Manufacturing Science and Technology
Room 103A - Session MS-MoM

Manufacturing for Next-Generation Energy Solutions
Moderator: Erik B. Svedberg, The National Academies

9:00am **MS-MoM3 Manufacturing Challenges in Batteries: Lessons from Current Technology for Future Energy Storage Developments***, **Yet-Ming Chiang**,* MIT **INVITED**

The evolution of today's highly successful lithium ion battery manufacturing technology over 25 years provides many useful lessons, pro and con, for future storage technologies. Along with the development of higher performance/lower cost cathodes, anodes, electrolytes or other cell components, efficient cell designs and low cost/highly scalable manufacturing techniques are needed for any new technology to suceed. This talk will discuss prevailing Li-ion cell design and manufacturing methods which, despite evident success, have inherent inefficiencies which in the author's view have prevented the full exploitation of Li-ion chemistry. A "clean sheet" redesign developed at MIT and 24M Technologies will be discussed, based on a new semi-solid electrode form that enables manufacturing of high performance Li-ion cells by radically simpler and lower-cost methods. Concepts from this case study that may be transferable to new storage technologies include the need to minimize of non-energy-storing materials content in any device design; the benefits of reducing the tortuosity of ion transport pathways; how multiple functions can be served by a single component, and the importance of developing manufacturing processes that are cost-effective at small production scale, yet can be readily scaled to GWh volumes.

Support for this work by the U.S. Department of Energy through the ARPA-E program, the Vehicle Technologies Office of EERE, and the Advanced Battery Materials Research (BMR) program is gratefully acknowledged.

9:40am **MS-MoM5 Efficient Manufacturing of Nano-structured Lithium Sulfide for Next Generation Batteries***, X. Li, Y. Yang, **Colin Wolden**,* Colorado School of Mines

Lithium ion batteries (LIBs) currently dominate the market and are expected to for another decade due to incremental improvements. However, with performance approaching intrinsic material limits, LIBs cannot meet the increasing demands of electric vehicles and stationary storage. In the next decade both solid state and lithium–sulfur (Li-S) batteries will begin displacing LIBs. Central to both technologies is Li_2S, which serves as the active cathode material in Li-S batteries and is the key precursor and cost driver for leading solid-state electrolytes. In both applications Li_2S has demonstrated excellent performance when used in nanoparticle (NP) form. However, Li_2S is very costly and commercially available only as micropowders with impurities being a major concern, reflecting the energy-intensive carbothermal reduction processes currently used for synthesis. In this paper we describe a green chemistry-inspired approach for synthesizing alkali sulfide (M_2S, M = Li and Na) NPs through the reaction of hydrogen sulfide (H_2S) gas with alkali metal-organic (M-R) complexes dissolved in solution. This thermodynamically favorable reaction occurs spontaneously and proceeds rapidly to completion with near 100% atom efficiency at ambient temperature, forming phase-pure, anhydrous M_2S nanopowders that are readily separated from solution. H_2S, a major industrial waste, is completely abated and the valuable hydrogen stored therein may be fully recovered as H_2. The overall stoichiometry of the two step process is:

$2M (s) + H_2S (g) \Rightarrow M_2S (s) + H_2 (g)$

As such, this innovative synthetic approach is expected to be scalable, energy-efficient, and cost-effective. In this presentation we describe the process chemistry, focusing on the role of the complexing agent (R) and solvent to control the yield, size, and morphology of the resulting M2S NPs.

10:00am **MS-MoM6 Anode Protection for Advanced Energy Storage Systems *via* Atomic Layer Deposition***, **Chuan-Fu Lin**, M. Noked,* Institute for System Research, University of Maryland*; A.C. Kozen,* Naval Research Laboratory*; A.J. Pearse,* University of Maryland, College Park*; K. Gregorczyk,* Institute for System Research, University of Maryland*; S.B. Lee,* University of Maryland College Park*; G. Rubloff,* Institute for System Research, University of Maryland

To meet the demand for higher capacity longer life batteries in a "next-generation batteries" technology, anodes with substantially higher energy density than current graphite are needed. One class - metal anodes (particularly Li) - offers far higher energy density, but to date their utilization has been impeded by their high surface reactivity (especially to the organic electrolyte) and tendency to form dendritic Li (a shorting and safety hazard). Another category - conversion materials, also high energy density – involves complex material reactions as part of lithiation/delithiation, degrading the material during cycling.

We have used atomic layer deposition (ALD) to create highly controlled, thin protective layers on Li metal anodes and on RuO_2 conversion anodes, testing their efficacy by cycling in batteries. ALD protection layers, including Al_2O_3 and the solid electrolyte LiPON (lithium phosphous oxynitride), were directly deposited on the anodes in controlled inert ambient conditions. For Li metal anode protection, both Al_2O_3 and LiPON markedly suppress corrosion and degradation, as evaluated in Li-S cells by charge/discharge cycling and a variety of other characterization methods, with considerably higher capacity for the LiPON. The ALD films also stabilize the Li metal surface, preventing Li dendrite formation upon repeated cycling above the threshold current density for dendrite formation. For the conversion anode, the ALD coatings suppress electrolyte decomposition, thereby enhancing capacity retention during cycling, and lowering overpotentials required for delithiation, effects attributed to the layers mechanically constraining the RuO_2 the lithiation products Li_2O and Ru, preserving structural integrity.

These results demonstrate promise for achieving high energy density anodes with significantly enhanced chemical stability, electrochemical cyclability, and dendrite protection as needed for a viable beyond Li ion battery technology.

10:40am **MS-MoM8 Controlling Nanomaterial Assembly to Improve Material Performance in Energy Storage Electrodes using Electrophoretic Deposition***, **Landon Oakes**, R.E. Carter, A.P. Cohn, C.L. Pint,* Vanderbilt University

Electrophoretic deposition (EPD) provides a promising tool for large-scale manufacture of nanomaterial systems using conventional liquid processing techniques. One major roadblock to commercially viable applications of nanomaterials, such as in energy storage devices, is the ability to cost-effectively manufacture electrode-scale films while still maintaining precise control over the nanoscale and microscale morphology. We emphasize the ability of EPD to control nanoscale assembly for high throughput battery manufacturing through the design of a benchtop roll-to-roll platform. Using this approach, we fabricate electrodes for a range of battery technologies, such as lithium-ion batteries, lithium-sulfur batteries, and lithium-oxygen batteries. This makes possible the development of binder-free, electrode assemblies with uniformity and control that enables improved performance across all of these battery platforms. Specifically, for lithium-sulfur batteries, we fabricate binder-free electrodes that achieve over 75% sulfur loading with a capacity greater than 1,200 mAh/g that retains more than 80% of the initial capacity after 100 cycles. For lithium-oxygen batteries, we demonstrate electrodes with improved overpotential of 50 mV during oxygen reduction and 130 mV during oxygen evolution in addition to a nearly 2X improvement in durability compared with conventional assembly methods. Overall, as battery manufacturing remains a critical barrier separating state-of-the-art research efforts from practical commercial energy storage innovations, we emphasize EPD as a versatile process able to provide the scalability, high throughput, and nanoscale control that are necessary to advance battery systems manufacturing.

11:00am **MS-MoM9 3D Architectures for Thin Film Batteries – from Science to Manufacturing***, **Gary Rubloff**, C. Liu, E. Hitz, S.B. Lee,* University of Maryland College Park*; K. Gregorczyk,* University of Maryland, College Park

Recent research has elucidated several guidelines for energy storage approaches to achieve higher power at high energy density along with good capacity retention during cycling. These guidelines address electrode surface areas and active storage layer thicknesses, integration of current collecting features with active layers, and overall structure of 3D electrode features at the micro or nano scale. Here we consider solid state thin film battery architectures fabricated by thin film processes, where the solid electrolyte enables complex interdigitated electrode arrangements. We report projected performance in power and energy density for several configurations as a function of design parameters, including scaling of nanopore batteries already achieved experimentally. Finally, we consider manufacturability aspects in terms of (1) process sequence complexity and (2) comparison to that of other existing technologies based on thin film processing.

11:20am **MS-MoM10 Advanced Manufacturing R&D for Clean Energy in the US Department of Energy***, Mark Johnson,* Advanced Manufacturing Office, U.S. Department of Energy **INVITED**

Manufacturing is a critical component of the U.S. economy, responsible for 12.5% [1] of GDP, direct employment for over 12 million people [1], and close to 75% [2] of U.S. exports of goods. The U.S. manufacturing sector, while it produces 17% [3] of the world's manufacturing output, also represents a quarter of the country's energy consumption [4]. On the R&D side, it is responsible for 70% of all business R&D performed (in 2010 and 2011) and nearly 60% of patent applications [5]. As such, DOE has a vested interest in broadly applicable energy efficiency technologies for use in energy intensive manufacturing, as well as platform materials and processes for use in the manufacturing of clean energy technologies.

This talk will review the Advanced Manufacturing Offices work towards making the U.S. manufacturing sector more energy productive—and the U.S. clean energy manufacturing sector more competitive—through targeted R&D and partnerships with industry, academia, technology incubators and other stakeholders.

Manufacturing Science and Technology
Room 103A - Session MS-MoA

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Moderator: Gary Rubloff, Institute for System Research, University of Maryland

1:40pm **MS-MoA1 Metrology of Laser-based Powder Bed Fusion Additive Manufacturing Systems*, John Slotwinski,*** The Johns Hopkins University Applied Physics Laboratory **INVITED**

Metrology of Laser-based Powder Bed Fusion Additive Manufacturing Systems

John A. Slotwinski, Ph.D.

