

Tuesday Morning, November 11, 2014

Nanometer-scale Science and Technology

Room: 304 - Session NS+HI-TuM

Nanopatterning and Nanolithography

Moderator: Nancy Burnham, Worcester Polytechnic Institute, Leonidas Ocola, Argonne National Laboratory

8:00am **NS+HI-TuM1 Nanoetching and Characterization Towards sub-5 nm Patterning**, *Deirdre Olynick, D. Staaks, D. Tierno, S. Dallarto, S. Sassolini, B. Muddiman, Z. Lui, G. Calafiore*, Lawrence Berkeley National Laboratory, *X. Gu, T.P. Russell*, University of Massachusetts, Amherst, *M. Kocsis*, Inpria Corporation

INVITED

Plasma etching is the ubiquitous method for high-resolution pattern transfer in semiconductor and related technologies. As lithographic techniques advance towards 5 nm half-pitch for applications in storage media, nanoelectronics, and plasmonic based devices, plasma etching processes must follow suit. This brings enormous and arguably insurmountable challenges using typical plasma hardware. For instance, very high etching selectivity must be achieved to accommodate mask heights (~1-2 times the feature size) which must shrink to mitigate pattern collapse in the lithographic and etching steps. In addition, line edge roughness at down to sub 1 nm levels must be achieved. To meet these enormous challenges we are investigating etching processes with temperatures down to -140 °C. Low temperature etching was first introduced by Tachi.¹ Lower etching temperatures can bring benefits such as higher selectivity processes, larger process windows, and reduced plasma damage which will be important for achieving sub-5 nm features.

We will discuss nanoscale cryogenic etching work in silicon, chromium, and silicon dioxide. With careful micron and deep nanoscale etching we show that cryogenic temperature etching of silicon, previously studied in great detail at the micron scale,^{2,3} can provide extreme selectivity and anisotropy at the nanoscale even with soft masks derived from block copolymer lithography. Selectivity is enhanced while maintaining pattern verticality because resist etch rates decrease as temperature is lowered. Changing to a chromium hardmask increases selectivity towards deeper sub-10 nm features at 20:1 aspect ratios. Studies of chromium etching show a temperature dependent etch rate that can be used to enhance profile control and limit mask undercut, necessary when nanometer controlled is required. Finally, we will discuss investigations into reduced temperature silicon dioxide etching for applications in patterned media and vertical NAND.

References:

1. S. Tachi, K. Tsujimoto and S. Okudaira, *Appl. Phys. Lett.* (8), 616-618 (1988).
2. X. Mellhaoui, R. Dussart, T. Tillocher, P. Lefaucheux, P. Ranson, M. Boufnichel and L. J. Overzet, *J. Appl. Phys.* (10), 104901-104910 (2005).
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8:40am **NS+HI-TuM3 Cut Patterning Challenges for the 14nm-Node and Beyond**, *Ryan Jung, J.R. Sporre, F.L. Lie, S. Kanakasabapathy, S. Sieg*, IBM Albany Nanotech Center, *A. Ranjan, S. Voronin, A. Raley, V. Rastogi, A. Ko*, TEL Technology Center, America, LLC, *D. Lee*, Samsung Electronics

In order to satisfy certain device architecture, fabrication of certain levels such as channel and gate is typically done by first forming line and space arrays, followed by removing or cutting some lines or parts of lines to form the final pattern. For instance, the method of Sidewall Image Transfer (SIT) patterning generates pairs of lines that are structurally connected at the line ends. Accordingly, the line/space patterning must be supplemented with a companion cut mask pattern to remove these undesired features. The cut mask, in addition to removing undesired features, also facilitates orthogonal line end control and dense array tip-to-tip control, such as in memory device, that cannot be achieved solely from lithography side using mask optical proximity control (OPC) and negative tone developed resist. With channel and gate pitch being scaled down to below 80nm, the ability to precisely place the cut mask edge and to control the line end taper angle has

a direct impact on defectivity and yield. The ability to control the critical dimension of the cut opening has a direct impact on the tip-to-tip CD and device density. This paper evaluates the advantages, technical challenges, and extendibility of various cut schemes for 14nm node and beyond, focusing on line edge profile and tip-to-tip control. This work was performed by the Research and Development Alliance Teams at various IBM Research and Development Facilities

9:00am **NS+HI-TuM4 Nanopore Memristors: Sub-10nm Devices Built on Membranes Milled with a Helium Ion Microscope**, *Douglas Ohlberg, J.P. Strachan, W. Thompson, Z.Y. Li, R.S. Williams*, Hewlett Packard

A novel platform has been developed to fabricate and study the performance of memristive devices smaller than 10 nm. The platform consists of free-standing silicon nitride membranes into which holes or pores with diameters ranging in size from 7nm – 50 nm have been milled with a helium ion microscope. In addition to serving as a substrate, the membrane also acts as an interlayer dielectric, and devices are fabricated by sequential deposition of materials above and below the membrane to fill the nanopore. Since deposition of the layers does not require intervening exposures to resists, developer solutions, or plasma cleans, all interfaces are clean and free of contaminants that would otherwise degrade device operation. This approach enables the compositional engineering of ideal device stacks at the nanometer scale decoupled from the problems often introduced by conventional lithographies. Working memristors are demonstrated that have been successfully fabricated around nanopores with diameters as small as 7nm. In addition, we demonstrate, how the sidewall profiles of the nanopores can be engineered in a variety of shapes from conical to hour glass by the deposition, prior to helium ion milling, of materials on the membrane that influence the scattering dynamics of the ions during milling.

9:20am **NS+HI-TuM5 Characterization of Cluster-Based High-Resolution Inorganic Resists**, *Rose Ruther, R.P. Oleksak, R. Frederick, B.T. Flynn, G.S. Herman*, Oregon State University

Both near- and long- term challenges for nanomanufacturing require significant advances in lithography to obtain sub-ten nanometer half-pitch. One approach to meet these challenges is through inorganic resists based on clusters and nanoparticles. Inorganic resists are of considerable interest due to the potential for both high resolution and low line width roughness (LWR), but generally suffer from low sensitivity. Recently, incorporation of radiation sensitive ligands into inorganic resists has enabled significantly improved sensitivity. For example, we have demonstrated the use of H₂O₂ as a radiation sensitive ligand for resists based on inorganic nano-clusters with the general formula, Hf(OH)_{4-2x-2y}(O₂)_x(SO₄)_y·qH₂O (HafSOx). By including H₂O₂ the HafSOx has significantly improved sensitivity to extreme UV photons and electrons, while still displaying high-resolution and low LWR.

In this presentation we characterize key steps in the lithographic process to gain insight into the nanodimensional patterning of HafSOx. Dynamic light scattering (DLS) and transmission electron microscopy (TEM) confirm the presence of nanoscale particles in the precursor solutions. TEM is further used to characterize cross-sections of spin-coated HafSOx films before and after pattern exposure and development. Combined with energy dispersive X-ray spectroscopy (EDS) this allows for *in situ* investigations of the dynamic nature of both structural and compositional properties with electron exposure pertinent to the patterning process. In particular, oxygen species are found to be very mobile during TEM analysis and migrate to the Si interface with an associated densification of the HafSOx film. Cross-sectional TEM of patterned lines down to approximately 10 nm half-pitch provides unique information on pattern profiles and reveals the presence of inter-line residual material consisting of discrete structures consistent with solution species. Both temperature programmed desorption (TPD) and electron stimulated desorption (ESD) are used to characterize the key desorption species that occur during thermal and radiative processes during patterning. ESD indicates that the peroxy species have radiation sensitivity, where the primary desorption products are O₂ and H₂O. We find that the time evolution of the O₂ and H₂O desorption yields indicate much faster kinetics for O₂ desorption, suggesting that the formation of the insoluble oxide network is driven initially by desorption of peroxide groups as opposed to thermal dehydration. These data provide insight into the radiation-induced changes responsible for the contrast mechanism of this system.

9:40am **NS+HI-TuM6 Development Characteristics of PMMA in Alternative Alcohol:Water Mixtures**, *Leonidas Ocola*, Argonne National Laboratory

In the past decade there has been a shift from toxic solvents used in lithography processing towards more environmentally safer chemicals. The most widely used resist in electron beam lithography is polymethylmethacrylate (PMMA). The standard developer is a solution mixture of isopropanol (IPA) and methyl isobutyl ketone (MIBK) in a ratio of 3:1. The Globally Harmonized System (GHS) Classification for MIBK includes the following entries: Flammable liquids (category 2), Acute toxicity, Oral (Category 5) and Inhalation (Category 4). The most popular environmentally friendly alternative is an IPA and water (H₂O) solution in a ratio of 7:3. Excellent results have been published using this developer. The mechanism of why this solution works, given the fact that pure IPA and pure H₂O do not develop exposed PMMA is not well understood. Furthermore, the IPA GHS Classification the following entry: Specific target organ toxicity - single exposure (Category 3), central nervous system. Our research is focusing on shedding light onto what would be the interaction of water with similar alcohols, such as methanol and ethanol, and environmentally safer alternatives to IPA. This turns out to be Ethanol. The only Ethanol GHS Classification entry is: Flammable liquids (Category 2). We find that ethanol water mixtures exhibit excellent contrast, sensitivity, and resolution, and should be considered as one of the most environmentally safe viable developer solution for PMMA. We will present results pertaining on our best lithography results using Ethanol:water solutions, and a possible explanation on the role of the water:alcohol interaction with the exposed PMMA resist. We do not believe it is just an issue of cosolvency but more of a localized molecular interaction. The goal of better understanding of this interaction is to help find safer developers for other resists that rely on solvent based development.

