

## Advanced Surface Engineering

### Room 101D - Session SE+2D+EM-WeA

#### Multifunctional Thin Films and Coatings

**Moderators:** Jolanta Klemberg-Sapieha, Ecole Polytechnique de Montreal, Canada, Michael Stueber, Karlsruhe Institute of Technology, Germany

**2:20pm SE+2D+EM-WeA1 Investigation of H<sub>2</sub>S Poisoning Process on Composite Material Made of Metal Oxides and Carbon Nanotubes, Yichen Duan, A.V. Teplyakov, University of Delaware**

The composite material based on a combination of SnO<sub>2</sub>, CuO and acid-treated carbon nanotubes is considered to be promising for H<sub>2</sub>S sensing applications. By applying large dosage of H<sub>2</sub>S (1% in volume) to the composite and evaluating the chemical changes spectroscopically via XPS, SEM and EDS, the poisoning mechanism of this type of sensing material is revealed. Specifically, metal sulfides (CuS and SnS<sub>2</sub>), metal sulfate (Sn(SO<sub>4</sub>)<sub>2</sub>) and thiols are formed as the products following the introduction of H<sub>2</sub>S. In fact, all the three components of the material are affected by H<sub>2</sub>S. Moreover, when oxygen is present, metal sulfides can be reoxidized back into metal oxides while metal sulfate and thiols remain unchanged. The measurements of the model sensor response also support the assessment of the poisoning process.

**2:40pm SE+2D+EM-WeA2 Graphene-family Nanomaterials Co-assembled with Nanostructured Cobalt Oxide Polymorphs as Hybrid Supercapacitive Electrodes and Enzymeless Glucose Detection Platforms, Sara Carrizosa, B. McDonald, S. Gupta, Western Kentucky University**

Graphene-family Nanomaterials Assembled with Cobalt oxides and Cobalt Nanoparticles As Hybrid Supercapacitive Electrodes and Enzymeless Glucose Detection Platforms We developed graphene/cobalt oxides and graphene/cobalt nanoparticles hybrid assembly highlighting the impacts of nanoscale surface morphology and microstructure producing tailored interfaces for improved electrochemical and electroanalytical properties. Molecular electrodeposition and facile hydrothermal synthesis techniques followed by thermal treatment are demonstrated to be effective approaches for nanoengineered electrochemical electrodes. Hybrid electrodes consisting of supercapacitive graphene nanosheets and pseudocapacitive nanostructured cobalt oxide polymorphs (CoO and Co<sub>3</sub>O<sub>4</sub>) as well as cobalt nanoparticles (CoNP) synthesized on two- and three-dimensional graphene nanosheets facilitate chemically bridged (covalently and electrostatically anchored) yet tunable graphene-cobalt interfaces. The intrinsic microstructure and surface of these hybrids were characterized by electron microscopy combined with elemental mapping, X-ray diffraction and Raman spectroscopy. The graphene/cobalt hybrid composites were investigated as asymmetric supercapacitor cathodes and as electroanalytical platforms for enzymeless detection of glucose. We demonstrate that Co<sub>3</sub>O<sub>4</sub>/ErGO and Co<sub>3</sub>O<sub>4</sub>/multilayer graphene hybrids are capable of delivering high specific capacitance of > 600 F g<sup>-1</sup> at a current density of 10 A g<sup>-1</sup> is achieved when the mass ratio of Co<sub>3</sub>O<sub>4</sub> to ErGO is equal to 80:20 as compared with other hybrids with excellent cycling stability in voltage range 0–1.2 V. It can also detect glucose with ultrahigh sensitivity of 4.57 mA mM<sup>-1</sup> cm<sup>-2</sup> and a remarkable lower detection limit of < 50 nM in the following order Co<sub>3</sub>O<sub>4</sub>/rGO<sub>HT</sub> < CoO/ErGO < CoNP/MLGNiFoam < Co<sub>3</sub>O<sub>4</sub>/MLGNiFoam. We attribute all of these remarkable findings due to interplay of (a) open pore system beneficial to ion diffusion and transport kinetics owing to larger accessible geometric surface area, (b) three-dimensional topologically multiplexed and highly conductive pathways provided by MLG, ErGO and rGO<sub>HT</sub> nanoscaffold architectures to ensure rapid charge transfer and electron/ion conduction (< 10 ms), and (c) synergistic integration of functional nanomaterials devoid of graphene sheets agglomeration with optimal transition metal (oxides) nanoparticles loading.