The Johns Hopkins University Applied Physics Laboratory

Additive Manufacturing (AM, aka 3DPrinting) is a potentially revolutionary manufacturing technology that is changing how both polymer and metal parts can be designed and fabricated. Geometrical complexity, gradient materials, and one-piece assemblies, all of which are difficult or impossible to fabricate with traditional removal processes, are all realizable with additive manufacturing. However, there are several technical challenges that are preventing more widespread adoption of additive manufacturing systems, especially for high-value, mission-critical parts. Chief among these challenges are a lack of full understanding of AM processes, especially for metal AM processes, and the factors influencing the mechanical properties of AM parts. In this talk I will give a brief overview of additive manufacturing processes, describe the technical challenges that are hindering broad adoption of AM parts for critical applications, and describe some recent efforts to measure and better understand both AM processes and AM material properties.

2:20pm **MS-MoA3 Investigation of Superconductive Heavily Doped Boron Diamond for Device Fabrication*, Delroy Green,*** *G.L. Harris,* Howard University*; R.D. Vispute,* Bluewave Semiconductor Inc.

Diamond has a wide bandgap of 5.47 eV at room temperature and is the hardest known naturally occurring material with a Knoop hardness of 10,400 kg/mm^2 or 10 on the Mohs scale. Due to the structure of the covalent bonding of its carbon atoms, diamond is extremely strong having each carbon bonded to four neighboring carbon atoms. Although diamond is hard, its toughness, when compared to most engineering materials, is poor. However, because of its hardness, it is an efficient cutting and drilling tool. With the exception of naturally occurring blue diamonds, which are semiconductors, diamond is a good electrical insulator. However, unlike most insulators, diamond has the highest thermal conductivity of 22 W/cm-K among naturally occurring materials. Although diamond is a good electrical insulator, it also shows semiconducting properties when doped with impurities. When diamond is heavily doped with boron the resulting material possess excess electron holes and as such it is classified as a p-type material. If excess boron doping is achieved, then the resulting material is found to behave like a superconductor at very low temperatures. In this superconducting state, the doped diamond conducts electricity.

A series of boron-doped diamond films were grown by hot filament chemical vapor deposition (HFCVD) and tested to determine the optimum technique for doping diamond with boron for superconductivity. The first technique involved the insertion of boron powder (B_2O_3) around the sample holder to dope seeded poly and nano diamond during growth. The second technique involves doping with diborane gas (B_2O_6).

Various processing parameters were optimized for diamond quality, structure, morphology, and doping. A combined analysis of scanning electron microscope, Raman mapping and Hall measurements at various temperatures were conducted to ascertain the superconductive nature of the material. Preliminary results of the boron solid source doping on diamond show a superconductive transition temperature of 2.3 ^{o}Kelvin at a doping concentration of

2.3 x 10^{20} cm^{-3}.

This research is conducted under research grants CIQM NSF DMR# 1231319 and PREM NSF DMR# 0611595

2:40pm **MS-MoA4 Two-Dimensional Layered Materials For Composites Applications*, Jorge Catalan,*** *A. Delgado, A.B. Kaul,* University of Texas at El Paso

Composite materials provide us with an alternative route to combine the characteristic properties from two different materials into one. At the same time, this characteristic of composite materials opens up a new window for different applications such as optoelectronic sensors, strain sensors, capacitive sensor and opto-electro-mechanical sensors. Initially, the isolation of single layered graphene by mechanical exfoliation and nowadays with different methods such ion intercalation, solvent based exfoliation and chemical vapor deposition (CVD) have allowed the utilization of two-dimensional (2D) materials as reinforcement particles into different polymer matrixes for composite materials. This is because 2D materials offer interesting semiconducting properties that might be able to be captured in a polymer-based matrix that provides a ductile medium make them suitable for printable flexible electronic devices and sensors. In this work we have explored graphene, MoS2, and WS2 as possible reinforcement material in different polymers matrixes. The first type of composite consisted of a poly-methyl-methacrylate (PMMA) matrix with different type of fillers (graphene, MoS2 and WS2). The second type of composite materials that we studied consists of a poly-isoprene matrix (natural rubber band) and graphene, MoS2, and WS2 as reinforcement material. We have conducted strain testing on the structures we have fabricated to make strain-dependent electrical and optical properties. The PMMA/filler material composite was optically and electrically characterized under different strains with the help of different fixtures with different radius of curvature. On the other hand, the poly-isoprene composites were characterized with the help of a self-made type of clamp that allows us to strain the rubber band like composite to different degrees and measure the electrical characteristics of the compound. The opto-electro-mechanical characterization was developed with the scope of utilizing these composite materials as strain or flexible sensors for health monitoring or non-destructive evaluation.

4:00pm **MS-MoA8 AIM Photonics – Manufacturing Challenges for Photonic Integrated Circuits*, Michael Liehr,*** SUNY Polytechnic Institute **INVITED**

Abstract: The recently established American Institute for Manufacturing Photonics (AIM Photonics) is a manufacturing consortium headquartered in NY, with funding from the US Department of Defense, New York State, California and Massachusetts, and industrial partners to advance the state of the art in the design, manufacture, testing, assembly, and packaging of integrated photonic devices . Dr. Michael Liehr, CEO of AIM Photonics, will describe the technical goals, operational framework, near-term milestones, and opportunities for the broader photonics community.

The scope of AIM Photonics will span several industry segments, with the most prominent and near term commercial segment of Datacom applications, to analog/RF, array and sensor applications that are expected to mature at a later time. Photonic Integrated Circuits (PIC) technology enables optical systems to be miniaturized and fabricated on semiconductor chips. Just as electronic integrated circuits revolutionized electronics by miniaturizing transistor circuitry, PICs integrate lasers and other optical devices to route and process information with reduced size and power. PICs can also scale in complexity to do things that would not be possible using conventional optical design approaches. By putting these components on a single platform, PICs have the potential to advance technology in ways never before possible.

Targeted markets include:

Ultra-high-speed transmission of signals for the internet and telecommunications

New high-performance information-processing systems and computing

Compact biomedical sensor applications enabling dramatic medical advances in diagnostics and treatment

Multi-sensor applications including urban navigation, free space optical communications, and quantum information sciences

Other military applications, including electronic warfare, analog RF sensing, communications, and chemical/biological detection

4:40pm **MS-MoA10 Development of III-Nitrides for Energy Harvesting Applications***, B. Kucukgok, N. Lu,* Purdue University*; **IanT. Ferguson,*** Missouri University of Science and Technology

III-Nitride wide-bandgap semiconductors have recently enabled state-of-the-art technologies for energy harvesting applications, such as photovoltaics and thermoelectrics. III-Nitride materials and devices have provided tremendous advantages due to their distinguished features, including tunable bandgap, superior electrical properties, high-temperature stability, enhanced chemical stability, and mechanical strength. Furthermore, InGaN with indium compositions up to 30% (2.5 eV band gap) have been developed for photovoltaic applications by controlling defects and phase separation. Additionally, InGaN solar cell design consists of 2.9 eV InGaN p-n junction sandwiched between p- and n-GaN layers results in internal quantum efficiencies as high as 50%; while devices utilizing a novel n-GaN strained window-layer enhanced the open circuit voltage. These results establish the potential of III-Nitrides and related materials in ultra-high efficiency photovoltaics. Moreover, thermoelectrics, conversion of waste thermal energy into electrical energy, have seen pioneering developments over the past 20 years. A figure of merit ZT, used to measure the efficiency of the thermoelectric materials. Various approaches have been taken to increase the efficiency of thermoelectric materials, such as electron quantum confinement and phonon scattering to increase the power factor and decrease the lattice thermal conductivity, respectively. The objectives of this study are to highlight the use of III-Nitrides in high efficient photovoltaic and thermoelectric energy harvesting applications. Some recent measurements of the thermoelectric properties–the Seebeck coefficient, the electrical conductivity and the power factor–of GaN and InGaN thin films will also be reported.

Manufacturing Science and Technology
Room 103A - Session MS+AS-TuM

Characterization and Processing for IC Manufacturing

Moderator: Alain C. Diebold, SUNY College of Nanoscale Science and Engineering

8:00am **MS+AS-TuM1 Thermal Decomposition Properties of Bis(cyclopentadienyl)magnesium for Various Gas Supply System Materials***, **Hidekazu Ishii**,* Tohoku University, Japan*; S. Yamashita, M. Nagase, A. Hidaka, K. Ikeda,* Fujikin Incorporated, Japan*; Y. Shiba, Y. Shirai, S. Sugawa,* Tohoku University, Japan

High purity Bis(cyclopentadienyl)magnesium(Cp_2Mg) is used as chemical vapor deposition material of semiconductor devises and dopant for obtaining p-type conduction in GaN based material devices. However, precise control of supply concentration of Cp_2Mg is very difficult because its vapor pressure is very low. Generally, Cp_2Mg is supplied from a precursor container to the film formation chamber by bubbling with the carrier gas. In this method, the tubing for the gas supply must be heated to avoid deposition of Cp_2Mg on the inner surface of tube, which leads to a concern of decomposition of Cp_2Mg. Thus, understanding of thermal decomposition properties of Cp_2Mg for various materials for gas supply tube is important. In this report, we report evaluation results of thermal decomposition properties of Cp_2Mg for various materials such as SUS316L stainless steel and Cr_2O_3, Al_2O_3-passivated stainless steel that are used for gas tubing, as well as Ni-Co alloy and Hastelloy C-22 that are used for a valve diaphragm.

8:40am **MS+AS-TuM3 High Volume Materials Characterization in the CMOS Industry***, **Paul van der Heide**,* GLOBALFOUNDRIES **INVITED**

In no time in the past has Materials Characterization been as pivotal to CMOS device R&D as it is today. This stems primarily from the fact that since the era of *Denard scaling* (shrinkage alone), new materials/ structures have had to be introduced in order for logic devices to continue to adhere to the dimension shrinkage implied by *Moore's law* (examples lie in the introduction of strain engineering (introduced in 90nm devices rolled out in 2003), HKMG structures (introduced in 45nm devices rolled out in 2007), and 3D structures (introduced in 22nm devices rolled out in 2011)). This timeline also begs the question: A*re we not at the precipice of the next innovation?* What is certain is that the CMOS industry will experience significant and in some cases unforeseen changes over the next 2 decades.