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

11:00am **NS+HI-TuM10 Room Temperature Electron Beam Assisted Oxygen Purification of Electron Beam Induced Pt Deposits: Towards Pure and High-Fidelity Nanostructures**, *Brett Lewis*, *M.G. Stanford*, University of Tennessee, *H. Plank*, Graz University of Technology, Austria, *J.H. Noh*, University of Tennessee, *J. Fowlkes*, Oak Ridge National Laboratory, *P.D. Rack*, University of Tennessee

Electron Beam Induced Deposition (EBID) is a direct write mode capable of fabricating highly precise nanoscale structures by employing a scanning electron beam to disassociate adsorbed precursor molecules which subsequently condense on a substrate. The major drawback of the EBID process is that high purity metallic deposition is rarely achieved due to residual impurities attributed to the inadequate disassociation of the precursor molecule remaining in the final structure. Thus, purification strategies for nanoscale EBID deposits has been a critically important research area as EBID is poised to impact many nanoscale science and technology applications. To this end, our recent work has been focused on the post-deposition purification of EBID structures. We demonstrate a room temperature purification method in which platinum-carbon nanostructures deposited from MeCpPtIVMe₃ are purified by the presence of oxygen gas during a post-electron exposure treatment. Deposit thickness, oxygen pressure and oxygen temperature studies suggest that the dominant mechanism is the electron stimulated reaction of oxygen molecules adsorbed at the defective deposit surface. In this presentation we will overview the electron-stimulated reaction regimes as a function of oxygen partial pressure and temperature, and electron beam current and energy. We will overview electron stimulated reaction and adsorption/diffusive transport models to demonstrate that, for our experimental regime, we believe the rate-limiting mechanism is oxygen adsorption/transport. In addition to purification, the post-deposition electron stimulated oxygen purification process enhances the resolution of the EBID process due to the carbon removal of the as-deposited materials. Notably, pure platinum deposits with low resistivity and retain the original deposit fidelity were accomplished at room temperature.

11:20am **NS+HI-TuM11 Prospects for Nanofabrication using the Combination of STM-based Depassivation Lithography, Selective ALD, and Material Etch Processes**, *Joshua Ballard*, Zyvex Labs, *S. Anz*, *S. Sando*, Systine, Inc., *M. Bischof*, University of North Texas, *D. Dick*, University of Texas at Dallas, *J. Fu*, National Institute of Standards and Technology (NIST), *D. Jaeger*, University of North Texas, *R. Longo*, University of Texas at Dallas, *J. Owen*, *E. Fuchs*, Zyvex Labs, *S. McDonnell*, University of Texas at Dallas, *R. Reidy*, University of North Texas, *Y.J. Chabal*, *R.M. Wallace*, University of Texas at Dallas, *J. Randall*, Zyvex Labs, *A. Cherala*, *S. Singhal*, *S. Sreenivasan*, University of Texas at Austin

Attaining the capability to produce top-down designed nanostructures at sizes and precisions at the nanometer and atomic scales will enable new classes of research into material and device behavior. First demonstrated in the mid-1990s, Scanning Tunneling Microscopy (STM) based hydrogen depassivation lithography has been shown to allow selective functionalization of surfaces with many types of molecules and materials with near absolute precision. Recently, selective Atomic Layer Deposition (ALD) of titania has also been demonstrated on such a functionalized surface, with the deposited material behaving as an etch mask in 3-D nanostructure formation. This results in a process that combines the high precision of STM with standard processing techniques to produce 3-D structures.

Already, 3-D structures with arbitrary shapes with full-pitches down to 13 nm have been fabricated using this process. This work will describe this process as well as provide an overview of the problems that need to be addressed to further reduce the minimum feature size and improve precision. Given the current and near term limitations of the process, classes of devices that have been and are possible to be fabricated will be described, including designed quantum dots, photonic structures, and NEMS apparatuses. Finally, pathways for scalability will be discussed.

Tuesday Afternoon, November 11, 2014

2D Materials Focus Topic

Room: 310 - Session 2D+AS+HI+MC+NS+PS+SP+SS-TuA

2D Materials Characterization including Microscopy and Spectroscopy

Moderator: Manish Chhowalla, Rutgers University

2:20pm **2D+AS+HI+MC+NS+PS+SP+SS-TuA1 Layer-Dependent Electronic and Physical Structure of 2D van der Waals Crystals, Richard Osgood, Columbia University** **INVITED**

Because of their weak Van der Waals interlayer bonding transition-metal dichalcogenide (TMDC) semiconductors can be fabricated into atomically thin two-dimensional (2D) crystals with substantial ~ 1-2 eV bandgaps. As one example, monolayer MoS₂ consists of a single layer of Mo atoms sandwiched between two layers of S atoms in a trigonal prismatic structure. The TMDC 2D system has attracted great interest because of its distinctive electronic and optical properties, such as (i) a transition from indirect-to-direct band gap in going from the multilayer to monolayer crystal due to a missing interlayer interaction in monolayer form and (ii) strong spin-orbit-coupling-induced split valence bands, i.e. 100's of meV, due to broken inversion symmetry, which makes TMDCs interesting for spin-physics physics and devices. Both properties have been predicted with density functional theory (DFT) calculations and indirectly demonstrated using photoluminescence and Raman spectroscopy.

Recently we have made a series of direct observations of the thickness-dependent electronic-band and crystal structure of TMDCs of both exfoliated and CVD grown sample. Because of the relatively modest sample sizes we have used micrometer-scale, angle-resolved photo-emission spectroscopy (micro-ARPES) of both the exfoliated and chemical-vapor-deposition-grown crystals; these measurements provide direct evidence for the shifting of the valence band maximum from gamma bar (Brillouin zone center) to kappa bar (Brillouin zone corner), as the sample thickness decreases from bulk to monolayer. Our initial results were with MoS₂ and are described in a preliminary way in Refs 1 and 2. Our TMDC experimental results are compared with rigorous DFT calculations of both the bands and the UV transitions matrix elements. The results show an evolution in band structure, which is consistent with an indirect-to-direct bandgap transition in going from few-layer to monolayer TMDC and can be attributed to changes in quantum confinement as the number of layer decreases. Our microARPES and, subsequently, higher resolution nanospectroscopy data provide clear measurements of the hole effective mass, the strain present in the monolayer crystal films, and the valence-band spin-orbit splitting. Our results explain the low hole mobility of monolayer MoS₂ compared to thicker MoS₂ and show clearly the strong orbit split energies. Our results, using nanoLEED and LEEM also provide insight into the structure and defects in monolayer films. Experiments using K-doping of single-crystal samples and resulting level shifts are also described.

1. W. Jin, P.-C. Yeh, N. Zaki, D. Zhang, J. T. Sadowski, A. Al-Mahboob, A. M. van der Zande, D.J.A. Chenet, J. I. Dadap, I. P. Herman, P. Sutter, J. Hone, R. M. Osgood, Jr., "Direct Measurement of the Thickness-Dependent Electronic Band Structure of MoS₂ Using Angle-Resolved Photoemission Spectroscopy." *Phys. Rev. Lett.* **111**, 106801 (2013)

2. Po-Chun Yeh, Wencan Jin, Nader Zaki, Datong Zhang, Jerzy T. Sadowski, Abdullah Al-Mahboob, Arend M. van der Zande, Daniel A. Chenet, Jerry I. Dadap, Irving P. Herman, Peter Sutter, James Hone, and Richard M. Osgood, Jr., "Probing substrate-dependent long-range surface structure of single-layer and multilayer MoS₂ by low-energy electron microscopy and microprobe diffraction," *Phys. Rev. B* **89**, 155408 (2014)

3:00pm **2D+AS+HI+MC+NS+PS+SP+SS-TuA3 X-ray Photoemission and Electron Energy Loss Spectroscopy Investigation of the Band Gap and Band Alignment for h-BN and MoS₂ Materials and Interfaces, Benjamin French, J. Brockman, M. French, M. Kuhn, J.D. Bielefeld, S.W. King, Intel Corporation, E. Bersker, G. Bersuker, SEMATECH, J. DiStefano, Y.C. Lin, J.A. Robinson, Penn State University**

Hexagonal boron nitride (h-BN) and molybdenum disulfide (MoS₂) are two dimensional (2D) materials of significant interest for future nano-electronic devices. Due to a wide band gap (~ 6 eV), close lattice matching (< 2%) and atomic planarity, hexagonal boron nitride (h-BN) is of primary interest as a potential substrate and gate dielectric in graphene channel transistor devices. In contrast, MoS₂ is a 2D semiconducting material with a band gap of ~ 1.8 eV that is attractive as a possible complement or alternative to

graphene for nano-electronic devices requiring a large band gap. A key property for the success of both h-BN and MoS₂ in such devices is the interfacial band alignment with graphene, the gate contact metallization and the surrounding insulating dielectric materials. In this regard, we have utilized x-ray photoelectron spectroscopy (XPS) to determine the Schottky barrier and valence band offsets present at the interfaces between plasma enhanced chemically vapor deposited amorphous h-BN:H and chemically vapor deposited MoS₂. In combination, we have utilized reflection electron energy loss spectroscopy (REELS) to investigate the band gap of both h-BN and MoS₂ materials to deduce the conduction band alignment. We show that in many instances the valence and conduction band offsets are significant and favorable for MoS₂/h-BN transistor devices.

