silicon substrates.

11:20am MN-FrM10 Feature Size Etch Rate Dependence in Bosch Process Deep Silicon Etching Due to Local Thermal Loading, *R. Kurkul, R. Gulotty, B. VanderElzen*, University of Michigan, Ann Arbor

Aspect ratio dependent etching (ARDE) is a common issue in reactive ion etching. This phenomenon results in narrow, deeply etched features exhibiting slower etch rate and a more reentrant profile than larger, more open features. The common mechanism indicated for this effect is ion flux. The narrow features restrict the ability of ions that are not perfectly perpendicular to make it to the bottom of the feature and drive the etch. Recent data obtained at the University of Michigan Lurie Nanofabrication Facility suggests that, in some circumstances, there is a feature size effect that is independent of aspect ratio. The mechanism proposed and studied herein is local thermal loading due to exothermic etch reactions.

The evolution of faster etch rates in silicon is a key enabling factor in MEMS production. However, this fast etching results in thermal management concerns. In an effort to understand these temperature effects better, as well as to determine the evolution of aspect ratio dependence, we performed a variety of rate tests at various stages of etching on different size features. A surprising result was that, after an initial substrate warm up time where etch rates increased slightly, etching rates were flat until aspect ratios approached 10:1 after which they began to drop off slightly. Even more notable is a very significant rate variance with feature size even at ARDE should not be significant. This strongly suggests a mechanism of feature size dependence separate from aspect ratio.

The mechanism proposed for this etch rate variance is local thermal loading. This agrees with data collected. Temperature is a significant factor in determining etch rate. The fluorine silicon reaction is exothermic. This has been shown to elevate the temperature of the substrate over the first few minutes of the process. The thermal load will also result in heating and a higher temperature at the etch interface within the features. Larger features will have a higher local thermal load and thus get hotter. This heating accelerates the etch and likely inhibits the ensuing etch resistant fluorocarbon deposition step that is characteristic of the Bosch Process. We will demonstrate the evolution of this thermal loading and its effects on etch rate within an etch step and through the first few etch cycles of a Bosch Process. We will then evaluate the effectiveness of possible methods of mitigating this effect.

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