Materials characterization is not only needed to support R&D efforts, but is also required to provide insight into manufacturing issues, along with the qualification of a) new fabrication processes, b) new process equipment, and c) process equipment coming off preventative maintenance cycles. Paramount in these areas is analytical precision, repeatability, data quality and speed (turn around time). This stems from the other aspect of *Moore's law*; that being that the cost associated with the development/implementation of a new device node must remain financially attractive. Topics covered in this presentation include: the support requirements of a high volume CMOS manufacturing site, merits of academia versus industrial labs, financial justifications of onsite lab/s, along with some recent analytical examples/capabilities.

9:20am **MS+AS-TuM5 Dynamics in SIMS Characterization for Advanced Nano-Technology: Challenges and Solutions for Novel Materials and 3-D Devices***, **Marinus Hopstaken**,* IBM T.J. Watson Research Center **INVITED**

Over the last few decades, SIMS depth profiling techniques and instrumentation has tremendously evolved to keep up with developments in advanced CMOS technology. I will discuss the main technology drivers, their implications for SIMS characterization, and review some of the analytical challenges and solutions:

- Continued dimensional scaling (*i.e.* lower film thicknesses, ultra-shallow junctions USJ) demands for progressive improvement of depth resolution. This has been enabled by continuous instrumental developments to provide high-density, stable, and low-impact energy primary ions beams to enable sub-nm depth resolution (*i.e.* 'Atomic layer' SIMS). I will give various applications of high resolution SIMS analysis of thin-film stacks / USJ, routinely employing sub-500 eV ion beams

- Advanced IC development in a manufacturing context demands at-line SIMS metrology with high throughput and reproducibility, often requiring small area analysis on patterned wafers. Key enablers for advances in SIMS metrology are availability of high-density primary ion beams, high level of automation to allow for unattended operation, and instrumental stability / drift correction. I will discuss implications for high-throughput SIMS full wafer mapping and considerations for patterned device wafer

- Paradigm shift towards 3D device architectures (*i.e.* FinFET) poses one of the greatest challenges, and appears fundamentally incompatible with low-energy (*i.e.* 'broad-beam') SIMS. This can be partially circumvented by averaging over a large regular arrays of FinFET structures, in combination with backfill and planarization to delineate the Fin sidewall ('SIMS through Fin technique;), which we have successfully employed at realistic Fin dimensions and pitch, relevant for 14 nm node and beyond

- Integration of novel and dissimilar material stacks demands novel SIMS calibration methods and/or quantification protocols. Potential solutions to deal with the higher complexity are cross-calibration with absolute external techniques (ion scattering techniques, 3D-APT, advanced TEM-EDX / EELS, etc…) and multi-standard approaches for explicit correction of SIMS yield variations with matrix composition. I will give selected examples for quantification of in-situ doping in $SiGe_x$ for wide variation in Ge% and different doping species in various III-V compounds

11:20am **MS+AS-TuM11 Characterization of Electrical Properties of Si and GaN Devices using Scanning Microwave Impedance Microscopy (sMIM) and Nano-scale Capacitance-voltage Curves***, **Stuart Friedman**, F. Stanke, Y. Yang, O. Amster,* PrimeNano, Inc

The use of Atomic Force Microscopy (AFM) electrical measurement modes is a critical tool for the study of semiconductor devices and process development. A relatively new electrical mode, scanning microwave impedance microscopy (sMIM), measures a material's change in permittivity and conductivity at the scale of an AFM probe tip [1]. sMIM provides the real and imaginary impedance (Re(Z) and Im(Z)) of the probe sample interface. By measuring the reflected microwave signal as a sample of interest is imaged with an AFM we can in parallel capture the variations in permittivity and conductivity and, for doped semiconductors, variations in the depletion layer geometry. An existing technique for characterizing doped semicondutors, scanning capacitance microscopy, modulates the tip-sample bias and detects the tip-sample capacitance with a lock-in amplifier. A previous study compares sMIM to SCM and highlights the additional capabilities of sMIM [2].

In this talk we focus on the detailed mechanisms and capabilities of the nano-scale C-V curves that can be obtained using sMIM to measure the tip-sample capacitance as a tip-sample bias is swept. Analogous to traditional macro-scale capacitance-voltage experiments, the nano-scale C-V curves probe properties such as doping concentration through their influence on the voltage dependent geometry of the depletion layer. In particular, in this talk we will address the ability to extract semiconductor properties, such as doping concentration, from the C-V curves. This study includes analytical and finite element modeling of tip-bias dependent depletion layer geometry and impedance. These are compared to experimental results on reference samples for both doped Si and GaN doped staircases to validate the systematic response of the sMIM-C channel to the doping concentration.

[1] S. Friedman, O. Amster, Y. Yang, "Recent advances in scanning Microwave Impedance Microscopy (sMIM) for nano-scale measurements and industrial applications." Proceedings of the SPIE, Volume 9173, id. 917308 8 pp. (2014)

[2] B. Drevniok, St.J. Dixon-Warren, O. Amster, S.L. Friedman, and Y. Yang, "Extending Electrical Scanning Probe Microscopy Measurements of Semiconductor Devices Using Microwave Impedance Microscopy", Proceedings of the 41st International Symposium on Testing and Failure Analysis (2015), pp. 77.

11:40am **MS+AS-TuM12 Results of the 2016 Triennial Review of the National Nanotechnology Initiative***, **James Murday**,* University of Southern California*; B.R. Rogers,* Vanderbilt University*; E.B. Svedberg,* The National Academies **INVITED**

The National Nanotechnology Initiative is a multi-agency effort to advance nanoscale science, engineering, and technology and to capture the associated economic and societal benefits. The NNI comprises the collective activities and programs among the more than two dozen participating federal agencies with diverse missions and presently a total annual investment of approximately $1.5 billion. Every three years the National Academies selects a committee of experts to review the NNI in accordance with the provisions of the 21st Century Nanotechnology Research and Development Act. A report on the most recent review has just been released. This report has paid particular attention to examining and commenting on the physical and human infrastructure needs for successful realization in the United States of the benefits of nanotechnology development and also the mechanisms used by the NNI to advance focused areas of nanotechnology towards advanced development and commercialization. We will report the findings and recommendations of this review.

Manufacturing Science and Technology
Room 103A - Session MS-TuA

Working with National Labs and User Facilities

Moderator: Bridget Rogers, Vanderbilt University

2:20pm **MS-TuA1 Southeastern Nanotechnology Infrastructure Corridor (SENIC) – A Nano Fabrication and Characterization Resource as part of the National Nanotechnology Coordinated Infrastructure (NNCI)**, ***Paul Joseph**, D. Gottfried, G. Spinner, O. Brand,* Georgia Institute of Technology

The Southeastern Nanotechnology Infrastructure Corridor (SENIC) is a partnership between two state-of-the-art nanofabrication and characterization facilities located at the Institute for Electronics and Nanotechnology (IEN), an interdisciplinary research institute at the Georgia Institute of Technology, and the Joint School of Nanoscience and Nanoengineering (JSNN), an academic collaboration between North Carolina A&T State University (NCA&T) and the University of North Carolina at Greensboro (UNCG). SENIC is one of 16 members of the National Nanotechnology Coordinated Infrastructure (NNCI), supported by the National Science Foundation, and coordinated by the NNCI Coordinating Office at Georgia Tech. NNCI is an integrated networked partnership of academic nanotechnology user facilities across the US, serving the needs of nanoscale science, engineering, and technology. The NNCI is a research facilitator, providing state-of-the-art equipment, staff expertise, and training to nanotechnology researchers. The shared-user, fee-based laboratories are open to academic, industry, and government clientele, offering a unique and comprehensive nanotechnology laboratory and teaming environment.

At Georgia Tech, the IEN has dedicated expertise and facilities for a broad range of micro and nanofabrication and characterization projects, including a focus on applications to bioengineering and biomedicine. IEN supports cleanroom and characterization facilities used by more than 700 researchers annually, with 20% from external institutions. The external users (off-campus users) can access the desired tool set after training (on-site work) or can send their samples for processing or analysis by IEN staff (remote work). IEN offers unique capabilities in e-beam lithography, photolithography, soft lithography, thin film deposition, etch processing, metallization, packaging, micro scale printing, imaging, metrology, and microanalysis. IEN-supported research themes include nanostructures, nanoelectronics, bio-MEMS, biological/chemical sensors and systems, biomaterials, photonics, materials growth and synthesis.

During this presentation, an overview of NNCI and SENIC will be given. Subsequently, we will discuss shared user lab resources, external user services, and education programs available at IEN.

2:40pm **MS-TuA2 The Cornell NanoScale Science and Technology Facility (CNF)**, ***Michael Skvarla,*** Cornell NanoScale Science and Technology Facility

The Cornell NanoScale Science and Technology Facility (CNF) is a member of NNCI, a network of open-access facilities partially subsidized by the US National Science Foundation to provide researchers with rapid, affordable, shared access to advanced nanofabrication tools and associated staff expertise. Hundreds of researchers worldwide (from academia, industry, and government) utilize CNF to make structures and systems from the nanometer scale to the centimeter scale. CNF offers unique capabilities in electron-beam lithography, advanced stepper photolithography, dedicated facilities for soft lithography, and direct-write tools for rapid prototype development, along with the flexibility to accommodate diverse projects and to deposit, grow, and etch a wide variety of materials. CNF's technical staff are dedicated full-time to user support, providing one-on-one help with process development, tool training, and troubleshooting. They can offer expertise for a very wide range of fabrication projects, including electronics, nanophotonics, magnetics, MEMS, thermal and energy systems, electrochemical devices, fluidics, and the life sciences and bioengineering (more than 30% of CNF's users now focus on biology). All users are welcome; no experience in nanofabrication is necessary and a central part of CNF's mission is to assist users from "non-traditional" fields seeking assistance to implement nanofabrication techniques for the first time. CNF's user program is designed to provide the most rapid possible access (typically 2 weeks) with the lowest possible barriers to entry (users retain full control of their IP, with no entanglement by CNF or Cornell University). Many of CNF's external academic users come from institutions with their own local cleanroom facilities, but still utilize CNF for advanced capabilities, staff expertise, or tool availability.