3:20pm **2D+AS+HI+MC+NS+PS+SP+SS-TuA4 STM/STS Characterization of MoS₂ Monolayers and Nanostructures, A. Mills, C. Chen, Virginia Tech, Y. Yu, L. Cao, North Carolina State University, Chenggang Tao, Virginia Tech**

Atomically thin molybdenum disulfide (MoS₂) and nanostructures have been the subject of intense research efforts for their fascinating properties and potential applications in future electronic and optical devices. Especially, monolayer MoS₂, an atomically thin semiconductor with a direct band gap, as opposed to an indirect band gap in bulk MoS₂, has been demonstrated as field effect transistors, optoelectronic devices and chemical sensors. In our experimental study, Monolayer MoS₂ and MoS₂ triangular nanostructures are synthesized through a self-limiting chemical vapor deposition (CVD) approach. The precursor materials, MoCl₅ and sulfur, react at high temperatures to produce MoS₂ species and subsequently precipitate onto substrates to yield MoS₂ films and triangular nanostructures. Using scanning tunneling microscopy (STM), we have investigated the structural and electronic properties of monolayer MoS₂ grown on glassy carbon and triangular MoS₂ nanostructures on highly ordered pyrolytic graphite (HOPG). We will also discuss our scanning tunneling spectroscopy (STS) measurements on these structures.

4:40pm **2D+AS+HI+MC+NS+PS+SP+SS-TuA8 Surface Characterization of Metal Oxide Layers Grown on CVD Graphene and Spin Precession Measurements, Akitomo Matsubayashi, University at Albany-SUNY, W. Nolting, University at Albany-SUNY, D. Sinha, University at Albany-SUNY, A. Jayanthinarasimham, J.U. Lee, University at Albany-SUNY, V.P. LaBella, University at Albany-SUNY**

Ultra thin metal oxide films grown on graphene can be utilized as dielectric barriers between metals and graphene to help isolate a metal contact from the graphene channel for device applications. This is particularly important for graphene based spintronic devices as tunnel barriers between the ferromagnetic metal as a spin injector and graphene have been known to increase the spin relaxation time measured utilizing non-local detection technique of spin precession by avoiding the conductivity mismatch problem. However, simply depositing metal oxide layers such as aluminum oxide on graphene results in non-uniform film lowering the quality of the interface barrier. We will present a systematic study of aluminum oxide layers grown on CVD graphene under ultra-high vacuum conditions with and without titanium seed layers. The aluminum oxide layers with the 0.2 nm titanium seed layers showed reduced surface roughness. The chemical and structural composition determined by XPS will be also presented that shows full oxidation of the aluminum and partial oxidation of the titanium. The I-V characteristic study performed to electrically evaluate the metal oxide and the preliminary results of non-local spin precession measurements will be also addressed.

5:00pm **2D+AS+HI+MC+NS+PS+SP+SS-TuA9 Morphology of CVD-grown Hexagonal Boron Nitride on Cu Foils, Karthik Sridhara, W.G. Cullen, University of Maryland, College Park, J.K. Hite, Naval Research Laboratory, M.S. Fuhrer, Monash University, Australia, D.K. Gaskill, B.N. Feigelson, Naval Research Laboratory**

Hexagonal boron nitride (h-BN) has grown into prominence as a dielectric for graphene heterostructures. h-BN and graphene have been grown using chemical vapor deposition on various transition metal substrates. Compared to graphene, the morphology of CVD-grown h-BN on Cu has not been as widely studied. Here, we present a systematic study of the morphology of hexagonal boron nitride (h-BN) grown on polycrystalline Cu foils by chemical vapor deposition. The growth of h-BN is performed at ~1000°C in atmospheric pressure CVD with Ammonia Borane (H₃NBH₃) as the precursor. The copper foils, used as catalytic substrates, are thermally annealed at ~1030°C for >5 hours prior to growth and cooled slowly following growth termination. We utilized Ultra-high vacuum Scanning Tunneling Microscopy (STM), ambient AFM and SEM to assess the morphology of the CVD grown h-BN films. Highly symmetric single

crystallites of h-BN are observed for sub-monolayer growth, in agreement with recent reports. We consistently observe a corrugated topographic structure within the h-BN crystallites which is distinctly different from the surrounding copper surface, and this is consistently seen in STM, AFM, and high-resolution SEM. Our aim is to understand the nature of this difference and whether it might be due to effects of differential thermal contraction between h-BN and copper. However, complications arise due to possible changes in the copper substrate topography post-growth due to surface oxidation of the copper. Preliminary results with lateral force microscopy (LFM, frictional mode) show that these corrugations are unidirectional in a single Cu grain irrespective of the orientation of the h-BN crystal and generate frictional forces 200% greater than on the surrounding copper surface, reminiscent of earlier reports of unique frictional behavior in atomically-thin membranes [1]. STM and AFM are also used to study the twin crystal boundaries of h-BN. Preliminary STM observations indicate that merging h-BN crystals consistently have a gap of about 5 nm between them. The results of this study are independent of small variations of growth conditions.

References:

[1] C. Lee, Q. Li, W. Kalb, X.-Z. Liu, H. Berger, R. Carpick, and J. Hone, "Frictional characteristics of atomically thin sheets," *Science* (New York, N.Y.), vol. 328, no. 5974, pp. 76–80, 01-Apr-2010.

5:20pm **2D+AS+HI+MC+NS+PS+SP+SS-TuA10 Influence of Chemisorbed Oxygen on the Growth of Graphene on Cu(100) and Cu(111) by Chemical Vapor Deposition, EngWen Ong**, University at Albany-SUNY, Z.R. Robinson, U.S. Naval Research Laboratory, T.R. Mowll, P. Tyagi, University at Albany-SUNY, H. Geisler, SUNY College at Oneonta, C.A. Ventrice, Jr., University at Albany-SUNY

The influence of chemisorbed oxygen on the growth of graphene by catalytic decomposition of ethylene in an ultra-high vacuum (UHV) chamber on both the Cu(100) and Cu(111) surfaces has been studied. A custom UHV compatible heater stage was constructed that allows heating of a crystal to temperatures as high as 1000 °C at hydrocarbon pressures of up to 100 mTorr. System recovery to the low 10^{-10} Torr range is achieved within a few minutes of opening the gate valve to the turbo pump. The crystal structure of the graphene films was characterized with in-situ low energy electron diffraction (LEED), and the growth morphology was monitored by ex-situ scanning electron microscopy (SEM). For the clean Cu(100) substrate, heating from room temperature to the growth temperature while dosing with ethylene resulted in the formation of epitaxial graphene films. The crystal quality was found to depend strongly on the growth temperature. At 900 °C, well-ordered two-domain graphene films were formed. For the Cu(111) surface, heating from room temperature to the growth temperature while dosing with ethylene did not result in the formation of graphene. This is attributed to the lower catalytic activity of the (111) surface and the relatively high vapor pressure of the Cu surface. The use of an Ar overpressure to suppress Cu sublimation during the growth resulted in the formation of predominately single-domain epitaxial graphene films. Predosing either the Cu(100) or Cu(111) surface with a chemisorbed layer of oxygen before graphene growth was found to adversely affect the crystal quality of the graphene overlayers by inducing a much higher degree of rotational disorder of the graphene grains with respect to the substrate. The SEM analysis revealed that the nucleation rate of the graphene islands dropped by an order of magnitude after predosing either the Cu(100) or Cu(111) surface with a chemisorbed oxygen layer before growth. On the other hand, the average area of each graphene island was observed to increase by at least an order of magnitude. Therefore, the presence of oxygen during graphene growth affects both the relative orientation and average size of grains within the films grown on both substrates.

5:40pm **2D+AS+HI+MC+NS+PS+SP+SS-TuA11 Novel Materials Properties at Atomically Thin Limit, Zhi-Xun Shen**, Stanford University
INVITED

In this talk, I will discuss recent progresses in uncovering novel materials properties at ultra-thin limit, with focus on mono-unit-cell superconductor FeSe and semiconductor MoSe2 respectively.

The observation of a large superconducting-like energy gap which opens at temperatures up to 65 K in single unit cell (1UC) thick iron selenide films on SrTiO3(FeSe/STO) has generated tremendous interest. A challenge is to understand the cause of enhanced Cooper pairing strength in this system, and possibly increase superconducting Tc. In this talk, we show angle-resolved photoemission spectroscopy, mutual inductance, and other measurements on 1UC and multi-UC thick FeSe films grown on Nb-doped SrTiO3. Our data provide clear evidence for strong cross-interface electron-phonon coupling in single UC, raising the possibility that large pairing gap are caused by the strong coupling between the FeSe electrons and certain

collective modes of SrTiO3. This suggests a pathway of "integrated functional components" approach to boost superconducting properties.