**3:00pm SE+2D+EM-WeA3 Cross-Bonding between Silicon, Silica and III-V Surface at the Nano-Scale Using Energy Analysis via Three Liquid Contact Angle Analysis (3LCAA) to achieve Hermetic Wet NanoBonding™, Ashley Mascareno, SiO<sub>2</sub> NanoTech LLC/Arizona State University Physics Dpt; N.X. Herbots, SiO<sub>2</sub> NanoTech LLC; C.F. Watson, SiO<sub>2</sub> NanoTech LLC/Arizona State University Physics Dpt**

Mobile ions such as Na, percolate from saline environments into marine and atmospheric sensors and limit their reliability to less than a week. Implantable glucose monitors for diabetics require replacement about every 3-7 days, with finger blood samples re-calibration daily. Hermetic bonding can yield economic, medical, and human benefits by extending

lifetime of such integrated sensors from days to years. Si-based surfaces such as thermally-grown amorphous a-SiO<sub>2</sub> on Si(100), and on III-V surfaces can be hermetically bonded with Wet NanoBonding™ to yield dense, hermetic cross-bonding. In Wet Nonbonding™, planarization is first accomplished at the nano-scale, then a-SiO<sub>2</sub> is etched with HF, while a 2 nm precursor β-cSi<sub>2</sub>O<sub>4</sub>H<sub>4</sub> phase is grown on Si(100) to initiate cross-bonding. Next, both surfaces are put into mechanical contact in a class 10 clean-room and nano-bonded under low temperature (T<180° C) steam pressurization.

Modifying the surface energy components of 2 surfaces can help optimize hermeticity by increasing the density of cross-bonding. Surface energy γ<sup>T</sup> can be measured via 3 Liquid Contact Angle Analysis (3LCAA) using the the Van-Oss theory, which models γ<sup>T</sup> for semiconductor and insulators in 3 interactions: (1) Lifschitz-Van der Waals molecular dipole interactions γ<sup>LW</sup>, (2) electron donor interactions γ<sup>+</sup>, and (3) electron acceptor interactions γ<sup>-</sup>. Successful NanoBonding™ can occur between one surface with high γ<sup>+</sup> and one with high γ<sup>-</sup>. 3LCAA extracts these from contact angles between several liquids with known surface energies and the surface. Sessile drop analysis with water, glycerin, and α-bromonaphthalene is conducted in a Class 100 hood using 4-8 drops per liquid for statistical accuracy. RCA cleaned Si(100) and Si(100) terminated with 2-nm β-cSi<sub>2</sub>O<sub>4</sub>H<sub>4</sub> via the Herbots-Atluri (H-A) process are used, in combination with Rapid Thermal Anneal and Oxidation (RTA and RTO), to grow a-SiO<sub>2</sub> [3]. The γ<sup>T</sup> of hydrophilic RCA-cleaned Si(100) is 47.3±0.5 mJ/m<sup>2</sup>, 25% higher than the γ<sup>T</sup> of ordered, hydrophobic β-cSi<sub>2</sub>O<sub>4</sub>H<sub>4</sub> Si(100), 37.3±1.5mJ/m<sup>2</sup>, and 30% higher than RTO oxides 34.5±0.5 mJ/m<sup>2</sup>. Interactions from γ<sup>LW</sup> account for 90-98±2% of γ<sup>T</sup> in ordered oxides, but only 76.5±2.0% of those in hydrophilic surfaces. Thus, 3LCAA detects changes in surface reactivity from defects, impurities, and dangling bonds. While γ<sup>+</sup> accounts for little to none of γ<sup>T</sup> for all but one surface, 180° C annealing during Wet NanoBonding significantly increases γ<sup>-</sup> in β-cSiO<sub>2</sub>. Conversely, HF etching significantly increases γ<sup>-</sup> for a-SiO<sub>2</sub>. When matching acceptor with donor interactions between surfaces via 3LCAA, cross-bonding density appears to increase, and NanoBonding™