This talk will explore the tools and the types of services and advice available to CNF users, and present examples of ongoing work with the hope of stimulating ideas and possibilities. We will also provide the latest details on the National Nanotechnology Coordinated Infrastructure (NNCI), a new NSF-sponsored network of shared facilities similar to CNF.

We invite you to explore the CNF and NNCI and discuss ways we can help bring your research visions to fruition. As a first step, CNF's User Program Managers will at no cost provide detailed processing advice and cost estimates for potential new projects. The CNF technical staff also meets every Wednesday afternoon for conference calls where we welcome questions about any topic related to nanofabrication. Visit cnf.cornell.edu to contact us and get started.

3:00pm **MS-TuA3 The CNST NanoFab at NIST: *Nanofabrication for US Commerce*, *Vincent Luciani,*** NIST Center for Nanoscale Science and Technology

The NIST Center for Nanoscale Science and Technology (CNST) supports the U.S. nanotechnology enterprise from discovery to production. As part of the CNST, the shared-use NanoFab provides its users rapid access to a comprehensive suite of tools and processes for nanoscale fabrication and measurement. The CNST NanoFab at NIST is part of the Department of Commerce and therefore puts a high priority on operating a business friendly, easily accessible facility. The same rates are applied to all users, whether from industry, academia or NIST. Applications are accepted at any time and are reviewed and processed every week. Also, NIST does not claim any inherent rights to inventions made in the course of a NanoFab project. Your intellectual property rights are not affected. The NanoFab features a large, dedicated facility, with tools operated within an ISO 5 (class 100), 750 m^2 (8,000 ft^2 cleanroom and in adjacent laboratories that have superior air quality along with temperature, humidity, and vibration control. Over 80 major process tools are available, including but not limited to e-beam lithography, 5x reduction stepper photolithography, nano-imprint lithography, laser writing for mask generation, scanning and transmission electron microscopy, 3 Focused Ion Beam (FIB) systems, metal deposition, plasma etching, chemical vapor deposition, atomic layer deposition, deep silicon etching, ion beam etching and a soft-lithography lab. The NanoFab staff consists of scientists, engineers and technicians that specialize in all areas of nanofabrication and provide training and ongoing technical assistance to users. Our goal is to be a catalyst to our users' success and to help nurture nanotechnology commerce in the Unites States. Project applications and instructions are easily available on the web at www.nist.gov/cnst/nanofab. Users inside NIST and from all around the country are provided on-line access to tool schedules and the tool reservation system. From physicists, engineers and biologists to medical researchers, users find common ground at the nanoscale in the CNST NanoFab.

3:20pm **MS-TuA4 In-Situ Characterization Tools for Materials Growth and Processing at NSLS-II**, ***Klaus Attenkofer**, E. Stavitski, K. Evans-Lutterodt, C. Nelson,* Brookhaven National Laboratory

Driven by the needs of sub-15nm integrated chip design, power electronics, and energy conversion devices, a wide range of coating and etching processes are in development which allow single layer growth or removal controlled by complex chemical processes on the substrate and/or in the gas phase resulting in self-limiting growth/etching approaches. The invention of new processes for conventional and spatial Atomic Layer Deposition (ALD) is one of the most prominent applications; even if it has a potentially high impact on the technology, used by everybody in future, it faces the challenge of an enormously large parameter space and costly and lengthy experiments developing the various precursor compounds. A theory inspired approach, combining combinatorial methods with computational modeling, may significantly reduce the risks and costs of the development process; however, to connect both approaches, an in-situ characterization tool will be required characterizing structure, and chemistry of the surface compounds, the gas and the film itself. X-ray spectroscopic and scattering/diffraction techniques may be the probes which provides chemical and structural sensitivity under complex reaction conditions.

NSLS-II had developed a set of beamlines which combine high-end state-of-the-art beamline design with optimized endstation design for materials growth. Specifically, the talk will provide an overview on the scattering and spectroscopy capabilities at In-Situ and Resonant scattering (ISR) beamline and the Inner Shell Spectroscopy beamline (ISS); two instruments build to study the growth of amorphous and crystalline films from the early seed formation to the bulk-like film. Next to an introduction into the beamline and its endstation equipment, we will also present experimental data which demonstrate the power of in-situ chracterization.

4:20pm **MS-TuA7 The Center for Nanophase Materials Sciences***, Michael Simpson,* Oak Ridge National Laboratory

The Center for Nanophase Materials Sciences (CNMS) at Oak Ridge National Laboratory (ORNL) is a multidisciplinary user facility that provides the research community with access to expertise and equipment to address the most challenging issues in nanoscience. Industrial, government and academic researchers from around the world may access capabilities in functional imaging, atom-precise synthesis, and nanofabrication. The CNMS is a leader in a range of advanced nanofabrication techniques including electron beam assisted deposition on the sub-10 nm level using both gas and liquid precursors as feedstock material, 3D fabrication and atomically precise material sculpting, as well as direct matter manipulation on the atomic level by electron beams to induce material functionality. Spatially resolved quantitative measurements of physical and chemical properties of materials are available to users through unique measurement capabilities of band excitation scanning probe microscopy, scanning transmission electron microscopy, helium ion microscopy, and atom probe tomography. Furthermore, theoretical and computational approaches are available to CNMS users, as frameworks for deep-data analytics methods for imaging, and computational prediction of functional and physical properties in nanostructures, benefiting from the broad ORNL computational capabilities. Located adjacent to the Spallation Neutron Source at ORNL, CNMS acts as a gateway for the nanoscience community to ORNL's world-class neutron science facilities, by providing diverse complementary capabilities such as selective deuteration, sample environments for multi-modal measurements, fabrication of templates for neutron reflectivity experiments, and many other materials science capabilities to complement neutron results. As one of the five Department of Energy Nanoscale Science Research Centers (see nsrcportal.sandia.gov), CNMS makes all of these capabilities, and the staff expertise to fully benefit from them, available free of charge to users who intend to publish the results, or at-cost for proprietary research, as described at cnms.ornl.gov. [*The CNMS at Oak Ridge National Laboratory is a DOE Office of Science User Facility.*]

4:40pm **MS-TuA8 User Opportunities at the Center for Nanoscale Materials***, Kathleen Carrado Gregar,* Center for Nanoscale Materials at Argonne National Laboratory

The Center for Nanoscale Materials (CNM) at Argonne National Laboratory is a premier user facility providing expertise, instrumentation, and infrastructure for interdisciplinary nanoscience and nanotechnology research. Academic, industrial, and international researchers can access the center through its user program for both nonproprietary (at no cost) and proprietary research.

The CNM is at the forefront of discovery of new materials, visualizing events with high resolution as they occur, understanding the physics and chemistry of energetic processes at the nanoscale, and manipulating nanoscale interactions to create useful, energy-efficient structures with new functionalities. Goals include the hierarchical integration of materials across the nanoscale to the mesoscale, in order to create energy-efficient and affordable functionality that advance the public good.

Unique capabilities at CNM include a premier clean room with advanced lithography and deposition capabilities, expansive synthesis and nanofabrication resources, a hard x-ray nanoprobe at the Advanced Photon Source synchrotron, myriad scanning probes including low temperature, ultrahigh vacuum STMs, TEMs with in situ holders and chromatic aberration-correction, a 30 TFlop supercomputer, and ultrafast optical probes. A key CNM asset includes outstanding staff with expertise in synthesis, nanophotonics, scanning probe and electron microscopy, nanofabrication, and theory, simulation and modeling. Core technological materials range from 2D layered materials to nanocrystalline diamond. All capabilities and expertise are available through peer-reviewed user proposals; access is free of charge for non-proprietary research in the public domain. CNM is one of DOE's premier Nanoscale Science Research Centers serving as the basis for a national program encompassing new science, new tools, and new computing capabilities for research at the nanoscale (https://nsrcportal.sandia.gov). Recent staff and user research highlights will be presented, painting a picture of present and future nanoscience and nanotechnology at the CNM (www.anl.gov/cnm).

The Center for Nanoscale Materials, an Office of Science user facility, is supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract no. DE-AC-02-06CH11357.