The intense interest of quantum systems in confined geometries is further amplified by the recent discovery of large enhancement in photoluminescence quantum efficiency and a potential route to "valleytronics" in atomically thin layered transition metal dichalcogenides (TMDs) MX2 (M = Mo, W; X = S, Se, Te), which are closely related to the indirect to direct band gap transition in the single layer limit. Using angle-resolved photoemission spectroscopy (ARPES) on high quality thin film samples of MoSe2 grown by molecular beam epitaxy (MBE), we have made a direct observation of a distinct transition from indirect to direct band gap as the thickness of the sample is reduced to a monolayer. The experimental band structure indicates a stronger tendency of monolayer MoSe2 towards direct band gap with larger gap size than theoretical prediction. A comparison of directly measured ARPES band gap and optical data led to important new insights on semiconductor physics in 2D. Moreover, our finding of a significant spin-splitting of ~180meV at the valence band maximum (VBM) of a monolayer MoSe2 film could greatly expand its possible application in spintronic devices.

If time permits, I will also discuss the superconductivity in CaC6 and its implication on a possible pathway for superconducting graphene.

2D Materials Focus Topic

Room: 310 - Session 2D+AS+HI+NS+SS-ThM

Nanostructures including 2D Heterostructures, Patterning of 2D Materials

Moderator: Kirill Bolotin, Vanderbilt University

8:00am 2D+AS+HI+NS+SS-ThM1 **Stitching and Stacking for Atomically Thin Circuitry**, Jiwoong Park, Cornell University **INVITED**

The development of large scale growth methods based on chemical vapor deposition (CVD) has enabled production of single-atom-thick films with diverse electrical properties, including graphene (conductor), h-BN (insulator), and MoS₂ (semiconductor). Precise vertical stacking and lateral stitching of these 2D materials will provide rational means for building ultrathin heterostructures with complex functionality. However, large scale production and control of these structures requires new characterization and fabrication approaches. In this talk, I will first discuss the structure and physical properties unique to CVD graphene in single and bilayers. Using the atomic-resolution imaging as well as a dark-field transmission electron microscopy (TEM) technique, our group investigated the structure of grain boundaries in CVD graphene and its impact on the mechanical, electrical, and chemical properties. This allowed us to produce CVD graphene with optimized electrical properties. We also reported a new patterned regrowth method to fabricate 2D lateral heterojunctions entirely made of graphene and h-BN, which enables the development of atomically thin integrated circuitry. If time allows, I will also discuss our recent results on the large scale growth of high quality single layer MoS₂ as well as graphene film with a uniform lattice orientation. Our characterization and growth approach would ultimately allow the fabrication of electrically isolated active and passive elements embedded in continuous, one-atom-thick sheets, which could be manipulated and stacked to form complex devices at the ultimate thickness limit.

8:40am 2D+AS+HI+NS+SS-ThM3 **Vertical and Lateral Heterostructures of Carbon Nanomembranes (CNMs) and Graphene**, Andreas Winter, University of Bielefeld, Germany, M. Woszczyzna, R. Stosch, T. Weimann, F. Ahrelrs, Physikalisches Technische Bundesanstalt, Germany, A. Turchanin, University of Bielefeld, Germany

Heterostructures of graphene with other 2D materials are of great interest for nanoscience and nanotechnology. However, their fabrication is still not a trivial task. Here we present the engineering and characterization of (i) vertical and (ii) lateral heterostructures of molecular thin (~1 nm) dielectric carbon nanomembranes (CNMs) made of aromatic molecules [1] and single-layer (SLG) graphene sheets. (i) The vertical CNM/SLG heterostructures with terminal amino-groups (NH₂-) are assembled via the mechanical transfer onto oxidized silicon wafers. We show by complementary spectroscopy and microscopy techniques as well as by electric transport measurements that functional amino groups are brought into close vicinity of the SLG sheets and that electric transport of the SLG is not impaired by this assembly, leading to the non-destructive chemical functionalization of graphene [2]. (ii) *The lateral heterostructures* are engineered using electron-irradiation-induced crosslinking of SLG sheets with CNMs. We demonstrate reliable production of well-defined laterally patterned CNM-SLG heterostructures of various sized and architectures on solid substrates and as free-standing sheets, and characterize their properties by Raman spectroscopy and helium ion microscopy.

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9:00am 2D+AS+HI+NS+SS-ThM4 **Gate Tunable Carbon Nanotube - Single Layer MoS₂ p-n Heterojunctions**, Deep Jariwala*, V.K. Sangwan, C.-C. Wu, P.L. Prabhumirashi, M.L. Geier, T.J. Marks, L.J. Lauhon, M.C. Hersam, Northwestern University

The isolation of graphene and the subsequent reports on its electronic properties have spurred tremendous interest in a variety of two dimensional (2D) materials for electronic device applications. Layered semiconducting transition metal dichalcogenides (TMDCs) of Mo and W have emerged as

promising alternatives to graphene for optoelectronic applications due to their finite band gap in the visible portion of the electromagnetic spectrum.¹ The atomically thin structure of these 2D materials coupled with van der Waals bonding between adjacent layers allows their stacking into atomically sharp heterostructures with defect-free interfaces, in contrast to epitaxially grown III-V semiconductor heterostructures where the material choices are constrained by lattice matching. Additionally, the few atom thickness of the individual layers enables doping modulation of the overlying layers in a heterostructure using a global back gate. While a large number of heterostructure devices employing graphene have been reported, it's gapless band structure prevents the formation of a large potential barrier for charge separation and current rectification. Consequently, a p-n heterojunction diode derived from ultrathin materials is notably absent and significantly constrains the fabrication of complex electronic and optoelectronic circuits. Here we demonstrate a gate-tunable p-n heterojunction diode using semiconducting single-walled carbon nanotubes (s-SWCNTs) and single-layer molybdenum disulphide (SL-MoS₂) as atomically thin p-type and n-type semiconductors, respectively. The vertical stacking of these two direct band gap semiconductors forms a heterojunction with electrical characteristics that can be tuned with an applied gate bias over a wide range of charge transport behavior, ranging from insulating to rectifying with forward-to-reverse bias current ratios exceeding 10⁴. In addition, the gate-dependent characteristics of this diode exhibit a unique 'anti-ambipolar' behavior with two off-states at either extremes of the gate voltage range and a maximum on-state current between them. This heterojunction diode also responds to optical irradiation with photoresponse time < 15 μs.² We anticipate that the novel properties and characteristics of this p-n heterojunction can be widely generalized to other atomically thin materials systems.

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9:20am 2D+AS+HI+NS+SS-ThM5 **Graphene Transfer onto sub 1nm Al₂O₃/TiOPc/Graphene Gate Stacks**, Ijfo Kwak, J.H. Park, University of California at San Diego, H.C.P. Movva, University of Texas at Austin, E.K. Kinder, H.L. Lu, University of Notre Dame, A.C. Kummel, University of California at San Diego

A novel transfer method with chemically controlled interfacial adhesion is reported for the fabrication of novel logic devices. This method allows direct transfer onto gate stacks and eliminates the possibility of Au electrodes deposition could shorting the thin oxide prior to transfer. The top graphene layer was grown on a Cu layer on a SiO₂/Si substrate by CVD. Au electrodes were deposited on top of the graphene by e-beam evaporation. To transfer the graphene layer, PIB (Polyisobutylene) were drop cast on top of graphene prior to bonding of the Au/graphene/Cu to a PDMS (Polydimethylsiloxane) film. The PIB serves to moderate the adhesion between the PDMS (Polydimethylsiloxane) and the Au electrodes. The PDMS provides mechanical support. Afterwards, the PDMS/PIB/Au/graphene/Cu/SiO₂/Si stack was immersed in ammonium persulfate solution to dissolve the Cu, releasing the top graphene stack. The bottom gate stack was HOPG (highly ordered pyrolytic graphite) with a sub-nano Al₂O₃ film on a monolayer TiOPc(titanyl phthalocynine) film. The monolayer TiOPc was deposited via MBE at 100C and annealed to 250C to insure a monolayer film. The TiOPc acts as a nucleation layer for the oxide ALD. The Al₂O₃ layer was deposited by ALD using TMA (Trimethylaluminum) and H₂O at 100 C. The PDMS/PIB/Au/Graphene stack was placed on the gate stack, and PDMS was removed. Using hexane solution, the rePIB layer was dissolved, leaving clean graphene surface. To measure the oxide characteristics, an AFM was converted into a capacitance meter. This measurement allows non-destructive probing of Au/graphene/Al₂O₃/TiOPc/graphene structure while conventional probe station could damage the oxide or electrodes.