**3:20pm SE+2D+EM-WeA4 Porous Materials for Solid Phase Microextraction by Sputtering and Chemical Vapor Deposition, Tuhin Roychowdhury, A. Diwan, B. Singh, M. Kaykhai, M.R. Linford, Brigham Young University**

Solid phase microextraction (SPME) is an important sampling tool. It consists of placing a coated fiber above a sample (headspace mode) or immersing it in a liquid such that molecules (analytes) of interest can be selectively extracted and concentrated. The captured species are then released by heating into a chromatograph for separation and identification. It is a 'green' method because no additional solvent is used in this process. We have developed a new class of SPME fibers that offer extraordinary capacity and speed. They are prepared by sputtering a material under conditions that lead to a nanoporous coating on the fiber. When silicon is sputtered under these conditions, its outermost surface can be additionally oxidized, leading to a high density of silanol groups than can be subsequently silanized. For example, the fibers can be derivatized with octadecyldimethylmethoxysilane by chemical vapor deposition (CVD), which creates a hydrophobic extraction medium. The performance of our 2 micron sputtered coatings has been compared to that of thicker (7 micron) commercial coatings. Our fiber consistently outperforms the commercial fiber, showing significantly higher capacity for alcohols, amines, aldehydes, and esters. Real world samples, e.g., hops and PAH from water, have also been analyzed. Different coating thicknesses have been prepared and evaluated. Sputtered coatings have been characterized by X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and wetting.

**4:20pm SE+2D+EM-WeA7 Ferroelectric Thin Films for Memory Applications, Joyprakash Chakrabarty, Institut national de la recherche scientifique (INRS), Canada**

Ferroelectric (FE) oxides draw attention in science community because of its spontaneous switchable polarization often used in electronic devices. FEs are earth abundant, easy to synthesis and low degradation of electronic properties while exposed to room atmosphere. One of its promising applications is in computer memory devices. FEs function as memory by storing data in its two polarization states normally defined as up and down state. However the challenges lie in enhancing data bit density at room temperature. Here we show four step ferroelectric polarization switching in BiFeO<sub>3</sub>(BFO)/SrRuO<sub>3</sub>(SRO)/BiMnO<sub>3</sub>(BMO) heterostructure thin films which act as FE memory devices. All crystalline films are grown on (100) oriented

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Niobium doped SrTiO<sub>3</sub> (NSTO) single crystal substrates by pulsed laser deposition. Our experimental results show a promising device concept, unique in FE memories that can enhance the data storage capacity in heterostructure capacitor devices at room temperature.

4:40pm **SE+2D+EM-WeA8 Thermoelectric and Optical Properties of Advanced Thermoelectric Devices from Ni/Bi<sub>2</sub>Te<sub>3</sub>/Ni and Ni/Sb<sub>2</sub>Te<sub>3</sub>/Ni Thin Films**, *Satilmis Budak, Z. Xiao, J. Cole, A. Kassu, D. Price, T. Davis, T. Strong, J. Gray*, Alabama A&M University

Thermoelectric devices were prepared from Ni/Bi<sub>2</sub>Te<sub>3</sub>/Ni and Ni/Sb<sub>2</sub>Te<sub>3</sub>/Ni thin films using DC/RF magnetron sputtering and E-beam deposition systems. Thermoelectric devices were annealed at different temperatures to form nanostructures in the multilayer thin films to increase both the Seebeck coefficients and electrical conductivity and decrease thermal conductivity. The thin film devices were characterized using Seebeck coefficient measurement systems; four probe van der Pauw measurement system to measure resistivity, sheet resistance, density, mobility and type of the charge carrier concentration. In addition to the Seebeck coefficient and van der Pauw measurements, the laser thermal conductivity system was used for the thermal conductivity measurements. The surface morphology of the fabricated thermoelectric films is characterized using Scanning Electron Microscope (SEM). Raman Spectroscopic technique is used for identification of inherent molecular specificity and analysis of chemical compositions of the films. The resonant features of the scattering spectra measured under the 532 nm and 785 nm wavelength excitation lasers are analyzed.