5:00pm **MS-TuA9 The Center for Integrated Nanotechnologies--Resources and Capabilities***, Dale Huber,* Sandia National Laboratories

A decade ago, five new Department of Energy user facilities, the Nanoscale Science Research Centers (NSRCs), entered full operations. These facilities offer unique capabilities, at no cost, to qualified researchers in the field. I will describe the Center for Integrated Nanotechnologies (CINT), its capabilities, and where it fits into the broader landscape of the NSRCs and the other major user facilities in the US. While this will necessarily be a broad overview, I will provide details for where and how to obtain further information including important online references, points of contact for general information, and will provide an opportunity to make connections for detailed interests. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

5:20pm **MS-TuA10 Using EMSL Capabilities in Combination with those from other User Facilities to Address Fundamental and Applied Problems***, Donald Baer, M.H. Engelhard, T.J. Law,* Pacific Northwest National Laboratory

Increasingly a wide range of advanced research tools and expertise are needed to address important scientific and societal questions. The Environmental Molecular Sciences Laboratory, EMSL, is one of several US Department of Energy user facilities provided to facilitate cutting-edge research. This talk will highlight the focus of EMSL, recent efforts to integrate activities at multiple user facilities and efforts being made to increase industrial use of user facilities. The vision of EMSL is to pioneer discoveries and mobilize the scientific community to provide the molecular science foundations that will address research priorities of the DOE Office of Biological and Environmental Research (BER) and our nation's critical biological, environmental and energy challenges. To accomplish these aims, EMSL science is focused in four areas: biosystem dynamics and design, atmospheric aerosol systems, terrestrial and subsurface ecosystems and molecular transformation. Molecular transformations that occur at surfaces and interfaces are critical in each of these areas, and EMSL provides a wide range of unique and state-of-the-art spectroscopy, microscopy, magnetic resonance and computational capabilities to advance science on these topics (www.emsl.pnnl.gov). Similar to all DOE user facilities, researchers typically use resources at EMSL for little to no cost if results are shared in the open literature, and access is provided by a proposal and peer review process. As a multi-disciplinary facility, we encourage proposals that combine instrumentation across our capability groups to advance scientific understanding. Increasingly, we are focusing on real-time *in situ* measurements in a variety of environments. Efforts over the last few years to enable cross facility access through a single proposal to examine novel ways for scientific user facilities to work together has resulted in the FICUS program—Facilities Integrating Collaborations for User Science. With the opportunity to pursue one research project at two or more institutions under FICUS, scientists can leverage disparate resources, shave years off their project times and amplify the impact of their work. Additional efforts are underway to address access policies and training reciprocity that will further streamline the user's experience and increase industrial use of these facilities as well.

Advanced Surface Engineering
Room 101C - Session SE+MS+TF-TuA

Innovations in PVD, CVD, Atmospheric Pressure Plasma and Other Surface Technologies

Moderators: Michael Stueber, Karlsruhe Institute of Technology, Germany, Robert Franz, Montanuniversität Leoben, Austria

2:20pm **SE+MS+TF-TuA1 Investigation of Critical Processing Parameters on Laser Surface Processing of Mg-Al-Zn Alloys: Impact on Corrosion Kinetics***, Michael Melia, D.C. Florian, J.R. Scully, J.M. Fitz-Gerald,* University of Virginia

Magnesium (Mg) and its alloys have been the topic of intense research over the past 15 years as the automotive and aeronautic industries strive to increase fuel efficiency by reducing the weight of vehicles. However their wide spread implementation is currently limited by poor intrinsic corrosion resistance. Preferential dissolution of the Mg matrix occurs due to the electrochemically noble secondary phases formed during traditional processing routes of Mg alloys. To mitigate the impact secondary phases (e.g. γ-Al_8Mn_5) have on corrosion, pulsed laser surface processing was employed in the ns time regime. Lasers operating in this time regime are capable of melting and solidification rates on the order of 10^9 K/s with the ability to extend the solid solubility limit of the alloying elements. The research herein shows the impact laser processing parameters have on the dissolution of the secondary phases and corrosion resistance in the Mg-Al-Zn alloy, AZ31B.

A KrF excimer laser (λ = 248 nm, pulse duration = 25 ns FWHM) was utilized with a cylindrical focusing lens, a laser spot size of 27 mm x 1.2 mm, and a pulse overlap of 95%. The processing parameters investigated include the laser fluence (0.7, 0.8 and 1.5 J/cm^2), irradiation dosage (pulse per area (PPA) = 20 to 400), and processing pressure (1 to 1280 Torr Ar). The dissolution of the γ-Al_8Mn_5 particles was observed by scanning electron microscopy equipped with a backscatter electron detector. Fiduciary image recognition was utilized to observe the change in γ-Al_8Mn_5 particle size before and after processing. Analysis of the H_2 evolution reaction rate, related to the quantity and density of electrochemically noble secondary phases, was performed by potentiodynamic polarization measurements in 0.6 M NaCl solution.

Results from the fluence study revealed significant dissolution of the γ-Al_8Mn_5 particles when processing was performed above the ablation threshold suggesting that material transport was afforded by a laser induced plasma pressure acting on the irradiated layer, increasing the extent of dissolution. This was also observed by an order of magnitude reduction in H_2 evolution reaction rate. The lowest pressure of Ar investigated, 1 Torr, consistently exhibited the smallest reduction in H_2 evolution reaction rate from the bulk material. All other processing pressures showed an order of magnitude reduction in H_2 evolution reaction rate when a fluence above the ablation threshold was used. The PPA study revealed a plateau in the reduction of the H_2 evolution reaction rate, observed dissolution of γ-Al_8Mn_5 particles, and time to breakdown of the corroding surface after 100 PPA.

2:40pm **SE+MS+TF-TuA2 Engineering a WC/Co Carbide Surface for PVD and CVD coatings***, **Aharon Inspektor,** P.A. Salvador,* Carnegie Mellon University*; D. Banerjee, C. McNerny, M. Rowe, P. Mehrotra,* Kennametal Inc.

The emergence of new coating technologies is driving the development of new cutting tools and improved metal cutting techniques. However, to reach these goals, the coating has to work in concert with the tool substrate material. Hence, building a functional surface that consists of coating and substrate working together, is a key step in the development of new cutting tool. In this paper we will look at the substrate side of the coating - surface interface and discuss how it affects the properties of the subsequent coating. The focus will be on surface engineering of WC/Co carbide surface for Physical Vapor Deposition, PVD, for Chemical Vapor Deposition, CVD, and for CVD diamond coatings. Specifically, Co mobility in the subsurface zone for CVD coatings and surface treatment for PVD coatings. Structure and properties of the resultant surface - coating combination will be presented and discussed.

3:00pm **SE+MS+TF-TuA3 Room-Temperature Ductility in Refractory Transition-Metal Carbides: Potential to Create Ultra-Tough, Flexible Thin Films***, **Suneel Kodambaka,*** University of California at Los Angeles **INVITED**

Transition-metal carbides are high-melting (> 3000 K), extremely hard (10s of GPa), mechanically robust, and chemically resilient compounds capable of operating in extreme environments and are attractive for aerospace and other industries. These hard materials are generally considered to be brittle at low temperatures. Improving their ductility, and hence toughness, is highly desirable but progress thus far has been limited by the lack of a basic understanding of the intrinsic deformation mechanisms in this class of materials. Here, using *in situ* transmission electron microscopy (TEM) coupled with uniaxial compression tests conducted on sub-µm-size pillars, in combination with density functional theory (DFT) calculations, we show that dislocations are mobile at room-temperature and lead to plastic deformation in NaCl-structured group IV and group V transition-metal carbide single crystals, zirconium carbide (ZrC) and tantalum carbide (TaC). We find that the yield strengths of ZrC crystals increase with decreasing size and ZrC(111) is softer than ZrC(100) crystals, an unexpected finding for NaCl-structured compounds. We attribute this anomalous behavior to surprisingly easy dislocation motion and low shear stresses along {001}<1-10> rather than along the commonly assumed {110}<1-10> slip systems. For TaC, in contrast to ZrC, the yield strengths are found to be independent of crystal size and orientation. Our observations suggest that multiple slip systems can be active and operate at room temperature in these hard, refractory ceramics and we expect similar behavior in other transition-metal carbides and nitrides. The insights gained from these studies may help in the development of new material architectures, such as tough and flexible membranes, for new small-scale structural applications.

4:20pm **SE+MS+TF-TuA7 Spray-Coated Carbon-Nanotubes for Crack-Tolerant Metal Matrix Composites as Photovoltaic Gridlines***, **Omar K. Abudayyeh,*** University of New Mexico*; N.D. Gapp, G.K. Bradshaw, D.M. Wilt,* Air Force Research Laboratories*; S.M. Han,* University of New Mexico

Microcracks developing in photovoltaic cells, due to growth defects or due to external mechanical factors, can lead to substantial power loss in solar cells. Microcracks can be critical as they propagate from the semiconductor bulk to the metal gridlines isolating portions of the cell and leading to decreased cell performance. In this work, multiwalled carbon nanotubes are being investigated for reinforcement of metal contacts on photovoltaic solar cells that serve as a secondary conductive network in the presence of cracks. In this effort we have focused on a silver-carbon-nanotube layer-by-layer microstructure. We present the use of a simple, cost-effective, and manufacturable method of depositing carbon nanotubes onto electroplated metal films to create metal matrix composite gridlines for photovoltaic cells. Carbon nanotubes are deposited using a spray coating method to create layer-by-layer microstructure composites. To increase adhesion strength to metal and achieve efficient metal-nanotube stress transfer, carbon nanotubes are chemically functionalized with carboxylic group prior to deposition. Initial strain failure tests show the ability of composite lines to remain electrically connected with fractures up to 28-µm-wide on average, where carbon nanotubes electrically bridge the gap. The metal-carbon-nanotube composites are electrically characterized though current-voltage (*I-V*) sweeps. Our composite lines can carry current densities ranging from 500 to 2500 A/cm^2 in the presence of cracks (5, 10, and 15-µ m-wide). MMC gridlines are successfully integrated on commercial triple-junction solar cells with measured fill factor and efficiency 86% and 26.8% respectively, closely comparing to current triple-junction cells with standard metallization. Dark *I-V* measurements indicate further improvement in the series and shunt resistances of the cells with the optimization of MMC integration process.