9:40am 2D+AS+HI+NS+SS-ThM6 **Effect of Monolayer Substrates on the Electronic Structure of Single-Layer MoS₂**, Alfredo Ramirez-Torres, D.T. Le, T.S. Rahman, University of Central Florida

We have performed first-principles calculations based on density functional theory (DFT) utilizing the optB88-vdW functional to study structural and electronic properties of a single layer of MoS₂ deposited on single-layer substrates of hexagonal boron nitride (BN), graphene and silicene. All have a honeycomb structure; hence the formation of heterostructures is expected. Since the lattice mismatch between MoS₂ and these substrates is large, we

* NSTD Student Award Finalist

have considered different periodicities among layers to reduce as far as possible the incommensurability between lattices. Our results show that BN barely affects the electronic structure of isolate single-layer MoS₂; the DFT gap remains proximately unchanged. Graphene and silicene severely modify the electronic structure introducing additional states within the optical gap. Adsorption on graphene produces that the system turns like a zero band gap semiconductor bringing the conduction bands of MoS₂ down to the Fermi level of graphene. Adsorption on silicene shifts both valence and conduction bands of MoS₂, towards the Fermi level of silicene, in addition to inducing a gap of about 50 meV in the silicene itself.

This work was partially supported by CONACYT (México) Postdoctoral Fellowship Program (number 204065) and DOE grant DE-FG02-07ER46354

11:00am **2D+AS+HI+NS+SS-ThM10 Ballistic Transport in Epitaxial Graphene Nanoribbons**, *Walt de Heer*, Georgia Institute of Technology
INVITED

Graphene nanoribbons are essential components in future graphene nanoelectronics. However, in typical nanoribbons produced from lithographically patterned exfoliated graphene, the charge carriers travel only about 10 nanometers between scattering events, resulting in minimum sheet resistances of about 1 kW. In contrast 40 nm wide graphene nanoribbons that are epitaxially grown on silicon carbide are single channel room temperature ballistic conductors on greater than 10 μm length scale, similarly to metallic carbon nanotubes. This is equivalent to sheet resistances below 1W surpassing theoretical predictions for perfect graphene by at least an order of magnitude. In neutral graphene ribbons, we show that transport is dominated by two modes. One is ballistic and temperature independent; the other is thermally activated. Transport is protected from back-scattering, possibly reflecting ground state properties of neutral graphene. At room temperature the resistance of both modes abruptly increases nonlinearly with increasing length, one at a length of 16 μm and the other at 160 nm. Besides their importance for fundamental science, since epitaxial graphene nanoribbons are readily produced by the thousands, their room temperature ballistic transport properties can be used in advanced nanoelectronics as well.

11:40am **2D+AS+HI+NS+SS-ThM12 Solution-Synthesized Graphene Nanoribbons**, *Alexander Siniitskii*, University of Nebraska - Lincoln

In this talk I will discuss a recently developed bottom-up approach for gram quantities of narrow graphene nanoribbons that are less than 2 nm wide and have atomically precise armchair edges. These graphene nanoribbons have been characterized by a number of microscopic (STM, AFM, SEM, TEM) and spectroscopic (XPS, UPS/IPES, UV-vis-NIR, IR and Raman spectroscopy) techniques. The properties of graphene nanoribbons could be tuned by incorporation of nitrogen atoms in their edges. Narrow graphene nanoribbons have a large electronic bandgap, which makes them promising for applications in field-effect transistors with high on-off ratios, as well as bulk applications, including coatings, composites and photovoltaic devices.

12:00pm **2D+AS+HI+NS+SS-ThM13 Graphene Silicon Interfaces at the Two-Dimensional Limit**, *Brian Kiraly, A.J. Mannix, M.C. Hersam*, Northwestern University, *N.P. Guisinger*, Argonne National Laboratory

Artificial van der Waals heterostructures have demonstrated both significant improvements of graphene's intrinsic properties and entirely new properties of their own. Early interest in these structures was based on nearly ideal carrier mobility in graphene on two-dimensional (2D) hexagonal boron nitride. Although exfoliation and reassembly of bulk vdW solids has yielded impressive initial results, this method inherently limits the geometry and constituent materials of these structures. Growth of 2D heterostructures has been demonstrated, but mainly limited to the prototypical graphene/hBN system. Adding new constituent materials, particularly those with electronic heterogeneity, to these 2D heterostructures allows them to be engineered with a variety of new properties.

We present the growth and characterization of interfaces between an atomically thin silicon layer and graphene. First, graphene is grown on Ag(111) via atomic carbon deposition at temperatures from 600°C -700°C. Following the growth of graphene, atomic silicon is evaporated on the graphene-covered Ag(111) substrate at 320°C-360°C. The resulting silicon growth results in faceted domains capped with a honeycomb lattice with periodicity 6.4 Å; Raman spectroscopy reveals peaks at 520 cm^{-1} and 900-1000 cm^{-1} that coincide precisely with bulk diamond cubic silicon, indicating these domains are comprised of sp^3 bonded crystalline Si. These 2D sheets of silicon demonstrate both semiconducting character and a honeycomb lattice is attributed to a silver-based reconstruction of the Si(111) surface. The resulting silicon domains grow in two different configurations with respect to the dendritic graphene: (1) silicon domains appear to grow directly on the Ag(111) surface and terminate at the graphene boundaries.

These in-plane interfaces are atomically-precise and clearly resolved via scanning tunneling microscopy. Electronically, the density of states of both isolated constituent materials persist to these interfaces within the resolution of the measurement, indicating little interaction at the border. (2) The silicon growth is observed *underneath* the existing graphene flakes. The vertically stacked silicon graphene domains are identified via atomically resolved imaging *through* the graphene domains at larger biases where graphene is transparent under STM. Furthermore, the vertical materials interfaces demonstrate distinct electronic signatures from either constituent material. The resulting interfaces represent atomically pristine interfaces between graphene and a sp^3 bonded semiconducting Si film, demonstrating a significant step forward in the diversification of van der Waals heterostructures.

Helium Ion Microscopy Focus Topic

Room: 316 - Session HI+2D+AS+BI+MC-ThM

Fundamental Aspects and Imaging with the Ion Microscope

Moderator: Gregor Hlawacek, Helmholtz-Zentrum Dresden - Rossendorf, Stuart Boden, University of Southampton

8:00am **HI+2D+AS+BI+MC-ThM1 He+ and Ne+ Ion Beam Microscopy and Microanalysis**, *David C. Joy*, University of Tennessee, Oak Ridge National Laboratory
INVITED

After one hundred years of use the electron microscope is now being overtaken by ion beam systems because of their many advantages. A wide variety of different ions are available, each of which has its own particular strengths, but the two most commonly used at present are Helium (He⁺) and Neon (Ne⁺). Changing from one to the other takes only a couple of minutes to complete. For operation at beam energies between 20 and 50kV both He⁺ and Ne⁺ generate 'ion induced secondary electrons' (iSE) which yield images which are comparable with those from a conventional SEM but offer image resolutions of 0.4nm or less even on bulk samples, a much greater depth of field, and an enhanced signal to noise ratio. At typical imaging currents between 10-12 to 10-14Amps damage to most samples is very limited for He⁺ although more severe for Ne⁺ but at higher beam currents both He⁺ and Ne⁺ can pattern, deposit, or remove, a wide range of materials. In such applications He⁺ provides the best resolution, but Ne⁺ is much faster.

The production of X-rays depends on the speed of the incident particle, not on its energy. At typical operating energies the He⁺ or Ne⁺ ions are traveling too slowly to generate X-rays so another approach is required for chemical microanalysis. The most promising option is "Time of Flight-Secondary Ion Mass Spectrometry" (TOF-SIMS). Here the incoming ion "splashes" material from the top few layers of the specimen surface. These fragments are then characterized by determining their mass to charge ratios. The chemical data this generates is much more detailed than the bare list of elements that is produced by X-ray microanalysis.

8:40am **HI+2D+AS+BI+MC-ThM3 Gas Field Ion Sources**, *Jason Pitters, R. Urban*, National Institute for Nanotechnology, Canada, *R. Wolkow*, University of Alberta and The National Institute for Nanotechnology, Canada
INVITED

Single atom tips (SATs) prepared by the spatially controlled field assisted etching method are proving to have utility as ion sources, electron sources and in scan probe applications.

As Gas Field Ion Sources (GFISs), there is potential for operation in scanning ion microscopes (SIMs) and our efforts to prepare and characterize SAT ion emission will be discussed. It will be shown that etching to a single atom tip occurs through a symmetric structure and leads to a predictable last atom. SATs can be prepared reproducibly with emission along a fixed direction for all tip rebuilds. It will also be shown that the emission properties of the SAT can be altered by shaping of the tip shank during the etching procedure. In this manner, the operating voltage can be controlled and a lensing effect of the tip base is demonstrated. During formation, the tip shape can be evaluated by using both helium and neon imaging gases. The stability of helium and neon ion beams generated by SATs will also be demonstrated and compared to other tip orientations. The remarkable robustness of these tips to atmosphere exposure will also be shown and the ability to prepare SATs from material other than tungsten will be demonstrated.

SATs also have utility in electron emission. By shaping the tip appropriately, electron emission characteristics can also be tailored and the coherence properties of an SAT will be presented as deduced from holographic measurements in a low-energy electron point source

microscope. Initial utility in scan probe experiments including atomic force microscopy and scanning tunneling microscopy will also be discussed.