## Acknowledgement

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5:00pm **SE+2D+EM-WeA9 Effects of Composition and Strain on Band Gaps of Pseudomorphic Ge<sub>1-x</sub>Si<sub>x</sub>Sn<sub>y</sub> on Ge**, *Nalin Fernando*, New Mexico State University; *R. Hickey, J. Hart, R. Hazbun, D. Zhang, J. Kolodzey*, University of Delaware; *S. Zollner*, New Mexico State University

Ge-Si-Sn alloys are interesting for CMOS applications for a variety of reasons. For example, adding Sn to Si-Ge lowers the band gap, which reduces parasitic contact resistance. Also, the lattice constant of Ge-Sn alloys increases when adding Sn. Therefore, such alloys could be used to impart strain on PMOS devices with Ge channels. We used deformation potential theory to determine the compositional dependence of the direct, indirect, E<sub>1</sub>, and E<sub>1</sub>+Δ<sub>1</sub> band gaps of pseudomorphic Ge<sub>1-x</sub>Si<sub>x</sub>Sn<sub>y</sub> on Ge and theoretical predictions are validated through spectroscopic ellipsometry measurements of the band gaps of pseudomorphic Ge<sub>1-y</sub>Sn<sub>y</sub> on Ge grown by MBE.

The band structure of Ge is a strong function of strain and alloy composition, and a transition from an indirect to a direct band gap has been observed for γ~6-10% for relaxed Ge<sub>1-y</sub>Sn<sub>y</sub> indicating the possibility of widespread applications of Ge-based photonic devices. The pseudomorphic nature of the Ge-based alloy layer on a substrate is important to keep dislocation densities low at the interface to improve the performance of the device. Band gap engineering of Ge by controlling strain and alloying with Si and Sn has attracted great interest since a Ge<sub>1-x</sub>Si<sub>x</sub>Sn<sub>y</sub> ternary alloy with two compositional degrees of freedom allows decoupling of the lattice constant and electronic structures.

The pseudomorphically grown Ge<sub>1-x</sub>Si<sub>x</sub>Sn<sub>y</sub> layer on Ge experiences a biaxial stress due to the lattice mismatch between the alloy layer and the Ge. The strain resulting from the stress affects the band structure of the alloy. Deformation potential theory is used to determine the compositional dependence of the band gaps of pseudomorphic Ge<sub>1-x</sub>Si<sub>x</sub>Sn<sub>y</sub> on Ge as a function of Si (x) and Sn (y) compositions. The predictions of the deformation potential theory are validated for pseudomorphic Ge<sub>1-y</sub>Sn<sub>y</sub> (for Si=0) on Ge through measurements of the optical properties. The complex pseudodielectric functions of pseudomorphic Ge<sub>1-y</sub>Sn<sub>y</sub> alloys grown on Ge by MBE were measured using ellipsometry in the 0.1-6.6 eV energy range for Sn contents up to 11%, to investigate the compositional dependence of the band gaps. Critical point energies (CP) and related parameters were obtained by analyzing the second derivative spectrum of the dielectric function. Our experimental results for E<sub>g</sub><sup>dir</sup>, E<sub>1</sub> and E<sub>1</sub>+Δ<sub>1</sub> gaps are in good agreement with the theoretically predicted CP energies. We will discuss the strain and compositional dependence of the band gaps and the effects of the growth temperature of the Ge buffer layer on Si to the band gaps.

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This work was supported by AFOSR (FA9550-13-1-00222). FTIR measurements were performed at CINT.