4:40pm **SE+MS+TF-TuA8 Atmospheric Pressure Plasma Enhanced CVD of High Quality Silica-Like Bilayer Encapsulation Films***, **Fiona Elam,*** FUJIFILM Manufacturing Europe B.V., Netherlands*; A.S. Meshkova,* DIFFER, Netherlands*; S.A. Starostin, J.B. Bouwstra,* FUJIFILM Manufacturing Europe B.V.*; M.C.M. van de Sanden,* Dutch Institute for Fundamental Energy Research (DIFFER), Netherlands*; H.W. de Vries,* DIFFER, Netherlands

Atmospheric pressure-plasma enhanced chemical vapour deposition (AP-PECVD) is an innovative technology that can be integrated into many existing manufacturing systems to facilitate the mass production of functional films; specifically encapsulation foils. These barrier films are essential to the flexible electronics industry, envisioned to protect devices such as flexible solar cells and organic light emitting diodes against degradation from oxygen and water.

Roll-to-roll AP-PECVD was recently used to produce smooth, 90 nm silica bilayer thin films comprising a ‘dense layer’ and ‘porous layer’ that demonstrated exceptionally good encapsulation performance with effective water vapour transmission rates in the region of 6.9×10^{-4} g m^2 day^{-1} (at 40°C, 90% relative humidity). By using the same material in the multilayer film architecture, and by having AP-PECVD as the deposition method, rendered this investigation highly industrially and commercially relevant to the eventual large scale production of flexible encapsulation foils. It was discovered that increasing the input energy per precursor gas molecule during the deposition of the dense layer, resulted in an improved encapsulation performance. However, the individual role performed by each layer in the overall success of the bilayer films is not yet fully understood, nor is the potential for energy conservation by varying process throughput.

A glow-like AP dielectric barrier discharge in a roll-to-roll set-up was used to deposit silica bilayer thin films onto a polyethylene 2,6 naphthalate substrate by means of PECVD. Tetraethyl orthosilicate (TEOS) was used as the precursor gas, together with a mixture of nitrogen, oxygen and argon. In each case, the deposition conditions for the synthesis of the dense layers were varied in order to study the effect of input energy per TEOS molecule and process throughput on the chemical composition and porosity of the layer. Deposition conditions for the porous layers were kept constant, with process throughput the only exception. Each film was characterised in terms of its water vapour transmission rate, its chemical composition and its morphology as a function of the input energy per TEOS molecule during the dense layer deposition and overall process throughput.

For the first time in AP-PECVD, it was found that the porous layer plays a critical role regarding encapsulation performance and surface smoothening of silica bilayer films. Due to increased throughput, the bilayer architecture also enables a 50% reduction in deposition energy consumption per barrier area, with respect to single layer silica films of equivalent encapsulation performance and thickness.

5:00pm **SE+MS+TF-TuA9 Plasma Polymerization of Organic Coatings at Atmospheric Pressure: Relationship between the Precursor Chemistry, the Plasma Chemistry and the Final Coating Chemistry**, *B. Nisol, N. Vandencasteele, J. Hubert, C. De Vos, J. Ghesquière, D. Merche,* ***François Reniers,*** Université Libre de Bruxelles, Belgium

The synthesis of organic coatings using plasma technologies has been developed since many decades. This paper investigates a full series of organic coatings, synthesized in the same dielectric barrier discharge system, in the same operational conditions. The >10 precursors vary from fully saturated molecules, with or without heteroatoms (hexamethylnonane, CxCly, CxFy), to anhydrides, acrylates, with or without double or triple bonds. It is shown that the presence of double bonds not only significantly increase the polymerization rate, but also protect the ester function in acrylates. A combined effect of the plasma power and the presence of double bonds on the C/O ratio is observed for all the relevant precursors used. Correlation between the plasma chemistry and the fragment pattern in the gas phase, as recorded by atmospheric mass spectrometry, with the final chemical composition of the coatings, determined by XPS and FTIR is established. Coatings properties can be easily tuned either by combining precursors, by varying the plasma power or by changing the main plasma gas. By an appropriate combination of the plasma parameters and the precursor, very high deposition rates can be achieved, highly hydrophobic or hydrophilic coatings can be synthesized. These macroscopic results are interpreted in terms of plasma properties, and chemical reactivity.

This work was supported by the Belgian Federal Government (IAP research project P7/34 – Physical Chemistry of Plasma Surface Interactions).

5:20pm **SE+MS+TF-TuA10 Innovations in Atmospheric Pressure Plasma Technologies for Surface Engineering**, ***David Ruzic,*** *Y.L. Wu, L. Na, S. Hammouti, I.A. Shchelkanov,* University of Illinois at Urbana-Champaign **INVITED**

The growing need for high efficiency-low cost coating tools for large area surfaces drives research efforts for development of innovative techniques. One of the options is an Evaporative Coating at Atmospheric Pressure process (ECAP). The principal of this deposition method is an evaporation of a material, with-in a plasma environment. The appealing advantage of this deposition technique is its atomic nature, and its environmentally safe process as no harmful chemicals compounds are used. With ECAP the evaporated material atoms end up deposited molecule-by-molecule or atom-by-atom as in a Physical Vapor Deposition but without the need for a vacuum chamber. This effect is achieved by using a thermal energy from the microwave plasma, when solid 99.99%+ purity metallic and ceramic target such as Al, Sn, Cr, Au, Ag and $AlCl_3$ could be evaporated and then produce a PVD-like coating on a work piece. The tool is designed to have the evaporated material being submerged into the center of the atmosphere microwave discharge. As the result evaporation occur in a controlled environment where a pure metals can be deposited or their compounds. For example in the aluminum case, a pure alpha phase of Al_2O_3 can be deposited using oxygen from the environment, or if a metallic coating of such a reactive metal as Al, is desired, the deposition can be performed in an pure Ar argon environment with the help of a special gas curtain. The tool provides deposition rate for metals as high as 1-5 um/min with high adhesion. The measured adhesion for copper on steel was at least 250 g/mm^2. The ECAP technology opens broad possibilities for surface processing at atmosphere without environmental impact.

6:00pm **SE+MS+TF-TuA12 Solid-state Dewetting: Control and Applications**, ***Lukasz Borowik,*** *Y. Almadori, N. Chevalier, J.-C. Barbé,* CEA, LETI, MINATEC Campus, France

The dewetting of ultrathin silicon layers, induced by the thermal budget, is an issue to develop Silicon On Insulator (SOI) and Silicon Germanium On Insulator (SGOI) based technologies. However, dewetting can be controlled to obtain well-arranged agglomerates with similar size or even inhibited. This experimental study aims at demonstrating: the effect of the strain, surface contamination, ion sputtering on the dewetting mechanism, and further applications of dewetting thanks to interfacial reaction between agglomerates and the silicon dioxide. For that purpose, we present the results obtained on: (1) (001) oriented ultrathin (8-22 nm) silicon layers on silicon dioxide, (2) (001) oriented 12 nm silicon-germanium layers on silicon dioxide. In order to understand the dewetting mechanism, samples were heated up to ~800°C under ultra-high vacuum (1×10^{-9} mBar) during tens of minutes. The dewetted samples were characterized by Atomic Force Microscopy (AFM) to put in evidence the influence of the different factors on dewetting mechanism.

In first part of our presentation we will present various methods to control dewetting process by using different parameters such as: strained silicon, [1] surface contamination [2] or argon pre-sputtering. [3] These parameters allow tuning agglomerates size, shape and density. In second part of the talk we will show possible applications of dewetting to form porous silicon dioxide via agglomerates interfacial reaction, and finally how to master stoichiometry of silicon germanium agglomerates. [4] All these methods are promising since permit an easy and fast implementation, it is thus of real interest, since it opens up Si and SiGe agglomerates with tuned Ge concentration to application in innovative technologies.

This work was performed in the frame of the ANR LOTUS project. The measurements were realized on the CEA Minatec Nanocharacterization Platform (PFNC).

References:

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[2] Ł. Borowik et al. Thin solid films 527, 133–136 (2013)

[3] F. Leroy et al. Surface Science Reports accepted

[4] Y. Almadori et al., 120, 7412–7420 (2016)

Manufacturing Science and Technology
Room Hall D - Session MS-TuP

Aspects of Manufacturing Science and Technology Poster Session

MS-TuP2 Study of Mechanical Properties of Nanographene/Al Composite Materials for Purpose of Industrial Applications*, **Yusuke Oguro**, A. Matsumuro,* Aichi Institute of Technology, Japan

Basic science of graphene with various superior characteristics has been made clear rapidly on the frontier technology. Especially, development of superior nano-scale electronic devices and bio systems has been studied energetically. Nevertheless, various surprising mechanical properties of graphene have been not attracted great attention, such as extreme low density, tensile strength with 100 times stronger than steel by weight, Young's modulus with 1 TPa and more flexible than rubber. Since our original successful isolation techniques of creating single layer nanographene sheets from nanographene with a several sheet and uniforming dispersion of nanographene within based materials, we have been challenging in fabrication innovative nanographene reinforced Al composite sintered materials and established the fabrication method. It has already been demonstrated that Vickers hardness of nanographene/Al composite pellet-formed sintered materials showed the maximum value of 323 Hv, which means about 5 times up in comparison with that of Al bulk material, and the density decreased down to 2.45 g/cm^3 . So, the specific strength increased up to 414 kN・m/kg. The value increased up to 1.4 times for sintered Al material, and the value surprisingly exceeded that of commercial used magnesium alloys. These results would suggest bringing a change in the concept of industrial use materials.

In this study, we investigated possibility that industrial applications of our nanographene /Al composite materials would take advantage of their bulk properties. Standard flat-plate type specimens consisted of our nanographene/Al composite materials were fabricated under the same sintered condition in order to compare various mechanical properties of the standard data. Bending, tensile and fatigue mode tests were performed in precisely. Elastic and fracture properties were analyzed using four-point bending test apparatus without few artificial errors. The results revealed that Young's modulus increased from 40 GPa of sintered Al up to 45 GPa of nanographene/Al composite materials. Fracture characteristics showed that the breaking stress of the composite material showed drastic improvement up to 75 %, and the breaking strain of the composite material also increased up to 70 %. These great improvements of mechanical properties can be attributed to reinforcement effect of nanographene. Other mechanical properties tests should show the same tendency. Therefore, nanographene/Al composite materials give us excellent possibility of the innovative industrial use materials with a promising future.