9:20am **HI+2D+AS+BI+MC-ThM5 Ion Beam Profiles Generated by W(111) Single Atom Tips**, *Radovan Urban, R. Wolkow*, University of Alberta and The National Institute for Nanotechnology, Canada, *J.L. Pitters*, National Institute for Nanotechnology, Canada

Single atom tips (SATs) gained significant attention over the past decade because they serve as high brightness, field emission electron sources and gas field ion sources (GFISs). Small virtual source size makes these attractive candidates for advanced scanning imaging applications such as SEM, TEM, and scanning ion microscopy (SIM) as well as for non-staining ion beam writing applications.

The ion beam diameter σ , together with total ion current I generated by a single surface atom of W(111) nanotip, are crucial parameters which determine angular current density and brightness of gas field ion sources. It is, therefore, essential to understand underlying mechanisms that govern beam width. Furthermore, mapping both σ and I to a large parameter space of tip temperature, imaging gas pressure, and extraction voltage is necessary to optimize gas field ion source operation. In this contribution we will explore both σ and I as a function of temperature and extraction voltage at different imaging gas pressures using a field ion microscope (FIM) to monitor beam shape and total current. The qualitative model of our results will be also discussed. Finding "the best imaging voltage" for a SAT will be briefly discussed.

9:40am **HI+2D+AS+BI+MC-ThM6 Defect Observation by using Scanning Helium Ion Microscopy**, *Hongxuan Guo, L. Zhang, D. Fujita*, National Institute for Materials Science (NIMS), Japan

Scanning helium ion microscopy (HIM) is an innovative method to characterize surface of various materials. With a secondary electron detector (SED) and a micro plate detector (CPD), Orion Plus system can obtain surface information including morphology, composition, and crystal orientation. [1, 2] Improve the abilities of characterization of materials with HIM will benefit the develop of new materials, such as structure materials including metals, ceramics and others.

In this presentation, we will show the investigation of the crystal structure of metal with HIM. We prepared a sample stage with a reflector that can be used to obtain the transmission helium ions intensities in the samples. With this sample stage, we observed the Ni-Co base super alloy and aerogel composed with hollow nanosphere. The Rutherford backscattered image (RBI) of metal surface show different orientation of poly crystal. The nano-twins and other defects in Ni-Co base superalloy were investigated by HIM in scanning and transmission mode. The nano-twins also be observed by other techniques, such as transmission electron microscopy and electron backscatter diffraction. The scattering of helium ions with different energy was analyzed. This work provide some new methods to improve the research on defects and structure of crystal.

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11:00am **HI+2D+AS+BI+MC-ThM10 Helium Ion Microscopy (HIM) for the Imaging of Biological Samples at Sub-nanometer Resolution**, *James Fitzpatrick*, Salk Institute for Biological Studies **INVITED**

Scanning Electron Microscopy (SEM) has long been the standard in imaging the sub-micrometer surface ultrastructure of both hard and soft materials. In the case of biological samples, it has provided great insights into their physical architecture. However, three of the fundamental challenges in the SEM imaging of soft materials are that of limited imaging resolution at high magnification, charging caused by the insulating properties of most biological samples and the loss of subtle surface features by heavy metal coating. These challenges have recently been overcome with the development of the Helium Ion Microscope (HIM), which boasts advances in charge reduction, minimized sample damage, high surface contrast without the need for metal coating, increased depth of field, and 5 angstrom imaging resolution. We demonstrate the advantages of HIM for imaging biological surfaces as well as compare and contrast the effects of sample preparation techniques and their consequences on sub-nanometer ultrastructure.

11:40am **HI+2D+AS+BI+MC-ThM12 Helium Ion Microscopy of Biological Cells**, *Natalie Frese, A. Beyer, M. Schürmann, B. Kaltschmidt, C. Kaltschmidt, A. Götzhäuser*, University of Bielefeld, Germany

In this presentation HIM images of biological cells are presented. The presented study focuses on neuronal differentiated human inferior turbinate stem cells, mouse neurons and mouse fibroblasts. The cells were prepared

by critical point drying or freeze drying and a flood gun was used to compensate charging, so no conductive coating was necessary.

Therewith, extremely small features at native cell surfaces were imaged with an estimated edge resolution of 1.5 nm. Due to the size of the structures and the preparation methods of the cells the observed features could be an indicator for lipid rafts. This hypothesis will be discussed.

12:00pm **HI+2D+AS+BI+MC-ThM13 Helium Ion Microscopy Analysis of Ag Nanoparticle Implanted Biological Samples for MILDI-MS (Matrix Implanted Laser Desorption/Ionization) Imaging**, *S. Shubeita*, Rutgers University, *L. Muller*, NIDA-IRP, *H.D. Lee, C. Xu*, Rutgers University, *D. Barbacci*, Ionwerks Inc., *K. Baldwin*, NIDA-IRP, *J.A. Schultz*, Ionwerks Inc., *L. Wielunski, Torgny Gustafsson, L.C. Feldman*, Rutgers University, *A.S. Woods*, NIDA-IRP

MILDI mass spectrometry is an emerging tool for detecting changes in brain tissue. An ~20 nm thick region of rat brain tissue implanted with $10^{13}/\text{cm}^2$ $\text{Au}_{(400)}^{4+}$ nanoparticle (NP) ions at 40 keV, produces analytically useful signals of lipids, peptides and proteins using a pulsed nitrogen laser [1]. When a dose of $10^{12}/\text{cm}^2$ 500 eV AgNP (approximately 6 nm diameter) is implanted as a matrix, only lipids are detected [2]. To understand this it is essential to measure the spatial distribution of the nanoparticles. We have used Rutherford Backscattering and Helium Ion Microscopy imaging to determine the Ag NP distributions and areal densities in an implanted coronal rat brain section. We then correlate the ion beam analysis and imaging with individual lipid intensities from several hundred MILDI mass distributions. The results show a high degree of uniformity of the Ag atomic and particulate distribution on a sub-micron scale among different regions of the tissue. Helium Ion Microscopy provides verification of NP matrix uniformity, validating the use of MILDI for quantitative mass analysis.

This work is partially supported by NSF (DMR 1126468), NIH (R44DA030853-03) and IAMDN.

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Thursday Afternoon, November 13, 2014

Helium Ion Microscopy Focus Topic

Room: 316 - Session HI+2D+AS+MC-ThA

Nanoengineering with Helium Ion Beams

Moderator: Armin Götzhäuser, University of Bielefeld, Germany, David C. Joy, University of Tennessee, Oak Ridge National Laboratory

2:20pm **HI+2D+AS+MC-ThA1 Helium Ion Microscopy (HIM) Technology for Imaging, Characterization, and nano-Fabrication for nano-Device Materials and Structures, Shinichi Ogawa, NeRI, AIST, Japan**
INVITED

Several unique applications of a helium ion microscopy (HIM) technology have been studied. In comparison with electron, helium ion has larger cross section, and it realized HIM observation with less current because of higher efficiency of secondary electron generation with maximum distribution energy of 1 eV [1], a few eV in a SEM case, for imaging, which results in less power implant (less thermal damage input) into samples. Utilizing these features, a low dielectric constant material pattern of 70 nm line with less deformation (thermal damage) and a Cu metal line underneath a 130 nm dielectric of band gap of a few eV were imaged [2]. Luminescence from a SiO₂ sample was detected at imaging conditions [3], in which no damage was observed by a transmission electron microscopy (TEM) - electron energy loss spectroscopy method [4]. As one of nano-fabrication applications, we found that a helium ion irradiation using the HIM functionalizes a gate control of carrier conduction in a single-layer graphene at an appropriate amount of helium ion dose to graphene which enable gate bias control of current with an on-off ratio of two orders of magnitude at room temperature [5], [6]. A few nm diameter tungsten particles were deposited onto a TEM sample under the helium ion beam irradiation in W(CO)₆ gas atmosphere with high special resolution accuracy, which realized precise electron tomography and re-construction [7], and tungsten pillars of a few um height with 40 nm diameter were formed with a straight hole of a few nm diameter through a center of the pillars [8]. The research on graphene material is granted by JSPS through FIRST Program initiated by CSTP.

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3:00pm **HI+2D+AS+MC-ThA3 MEMS Temperature Controlled Sample Stage for the Helium Ion Microscope, Jose Portoles, P.J. Cumpson, Newcastle University, UK**

The Helium microscope allows the imaging of samples with magnifications beyond those of electron microscopes with the added advantages of directly imaging insulators without being so critically dependent on a need to conductive coating the samples. This facilitates the imaging of for instance organic structures without the need of surface modification. The large depth of focus allows simultaneously focusing details of the sample at different depths. When using a temperature controlled stage this allows the samples to stay focused as thermal expansion produces vertical displacements of the sample surface, however due to the large magnifications in-plane thermal expansions are still an issue. We have investigated a solution based on a thermally actuated X-Y MEMS stage by exploiting the ability of MEMS actuators to provide smooth electronic control of lateral displacements in the micron range in order to compensate for lateral thermal expansion at the point of observation. The difficulties involved in producing relatively large out of plane displacements with a MEMS device can be neglected due to the large instrumental depth of focus. The device we present has been fabricated using a "silicon on insulator" (SOI) MEMS process, and can be driven at low voltages and currents using a standard vacuum feedthrough to the instrument's analysis chamber and compensate lateral thermal expansion in order to keep any spot on a small specimen in the field of view at high magnifications. The small size of the heating stage makes it rapid in its thermal response.