5:20pm **SE+2D+EM-WeA10 MBE Growth of Hexagonal Boron Nitride for use in Novel Electronic Devices**, *Adam Barton, R. Yue, C.M. Smyth, R. Addou, L. Cheng, R.M. Wallace, J. Kim, M. Kim, J. Hsu, K.J. Cho*, The University of Texas at Dallas; *L. Colombo*, Texas Instruments; *C.L. Hinkle*, The University of Texas at Dallas

2D materials offer unique opportunities in device fabrication due to the weak van der Waals interaction between crystalline layers that allows for the growth of high-quality heterostructures with significantly less impact from lattice mismatch with the substrate. Hexagonal boron nitride (hBN) has a honeycomb structure similar to graphene except with alternating boron and nitrogen atoms. The hexagonal rings are composed of six sp<sup>2</sup>-hybridized atoms (three boron atoms and three nitrogen atoms). The electronic structure results in a bandgap of 5-7eV, a low-κ dielectric constant of 2-4ε<sub>0</sub>, and an electron affinity of roughly 2 eV. These electronic properties make hBN an exciting material for a wide range of applications in electronic devices. In particular, we are interested in coupling hBN with transition metal dichalcogenides (TMDs) for low-power tunnel FET applications. Previous publications have primarily utilized chemical vapor deposition (CVD) to grow hBN on catalyzing transition metal substrates (Co, Ni, Cu, etc.) at growth temperatures ranging from 800-1200°C. However, these substrates and growth temperatures are not practical for the majority of device applications. Chalcogen loss in TMDs, for example occurs well below those temperatures.

In this work we report on our recent findings on the growth and characterization of hBN thin films grown by molecular beam epitaxy (MBE). This will include a detailed discussion of the growth mechanism on a variety of substrates (MoS<sub>2</sub>, HOPG, WSe<sub>2</sub>, Bi<sub>2</sub>Se<sub>3</sub>, and sapphire) using substrate growth temperatures ranging from 300-800°C. The impact of the source fluxes, substrate temperatures, and in particular, the presence of atomic hydrogen during growth will be presented. The hexagonal phase of BN is achieved as determined by diffraction, Raman, and XPS. AFM, TEM, and RHEED are also used to assess film quality and the experimentally determined bandgap and band alignment will be presented. We will also present our recent work on coupling hBN with ALD-deposited Al<sub>2</sub>O<sub>3</sub> to enable higher-k gate dielectrics on top of 2D materials heterostructures.

This work is supported in part by the SWAN Center, a SRC center sponsored by the Nanoelectronics Research Initiative and NIST. This work was also supported in part by the Texas Higher Education Coordinating Board's Norman Hackerman Advanced Research Program.

5:40pm **SE+2D+EM-WeA11 Passivation of Interfaces Between High-k Dielectrics and SiGe: Ex Situ Wet Sulfur Dioxide vs. In Situ Plasma Nitridation**, *Kasra Sardashti*, *M.S. Clemons*, UC San Diego; *M. Yakimov*, SUNY College of Nanoscale Science and Engineering; *K. Tang*, Stanford University; *S. Oktyabrsky*, SUNY College of Nanoscale Science and Engineering; *P.C. McIntyre*, Stanford University; *L. Dong, N. Yoshida*, Applied Materials, Inc.; *A.C. Kummel*, UC San Diego