MS-TuP3 Development of High-Strength Resin Composite Materials Reinforced with Nanocarbon for 3D Printing Manufacturing*, **Hiroaki Sakaguchi**, A. Matsumuro,* Aichi Institute of Technology, Japan

Now technologies related to 3D printing are strongly leading the industrial revolution in all fields. However, unavoidable basic technical problems of 3D printing have prevented from an ideal technology of manufacturing products with conventional characteristics. This serious problem must be caused by the layer structure of products used by additive process, and the insufficient mechanical strength of the molding materials specific to each type of 3D printer.

In this study, we strongly focused on develop innovative high strength resin-based composite materials reinforced with nanocarbon, i.e. C_{60}, CNT and graphene. These nanocarbon show extraordinary mechanical, physical and chemical properties with each superior characteristics. It is worth noticing that the mechanical properties, such as a strength, elastic modulus, hardness and so on, can be investigated incommensurably high values compared with those of commercial materials. Furthermore, these kinds of nanocarbon have superior geometric and nanometer-scale dimension characteristics as reinforcement materials considering various 3D printing processes. We researched the possibility of the application of nanocabon composite materials with ABS base resin used widely in industry for the purpose of innovative strength increment of 3D printed products. To clarify each characteristic of C_{60}, CNT and graphene to ABS resin composites, these three-type nanocarbon composite specimens were fabricated. The fabrication method with a uniform composite material reinforced with distributed each nanocarbon have established on our own. The uniform and dispersion of nanocarbon around ABS base powder particles should be the key point for fabrication of specimen by melt and solidification method. We applied our original technique using ultrasonic vibration with isopropyl alcohol as a solvent for 4 hours. And we investigated the optimal composition rates of each composite material. Each pellet-type solidified specimens was prepared at about 500 K for 30 minutes by atmospheric furnace cooling.

As the result of one of representative mechanical property, Vickers hardness for each specimen excellently increased up to about 10 % for 7.0 wt.% C_{60} and 7 % for 1.0 wt.% graphene in comparison with the value of pure ABS resin bulk sample. The evaluation of important other mechanical properties such as pulling strength, break strength, fatigue strength, elastic modulus and so on have been examined in detail. These results should give us great conclusive evidence for achievement of this study.

MS-TuP4 Development of Innovative 3D Printer with Superior Multifunctional Surface Modification*, **Kentaro Horiuchi**, Y. Hasegawa, A. Matsumuro,* Aichi Institute of Technology, Japan

Direct manufacturing process using 3D printing technologies is spreading rapidly in all fields. Many difficult problems should be overcome in order to establish 3D printing technologies as the general industrial products manufacture method. One of the representative problems is the durability or strength of the products. But now this problem is overcoming rapidly by passionate R&D for specific products, and the material stead studies with various characteristics to satisfy the requirement of each product have conducted wide fields. Furthermore, studies of 3D printing systems with superior functions of various post processes at molding simultaneously are indispensable. The particular expected functions are removal of support materials from printed products, full-color painting, and surface modification for improvement of the mechanical, electrical, chemical and biological properties. The development of removal of support materials and full-color molding have been already solved using automatic machine cutting machine and ink-jet-type paint system equivalent to the same mechanism of a printer used in the office as typical examples, respectively. On the other hand, the surface modification technologies have been applied in wide industrial field in order to control great various characteristics of hardness, wear resistance, electrical conductivity, thermal conductivity and so on at arbitrary places of metal products using vacuum apparatus. So the surface modification technologies should be necessary applied to 3D printing products using resin materials.

In this study, our unique 3D printing system with multifunctional surface modification has been developed using the conventional air brush painting technique for resin and metal products. The air brush apparatus consist of only three parts: paint flow nozzle, paint container and air compressor. So high extensibility of the function and the mechanism of the spray system, the synchronization with the printer and numerical flexibility of the air brush can be possible easily. For confirmation of our trial apparatus, the full-color painting on the surface of 3D printing products has been succeeded by mix-spraying from three air brushes with suitable spray ratio using paint of cyan, magenta and yellow. The commercial paints using a air brush or a spray gun are widely used due to improve many kinds of charming surface characteristic such as lubricity, electric property, glossiness, fireproof property, corrosion resistance, luminous color property, et cetera. Therefore we could find great attractive possibility of realization of development of the innovative 3D printer with multifunctional surface modification.

MS-TuP5 Controlling the Diameter, Uniformity, and Spatial Distribution of Electrospun PVDF Nanofibers through Experiment and Simulation*, **Omar Ali**, T. Grier, A. Ueda, C. Marvinney, S. Avanesyan, C.S. Carson, W.E. Collins,* Fisk University*; J. DeCoste,* US Army Research, Development, and Engineering Command*; R. Mu,* Fisk University

Polyvinylidene fluoride (PVDF) is a polymer which has important applications in insulation, sensors, and battery production. The thermal, electrical, elastic, and morphological behaviors and properties of PVDF, on which the applications depend, are intimately related to the structure of the polymer on various physical length scales. Retained PVDF solid structures on different scales are also related. For example, stretching and poling a PVDF membrane causes the PVDF monomer chain to change phase from the non-polar alpha phase to the polar beta phase, which imparts significant piezoelectric and pyroelectric properties. Controlling the properties of PVDF by tuning the solid structures is, therefore, of key importance for various applications. Efforts have been made by our team into electrospinning technology, which is a relatively simple and scalable method of nanofiber production, for energy storage and biomedical applications.

We have investigated the effect of varying the electrospinning conditions and polymer solution properties on the diameter size and uniformity, and the spatial distribution of the produced PVDF nanofibers. Experimentally, we varied both the concentration of PVDF in the initial solution, and the voltage applied during electrospinning. Using optical imaging and scanning electron microscopy (SEM), we obtained a set of images for several voltage-concentration parameter pairs. Based on the experimental data, we were able to examine the diameter and uniformity as functions of the concentration and applied voltage. Additionally, we simulated different electric field distributions in an effort to design a method for controlling fiber deposition area.

The results indicate that increased concertation can lead to a significant increase in average fiber diameter. Fibers produced at lower concentrations are not only thinner, but also had significantly less uniform diameters and a greater number of beads in the fibers. The effect of voltage on diameter size and uniformity was somewhat less clear. There does not appear to be any correlation between voltage and average diameter size. However, voltages around the middle of the range we studied seem to lead to slightly more uniform fibers. Further, COMSOL simulation has been conducted and showed that using conduction rings to manipulate the electric field lines can make the field diverge sufficiently at first to allow for the electrospinning process to take place and then converge to direct the fibers to a smaller area of the grounded substrate. Such control over where the fibers are distributed can be very useful when producing fibrous mats and other macro-structures from nanofibers.

MEMS and NEMS
Room 102B - Session MN+MS-FrM

Radiation Effect in Emerging Micro/Nano Structures, Devices, and Systems

Moderators: Michael Alles, Vanderbilt University, Philip Feng, Case Western Reserve University

8:20am **MN+MS-FrM1 Radiation Effects in Emerging MEMS/NEMS Devices***, **Jacob Calkins,*** Defense Threat Reduction Agency

Recent advance in MEMS based computing, signal filtering, and sensing offer significant opportunities for reducing the size, weight, cost, and power requirements of Department of Defense (DoD) systems while improving reliability in extreme environments. Natural and manmade high radiation environments are one of the most extreme environments where DoD systems must both survive and continue to function. This high radiation environment includes satellites in Earth orbit and robots or unmanned vehicles responding to nuclear disasters. This Defense Threat Reduction Agency (DTRA) basic research program seeks to investigate, understand, and model the fundamental effects of radiation on MEMS/NEMS devices.

Gamma rays, x-rays, as well electrons and ions are expected to deposit charge in dielectrics and may also charge the sealed gas environment in which the devices operates. Neutrons, protons, and ions may change the underlying mechanical properties, optical properties, and ferroelectric or piezoelectric properties of MEMS materials. All types of radiation may alter the surface chemistry and morphology causing changes in the performance of physical and electrical contacts. It is expected that MEMS devices will be resistant to upsets or changes in state due to individual strikes by high energy particles. However, nanoscale devices may be susceptible to either the physical impact or the associated charge deposition. It is important to understand these fundamental effects of radiation on MEMS/NEMS devices and materials before they are incorporated in critical DoD systems.

This talk introduces the DTRA basic research radiation effects program and the radiation effects in MEMS/NEMS topic.

8:40am **MN+MS-FrM2 Effect of Top Electrode Material on Radiation-Induced Degradation of Ferroelectric Thin Films***, **Nazanin Bassiri-Gharb,** S.J. Brewer, C.Z. Deng, C.P. Callaway,* Georgia Institute of Technology*; M.K. Paul,* Woodward Academy*; K.J. Fisher,* Riverwood International Charter School*; J.E. Guerrier, J.L. Jones,* North Carolina State University*; R.Q. Rudy, R.G. Polcawich,* Army Research Laboratory*; E.R. Glaser, C.D. Cress,* Naval Research Laboratory **INVITED**

The effects of gamma irradiation on the dielectric and piezoelectric responses of $Pb[Zr_{0.52}Ti_{0.48}]O_3$ (PZT) thin films were investigated for structures with conductive oxide (IrO_2) and metallic (Pt) top electrodes. All samples showed a general degradation of various key dielectric, ferroelectric, and electromechanical responses when exposed to 2.5 Mrad(Si) ^{60}Co gamma radiation. However, the low-field, relative dielectric permittivity, ε_r, remained largely unaffected by irradiation in samples with both types of electrodes. Samples with Pt top electrodes showed substantial degradation of the remanent polarization and overall piezoelectric response, as well as pinching of the polarization hysteresis curves and creation of multiple peaks in the permittivity-electric field curves post irradiation. The samples with oxide electrodes, however, were largely impervious to the same radiation dose, with less than 5% change in any of the functional characteristics. The results suggest a radiation-induced change in the defect population or defect energy in PZT with metallic top electrodes, which substantially affects motion of internal interfaces such as domain walls. Additionally, the differences observed for devices with different electrode materials implicates the ferroelectric-electrode interface as either the predominant source of radiation-induced effects (Pt electrodes) or the site of healing for radiation-induced defects (IrO_2 electrodes).