3:20pm **HI+2D+AS+MC-ThA4 Monte Carlo Simulations of Focused Neon Ion Beam Induced Sputtering of Copper, Rajendra Timilsina, P.D. Rack, The University of Tennessee Knoxville, S. Tan, R.H. Livengood, Intel Corporation**

A Monte Carlo simulation has been developed to model the physical sputtering and nanoscale morphology evolution to emulate nanomachining with the Gas Field Ion Microscope. In this presentation, we will present experimental and simulation results of copper vias milled by a focused neon ion beam. Neon beams with a beam energy of 20 keV and a Gaussian beam profile with full-width-at-half-maximum of 1 nm were simulated to elucidate the nanostructure evolution during the physical sputtering of high aspect ratio features. In this presentation we will overview our simulation attributes which includes an evolving real-time sputtered via profile considering both the sputtered and re-deposited material. The sputter yield and sputter profile vary with the ion species and beam parameters and are related to the distribution of the nuclear energy loss in the material. We will also illustrate how the effective sputter yield is aspect-ratio dependent due to the change in the effective escape angle of the sputtered species. Quantitative information such as the sputtering yields, dose dependent aspect ratios and resolution-limiting effects will be discussed. Furthermore, we will show that the calculated nuclear energy loss and implant concentration ahead of the sputtering front correlates to observed damage revealed by transmission electron microscopy.

4:00pm **HI+2D+AS+MC-ThA6 Circuit Edit Nanomachining Study using Ne+ & He+ Focused Ion Beam, Richard Livengood, S. Tan, Intel Corporation**
INVITED

FIB nanomachining has been used extensively for over 20 years for the purpose of rewiring integrated circuits to validate design changes, isolate process faults, and generate engineering samples. During this time frame, the minimum feature size of an IC (Moore's Law) has scaled from 500nm to 14nm (36X) compared with ~6X scaling of Ga+ FIB. As a result FIB nanomachining capabilities have been steadily eroding over the last several generations, limiting the types of circuit modifications that can be successfully completed. There are however, several promising new ion beam scaling R&D initiatives that provide hope of enabling further nanomachining scaling into the sub 10nm process node.

One such technology is GFIS (gas field ion source) technology. He+ GFIS based FIBs have been successfully used to image with sub 0.5nm resolution and nanomachine sub 10 nm structure in Au, Graphene, and other thin film structures.[1, 2, 3] More recently He+ and Ne+ GFIS sputtering properties have been studied for nanomachining in bulk semiconductor films.[4] In this paper, we will show our latest results on GFIS FIB GAE (gas assisted etch) nanomachining and IBID properties and electrical invasiveness impact.

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4:40pm **HI+2D+AS+MC-ThA8 Evaluation of EUV Resist Performance below 20-nm CD using Helium Ion Lithography, D.J. Maas, TNO Technical Sciences, Netherlands, Nima Kalhor, TU Delft, Netherlands, W. Mulckhuyse, E. van Veldhoven, TNO Technical Sciences, Netherlands, A. van Langen-Suurling, P.F.A. Alkemade, TU Delft, Netherlands, S. Wuister, R. Hoefnagels, C. Verspaget, J. Meessen, T. Fliervoet, ASML, Netherlands**
For the introduction of EUV lithography, development of high performance EUV resists is of key importance. This development involves studies into sensitivity, resolving power and pattern uniformity. We have used a sub-nanometer-sized 30 keV helium ion beam to expose chemically amplified (CAR) EUV resists.

There are remarkable similarities in the response of resists to He⁺ ions and EUV photons. Both primary particle beams traverse the resist and meanwhile interact with the target atoms. The low backscattering of the He⁺ ions results in ultra-low proximity effects, which is similar to EUV exposure. Absorption of an EUV photon creates a high-energy electron that relaxes by the excitation of Secondary Electrons (SEs). A collision of a 20-30 keV helium ion with a target atom directly releases low-energy SEs.

Each ion scatters several times in the resist layer, thus enabling resist exposures at very low doses per CH. The energy spectra of SEs generated by EUV and He⁺ are remarkably alike. These SEs, in turn, activate the resist.

In this paper we show 30 keV He⁺ ions exposures of contact holes and lines with a CD of 8 – 30 nm at 20 nm half-pitch in a chemically amplified EUV resist. We will demonstrate the potential of using He⁺ ion lithography [1,2] in the study of EUV resists.

[1] V. Sidorkin et al., *Sub-10-nm nanolithography with a scanning helium beam*, J. Vac. Sci. Technol. B **27**, L18 (2009)

[2] D. Maas et al., *Evaluation of EUV resist performance below 20nm CD using helium ion lithography*, SPIE Proc. **9048**, 90482Z (2014)

5:00pm **HI+2D+AS+MC-ThA9 Helium Ion Beam Lithography for Nanoscale Patterning**, X. Shi, University of Southampton, UK, D.M. Bagnall, University of New South Wales, UK, **Stuart Boden**, University of Southampton, UK

Electron beam lithography (EBL), the modification of thin films of resist by a focused beam of electrons to create a pattern that is subsequently transferred into the substrate, is a key technology in the development of nanoscale electronic devices. However, with the demand for ever smaller features and pattern dimensions, new lithographic techniques are required to extend beyond existing limits of EBL. One such emerging technology is helium ion beam lithography (HIBL), driven by the development of the helium ion microscope, a tool capable of producing a high intensity beam of helium ions focused to a sub-nanometer spot [1]. Preliminary studies on HIBL using typical EBL resist materials such as PMMA and HSQ have shown that HIBL has several advantages over EBL, including a smaller spot size (potentially leading to higher resolution patterning) and a decrease in the exposure dose required and so the potential for faster pattern definition and therefore higher throughput. Furthermore, proximity effects, which are caused by beam scattering leading to inadvertent exposure of surrounding material, and are problematic when producing high density patterns in EBL, are massively reduced in HIBL [2], [3].

Here, the latest results from an experimental investigation into the HIBL technique will be presented. Areas of PMMA films of various thicknesses are exposed to different helium ion doses. After subsequent development in MIBK/IPA, atomic force microscopy is used to measure residual layer thickness in order to generate exposure response curves for different initial thicknesses of resist. High sensitivity is confirmed with full exposure of 50 nm thick layers achieved with a helium ion dose of only ~2 μC/cm². Experiments to characterise minimum feature size and proximity effects are currently underway. The use of other high resolution resists will also be investigated with the aim of providing a thorough assessment of the capabilities and limitations of this emerging nano-patterning technique.

[1] L. Scipioni, L. A. Stern, J. Notte, S. Sijbrandij, and B. Griffin, "Helium Ion Microscope," *Adv. Mater. Process.*, vol. 166, pp. 27–30, 2008.

[2] D. Winston, B. M. Cord, B. Ming, D. C. Bell, W. F. DiNatale, L. A. Stern, A. E. Vladar, M. T. Postek, M. K. Mondol, J. K. W. Yang, and K. K. Berggren, "Scanning-helium-ion-beam lithography with hydrogen silsesquioxane resist," *J. Vac. Sci. Technol. B.*, vol. 27, no. 6, pp. 2702–2706, 2009.

[3] V. Sidorkin, E. van Veldhoven, E. van der Drift, P. Alkemade, H. Salemink, and D. Maas, "Sub-10-nm nanolithography with a scanning helium beam," *J. Vac. Sci. Technol. B.*, vol. 27, no. 4, p. L18, 2009.

5:20pm **HI+2D+AS+MC-ThA10 Sub-100nm Nanofabrication using Helium and Neon Ion Beams**, James Sagar, C. Nash, N. Braz, T. Wootton, M.J.L. Sourribes, T.-T. Nguyen, R.B. Jackman, P.A. Warburton, London Centre for Nanotechnology, UK

Sub-100nm Nanofabrication using Helium and Neon Ion Beams

J. Sagar¹, C. R. Nash¹, N. Braz^{1,2}, T. Wootton^{1,2}, M. J. L. Sourribes^{1,2}, T.-T. Nguyen^{1,2}, R. B. Jackman^{1,2}, and P. A. Warburton^{1,2}

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Using a Zeiss Orion NanoFab we have created sub-100nm devices for experiments in quantum electronics and nanophotonics. The Orion NanoFab has the ability form an ion beam with either helium or neon gas. This makes the Nanofab a much more versatile instrument for nanofabrication since large area mills can be performed using Ne without the need for a Ga FIB column. The use of a Ne gas field ion source (GFIS) in the Orion NanoFab allows fabrication of sub-100nm devices on timescales comparable to that of conventional liquid Ga FIB but with considerably enhanced fidelity due to an increased sputter yield (ten times greater than that of He) whilst retaining a small probe size (≤ 5nm). Using a Ne ion beam we have

fabricated two kinds of nanoscale superconducting devices: a superconducting nanowire based on a compound low-T_C superconductor; and an array of nanoscale Josephson junctions based on a compound oxide high-T_C superconductor. The use of an inert-gas ion species in these devices is extremely important as Ga implantation into superconducting materials has previously been shown to suppress superconductivity. The extremely small probe size of the He GFIS has allowed us to create sub-20nm apertures in a variety of materials. Sub-20nm apertures in InAs nanowires and in graphene have been fabricated for experiments in quantum coherent electronics and quantum nanophotonics respectively.