Silicon-Germanium is a promising channel material to be used in novel CMOS device architectures such as FinFET and Nanowire FET, due to its high hole mobility. Moreover, it enables control of carrier mobility by mechanical stress and band gap by variation in Si/Ge content in multilayer design. One of the important issues to address in the integration of SiGe in the MOS devices is formation of low-defect interfaces with very thin high-k gate dielectrics such as Al<sub>2</sub>O<sub>3</sub>, HfO<sub>2</sub> and ZrO<sub>2</sub>. Due to adverse effect of Ge sub-oxide (GeO<sub>x</sub>) formation and subsequent Ge out-diffusion on the performance of the devices, robust strategies to control the Ge reactions during and after oxide deposition are essential to further the development of SiGe FETs. This study determines the effects of two passivation methods on the quality of the interfaces between SiGe and high-k dielectrics: 1) Ex-situ wet sulfur passivation using (NH<sub>4</sub>)<sub>2</sub>S solution; 2) In-situ NH<sub>3</sub> plasma nitridation. Al<sub>2</sub>O<sub>3</sub> and HfO<sub>2</sub> were deposited on SiGe surfaces by atomic layer deposition (ALD). Electrical and chemical properties of the interfaces were evaluated by capacitance-voltage (C-V) spectroscopy, angle-resolved photoelectron spectroscopy (AR-XPS) and time-of-flight secondary ion mass spectroscopy (ToF-SIMS). Both sulfur passivation and plasma nitridation resulted in smaller density of interface traps with a large majority of the trap energy levels adjacent to the valence band edge. Ex-situ sulfur passivation was found to improve the interface quality by reducing the extent of GeO<sub>x</sub> formation at the high-k/SiGe interface, therefore constraining the extent of Ge out-diffusion within the oxide. The

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mechanism is distinct; sulfur forms much stronger bonds to Ge than to Si due to the d-orbitals on the Ge; this promotes selective oxidation of the Si and prevents formation of GeOx. Using plasma nitridation, a thin layer of SiON forms selectively at the high-k/SiGe interfaces, preventing the presence of thermally-unstable species such as GeOx and GeON. In addition, SiON can act as a physical diffusion barrier to Ge out-diffusion. The nitridation is selective due to the higher enthalpy of formation for Si vs Ge oxynitrides. Both ex-situ sulfur passivation and in-situ NH<sub>3</sub> plasma nitridation were found to be effective approaches in preparing low-defect Al<sub>2</sub>O<sub>3</sub>/SiGe and HfO<sub>2</sub>/SiGe interfaces; the different chemical mechanisms show there are multiple paths to selective bond formation to SiGe which can be utilized to engineer low-defect stable interfaces.

6:00pm **SE+2D+EM-WeA12 Enhanced Voltage Control of Perpendicular Magnetic Anisotropy in Magnetic Tunnel Junctions Using Ultrathin PZT Composite Oxide Tunneling Barriers**, *Kevin Fitzell, X. Li, K. Wong, G. Yu, S. Robbennolt, S.H. Tolbert, P.K. Amiri, K.L. Wang, J.P. Chang*, University of California at Los Angeles

In contrast to manipulating magnetization with applied current, using an applied electric field can significantly reduce the required energy and result in less heat generation, leading to increased energy density. This can be accomplished using the voltage-controlled magnetic anisotropy (VCMA) effect, which forms the basis of next-generation magnetoelectric MRAM devices. Specifically, applying an electric field across a CoFeB/MgO interface can decrease the perpendicular magnetic anisotropy field as a result of the altered electron density at the interface, thus destabilizing the magnetization state and allowing for its efficient and deterministic reorientation with a small applied magnetic field. This operation principle stands in contrast to that of STT-RAM, which uses upwards of 100 fJ to write a single bit (300,000 times more energy than the actual energy barrier to switching).

Previous research on CoFeB/oxide interfaces has shown that increasing the dielectric constant of the oxide layer also increases the sensitivity of the interfacial magnetic anisotropy energy to an applied electric field. Lead zirconate titanate (PZT), having excellent ferroelectric properties including a large dielectric constant, is therefore a prime candidate for integration into such oxide layers in an attempt to maximize the VCMA effect. Using atomic layer deposition (ALD), PZT was thus incorporated into magnetic tunnel junctions having MgO/PZT/MgO multilayer composite tunneling barriers. Our group has shown that MRAM devices fabricated using these MgO/PZT/MgO tunneling barriers resulted in a 40% increase in the VCMA coefficient (and thus substantial energy savings) over magnetic tunnel junctions simply employing MgO tunneling barriers, despite the PZT layer being amorphous. Our most recent work has focused on the crystallization of these ultrathin PZT layers, which would cause an even larger dielectric response (even greater energy savings) and open an avenue toward four-state memory devices relying on the ferroelectric polarization of the PZT.

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