9:20am **MN+MS-FrM4 Radiation Survivability of MEMS Microelectronic Circuits with CNT Field Emitters***, **Jason Amsden**, E.J. Radauscher, T. von Windheim,* Duke University*; K.H. Gilchrist,* RTI International*; S.T. Di Dona, Z.E. Russell,* Duke University*; L.Z. Scheick,* Jet Propulsion Laboratory, California Institute of Technology*; J.R. Piascik,* RTI International*; C.B. Parker,* Duke University*; B.R. Stoner,* RTI International*; J.T. Glass,* Duke University **INVITED**

Radiation survivability of MEMS microelectronic circuits with carbon nanotube field emitters

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INVITED ABSTRACT

Solid-state technology dominates the consumer electronics market because of the low cost associated with large-scale integration. However, there are numerous applications in which solid-state devices are unreliable or do not provide adequate performance, particularly applications with military systems operating in high radiation environments. In these applications, vacuum microelectronic devices present an attractive alternative. Despite the performance advantages, the use of vacuum electronics has been limited because there is no versatile and reliable microscale platform that enables integration of large numbers of vacuum circuit elements on a single substrate. To address this need, RTI International in collaboration with Duke University has been developing a Microelectromechanical systems (MEMS) platform that enables integration of high-performance microelectronic vacuum components into functional circuits on a single silicon substrate.[1] We have demonstrated a wide variety of vacuum electronic devices including vacuum triodes and ion sources using a freestanding panel approach fabricated with the Polysilicon Multi-User MEMS Process (PolyMUMPS) with integrated carbon nanotube field emission cathodes. While these devices avoid the radiation-induced charge carrier problems in solid-state devices, other effects of radiation on this MEMS platform have not been studied. The presentation will present our preliminary findings on the radiation effects on this vacuum microelectronics platform.

1. Stoner, B. R.; Piascik, J. R.; Gilchrist, K. H.; Parker, C. B.; Glass, J. T., A Bipolar Vacuum Microelectronic Device. *Electron Devices, IEEE Transactions on* **2011**, *58*, 3189-3194.

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10:00am **MN+MS-FrM6 Radiation Effects in Integrated Photonics and Nano-OptoMechanical Systems***, Q. Du,* Massachusetts Institute of Technology*; B Li,* University of Minnesota*; D. Ma, A. Agarwal, **Juejun Hu**,* Massachusetts Institute of Technology*; M. Li,* University of Minnesota **INVITED**

We present integrated photonic devices and Nano-OptoMechanical Systems (NOMS) as highly sensitive

and accurate platforms for quantifying radiation effects in optical materials and MEMS/NEMS systems.

Our approach benefits from the high precision of optical interrogation techniques. Further, compared to

traditional capacitive or piezoelectric actuated devices, NOMS are immune to extrinsic

degradation associated with driving circuit and metal contact failure or charge-trapping in dielectrics, and

are thus ideal test vehicles to measure radiation-induced, intrinsic optical and mechanical material

property modifications. In this talk, we will discuss design, fabrication and characterization of optical and

NOMS devices based on Si, SiN and SiC materials as well as gamma-ray radiation effects in these

devices. Defect identities responsible for the observed optical and mechanical property shifts are clarified

through spectroscopy and microscopy characterization techniques to unravel the radiation damage

mechanisms in these devices.

10:40am **MN+MS-FrM8 Radiation Effects in 2D Materials and Nano Electrical Mechanical Devices***, **Michael Alles**, K.I. Bolotin,* Vanderbilt University*; A. Zettl,* University of California at Berkeley*; B. Homeijer,* Sandia National Laboratories*; J.L. Davidson, R.D. Schrimpf, R.A. Reed, R.A. Weller, D.M. Fleetwood, W. Liao, R.J. Nicholl,* Vanderbilt University **INVITED**

The dramatic reduction in size and power consumption offered by micro- and nano-scale electromechanical systems (M&NEMS) offer compelling advantages for adoption in many areas, including space and military systems. Mission reliability and survivability of next-generation space-borne systems depends critically upon a detailed understanding of the radiation response and reliability of the constituent devices when exposed to the relevant radiation environment. M&NEMS may be employed for a variety of sensor and actuation applications, oscillators, and have potential to be incorporated as high-reliability solid-state (logic) switches.

While there has been considerable study of radiation effects on some of the common materials (single and poly crystalline silicon, and silicon-dioxide), the study in the context of MEMS has been more limited, and mainly experimental. The general indication is that depending on the operating principal, MEMS can be sensitive to radiation-induced charging of insulators, and to displacement damage at very high exposure doses of energetic particles.

More recently, fabrication process, namely the ability to create nano-scale structures, and the introduction of new materials (esp. 2D and CNTs), have expanded the opportunities for M&NEMS. The Defense Threat Reduction Agency (DTRA) has recently initiated multiple projects in the study of various aspects and configurations of M&NEMS. This talk will discuss a specific project which seeks to advance the understanding of the effect of radiation on the relevant electro-mechanical properties of the constituent materials and structures, focusing on 2D materials and CNTS, and the implications for space and military applications.

The key question for this work is: *How does radiation damage to constituent materials impact the mechanical and electrical basis of operation of M&NEM structures?* In particular, cumulative damage by non-ionizing energy loss can, in principle, alter the mechanical properties of structures such as cantilever's and 2D membranes, and surface effects and trapped charge in insulators can impact electrical operating conditions. Presently, the extent to which such effects impact the operation of advanced M&NEM devices is unclear. This particular project combines expertise and experience in materials science, M&NEMS, and radiation effects of the University of California at Berkeley, Sandia National Laboratories, and Vanderbilt University to conduct a systematic study of the impact of relevant radiation types on the key operating properties of novel M&NEM structures, and to capture observed effects in finite element models.

11:20am **MN+MS-FrM10 Influence of Radiation on MEMS Oscillators***, **Bruce Alphenaar**,* University of Louisville*; M.L. Alles, H. Gong,* Vanderbilt University*; P. Deb Shurva, J.T. Lin,* University of Louisville*; J.L. Davidson,* Vanderbilt University*; S. McNamara, K. Walsh,* University of Louisville*; W. Liao, R.A. Reed,* Vanderbilt University **INVITED**

The beneficial size, weight, and power requirements provided by micro-electromechanical (MEMS) and nanoelectromechanical (NEMS) memory, logic and sensors for systems operating in extreme radiation environments make it essential that the effects of radiation on MEMS/NEMS devices be investigated. Here we report on the influence of radiation on a micro-scale oscillator consisting of a electrostatically driven silicon cantilever fabricated from a silicon-on-insulator wafer. The position of the cantilever is detected by the piezoresisitve change in an asymmetric silicon beam supporting the cantilever. Prior to radiation exposure, the frequency response of the oscillator remains very stable, provided temperature and pressure are kept constant. Four different radiation sources are used, covering a range of excitation energies: 1) UV B (265 nm) photons 2) 10 keV X-rays 3) 0.8 MeV protons and 4) 2.0 MeV protons. Exposure to 10 keV X-rays at 10.5 krads/minute causes the resonant frequency to shift downwards by approximately 0.4 Hz (50 ppm change) following 2.1 Mrads irradiation. Once the X-ray exposure is removed, the device returns to its original state after an annealing time of 10 hours. The long annealing time and a correlation to the silicon resistance change shows that sample heating is not an important factor. A comparison to UV B results suggests that the frequency shift is related to charge accumulation in the silicon cantilever. Exposure to 0.8 MeV shows permanent change to the resonant frequency suggesting that displacement damage has occurred. This work was funded by the Defense Threat Reduction Agency under contract HDTRA-15-1-0027.

12:00pm **MN+MS-FrM12 Radiation Effects on Silicon Carbide (SiC) Nanomechanical Devices***, **Philip Feng**,* Case Western Reserve University

We report on exploratory research effort on investigating fundamental radiation effects in resonant-mode and contact-mode microelectromechanical systems (MEMS/NEMS) enabled by silicon carbide (SiC) micro/nanostructrues. We calibrate and compare the radiation effects in a few selected MEMS/NEMS resonators – namely, SiC microdisk resonators, SiC membrane resonators, SiC-on-insulator (SiCOI) cantilever beam resonators, and resonant switches.

We focus on discovering fundamental effects of SiC MEMS/NEMS devices due to exposure to radiation sources representative of space and nuclear environments, and attaining comprehensive, in-depth understandings of how these radiations affect the key attributes and performance of the devices. The main properties and metrics include resonance frequency, quality (*Q*) factor, multimode responses, off-state leakage, on-current, on-voltage, switching speed, surface and nanocontact properties, reliability and lifetime, *etc*.

In this work, we have carefully performed experiments on extensive testing of multimode SiC MEMS/NEMS resonators and resonant switches, before and after controlled radiation exposure with designed dosage and energetic parameters of the radiation sources. Furthermore, we have done extensive computer simulation studies on the effects of implanting protons (hydrogen ions, H+) into SiC thin layers on silicon (Si) substrate and on SiO2, and explore the ion implantation conditions that are relevant to experimental radiation of SiC device layers.

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