Thursday Evening Poster Sessions

Helium Ion Microscopy Focus Topic

Room: Hall D - Session HI-ThP

Aspects of Helium Ion Microscopy Poster Session

HI-ThP1 Fabrication of Single Atom Tip and Characteristics of Gas Field Ion Source at Room Temperature, In-Yong Park, B. Cho, C. Han, J. Kim, N.-K. Chung, S.J. Ahn, KRISS, Korea

For a long time, scanning electron microscope (SEM) and transmission electron microscope (TEM) have been playing a significant role in research and industry, especially for high resolution imaging. However, both of them stand in need of sample preparation, such as coating with metal for non-conducting material and slicing for electron transmission, etc. Recently, helium ion microscope (HIM) shows that sub-nanometer imaging resolution could be possible regardless of conducting or non-conducting material without sample metal coating, including nanometer resolution patterning. HIM uses a gas field ion source (GFIS) which is different from tungsten filament, Schottky emitter and liquid metal ion source. In this work, we center around assessment of the characteristics and processes of GFIS in respect to temperature.

The needle like tip to which a high electric field is applied has a fundamental component in GFIS and much of the source physics of GFIS are very similar with field ion microscopy (FIM) [1]. When gases are introduced around the tip, polarized gases are attracted to tip and then ionized at an apex sites by transferring electron from gas to tip. If an ionization sites are confined to only a few atoms, then angular current density can be increased and adaptable to a charged particle microscope although the total current of ion beam is picoampere level. There are a lot of required conditions for generation of stable and high ion beam current. Among them, tip temperature which is related to stability, current density and energy spread is very important. Generally, in order to get an enough ion beam current, tip is cooled by refrigerant and maintained continuously. Supposing a source tip can be used for ion microscope at room temperature without cooling system, maintenance cost and price of apparatus will be reduced with simplified microscope. The aim of this study is to provide an overview of comparison of ion beam in cooled and uncooled tip. We observed the FIM of tungsten tip and fabricated the single atom tip through field assisted nitrogen etching [2]. Furthermore, to simplify the whole process, we did not anneal the tip for tip cleaning through resistive heating. We measured the total ion beam current generated from multi atom and single atom according to gas species, applying voltage, gas pressure and temperature. After that, we examined the potential to be an ion source, which generates ion beam at room temperature, for ion microscope by calculating an angular current density and stability.

[1] Müller, E.; Bahadur, K., Phys. Rev. 102, 624, 1956

[2] Rezeq, M., Pitters, J. and Wolkow, R, J Chem Phys. 124, 204716, 2006

HI-ThP2 Probing Structural Aspects of <10 nm-sized Young Soot, M. Schenk, University of Bielefeld, Germany, S. Lieb, University of Southern California, H. Vieker, A. Beyer, Armin Götzhäuser, University of Bielefeld, Germany, H. Wang, University of Southern California, K. Kohse-Höinghaus, University of Bielefeld, Germany

Because of its importance for climate, environment and health, detailed information on soot emission is needed. It is particularly important to understand how characteristic parameters, such as size, morphology, and chemical reactivity of soot particles depend upon the formation process. Small soot particles can be particularly dangerous since they can penetrate deeply into the respiratory tract and they may also be more reactive than more mature, larger particles [1]. Investigating the nature of <10 nm soot particles is thus of particular interest. We have studied the morphology of nascent soot probed from previously well-characterized burner-stabilized ethylene flames [2] with Helium-Ion Microscopy (HIM). HIM allows unambiguous recognition of smaller nascent soot particles than those observed in previous transmission electron microscopy studies. With this technique, surface details are visible down to approximately 5 nm, and particles as small as 2 nm are detectable. The results demonstrate that nascent soot is structurally and chemically inhomogeneous, and even the smallest particles can have shapes that deviate from a sphere [3]. Structural details will be discussed.

[1] B. Frank, R. Schlögl, D.S. Su, Environ. Sci. Technol. 47, 3026-3027, 2013.

[2] A.D. Abid, N. Heinz, E.D. Tolmachoff, D.J. Phares, C.S. Campbell, H. Wang, Combust. Flame 154, 775-788, 2008.

[3] M. Schenk, S. Lieb, H. Vieker, A. Beyer, A. Götzhäuser, H. Wang, K. Kohse-Höinghaus, ChemPhysChem 14(14), 3248-3254, 2013.

HI-ThP3 Fabrication of Carbon Nanotube Nanogap Electrodes by Helium Ion Sputtering for Molecular Contacts, C. Thiele, Karlsruhe Institute of Technology, Germany, H. Vieker, André Beyer, Bielefeld University, Germany, B.S. Flavel, F. Hennrich, Karlsruhe Institute of Technology, Germany, D.M. Torres, T.R. Eaton, University of Basel, Switzerland, M. Mayor, M.M. Kappes, Karlsruhe Institute of Technology, Germany, A. Götzhäuser, Bielefeld University, Germany, H.V. Löhneysen, R. Krupke, Karlsruhe Institute of Technology, Germany

We use helium ion beam lithography to sputter nanogaps of 2.8 ± 0.6 nm size into single metallic carbon nanotubes embedded in a device geometry (1). The high reproducibility of the gap size formation provides a reliable nanogap electrode testbed for contacting small organic molecules. To demonstrate the functionality of these nanogap electrodes, we integrate oligo(phenylene ethynylene) molecular rods, and measure resistance before and after gap formation and with and without contacted molecules.

(1) C. Thiele, H. Vieker, A. Beyer, B.S. Flavel, F. Hennrich, D.M. Torres, T.R. Eaton, M. Mayor, M.M. Kappes, A. Götzhäuser, H.v. Löhneysen, R. Krupke: Fabrication of carbon nanotube nanogap electrodes by helium ion sputtering for molecular contacts, Appl. Phys. Lett., doi: 10.1063/1.4868097 (2014)

HI-ThP4 High Resolution UHV Helium Ion Microscopy of Work Function, Step Edges and Crystal Structure, Gregor Hlawacek, Helmholtz-Zentrum Dresden - Rossendorf, Germany, M. Jankowski, R. van Gastel, H. Wormeester, H.J.W. Zandvliet, B. Poelsema, University of Twente, Netherlands

Helium Ion Microscopy---in particular under UHV conditions---is well known for its high resolution imaging capabilities and the exceptional surface sensitivity. Here, we utilize both of these outstanding characteristics of this technology to visualize step edges, minute changes in composition and structural properties of a Ag/Pt alloy layer grown on Pt(111). A work function contrast of only a few ten's of meV allows to distinguish between areas of different Ag content in the alloy layer. As a result step edges on the Pt(111) crystal overgrown by the alloy layer become visible. Furthermore, the regular arrangement of FCC and HCP areas in the alloy layer could be revealed using fast Fourier image analysis and dechanneling image contrast. The measured spacing of 6 nm agrees well with the expected value. Low energy electron microscopy has been used to cross check the results and further analyze the alloy layer.

This research is supported by the Dutch Technology Foundation STW, which is the applied science division of NWO, and the Technology Programme of the Ministry of Economic Affairs.

HI-ThP6 Ion Beam Analysis in a Helium Ion Microscope, Nico Klingner, R. Heller, G. Hlawacek, S. Facsko, J. von Borany, Helmholtz-Zentrum Dresden - Rossendorf, Germany

Helium ion microscopes (HIM) have become powerful imaging devices within the last decade. Their enormous lateral resolution of below 0.3 nm and the highest field of depth make them a unique tool in surface imaging. So far the possibilities to identify target materials (elements) are rather limited.

In the present contribution we will show concepts as well as preliminary studies on the capability, efficiency and the limits of applying (Rutherford) Backscattering Spectrometry (RBS) within a HIM device to image samples with target mass contrast and to analyze target compositions.

We will present different concepts of how to realize RBS in a HIM and point out mayor challenges and physical limitation.

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 Wolkow, R.: HI+2D+AS+BI+MC-ThM3, 6;
 HI+2D+AS+BI+MC-ThM5, 7
 Woods, A.S.: HI+2D+AS+BI+MC-ThM13, 7
 Wootton, T.: HI+2D+AS+MC-ThA10, 9
 Wormeester, H.: HI-ThP4, 10
 Woszczyna, M.: 2D+AS+HI+NS+SS-ThM3, 5
 Wu, C.-C.: 2D+AS+HI+NS+SS-ThM4, 5
 Wuister, S.: HI+2D+AS+MC-ThA8, 8
 — **X** —
 Xu, C.: HI+2D+AS+BI+MC-ThM13, 7
 — **Y** —
 Yu, Y.: 2D+AS+HI+MC+NS+PS+SP+SS-TuA4, 3
 — **Z** —
 Zandvliet, H.J.W.: HI-ThP4, 10
 Zhang, L.: HI+2D+AS+BI+MC-ThM6, 7